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WL-TR-93-4025

STUDENT SUPPORT TO WL/ML AND WL/AA

WANDA VOGLER

SOUTHWESTERN OHIO COUNCIL
FOR HIGHER EDUCATION
2900 ACOSTA STREET, SUITE 142
DAYTON OHIO 45420-3467

JAN 1993

FINAL REPORT FOR 05/01/90-12/31/92

434 pg

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MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT PATTERSON AFB OH 45433-7734

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This technical report has been reviewed and is approved for publication.

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No 0704 0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
JAN 1993

3. REPORT TYPE AND DATES COVERED
FINAL 05/01/90--12/31/92

4. TITLE AND SUBTITLE
STUDENT SUPPORT TO WL/ML AND WL/AA

5. FUNDING NUMBERS
C F33615-90-C-5904
PE 62102
PR 2418
TA 04
WU 76

6. AUTHOR(S)
WANDA VOGLER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
SOUTHWESTERN OHIO COUNCIL
FOR HIGHER EDUCATION
2900 ACOSTA STREET, SUITE 142
DAYTON OHIO 45420-3467

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT PATTERSON AFB OH 45433-7734

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
WL-TR-93-4025

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

This Technical Report gives a summary of the research performed in the materials and optical sciences in support of the Wright Laboratory Materials Directorate under a Student Support contract. The report contains an abstract covering the nature of the research and the results for each of 111 tasks completed under the contract.

14. SUBJECT TERMS
Nonmetallic Materials, Mechanics & Surface Interactions, polymers, nonstructural materials, metals & ceramics, Electromagnetic, Matl's, Electronic Manufacturing & Optical materials, system support

15. NUMBER OF PAGES
434

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

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Administration/Management

In May 1990, Wright Laboratory, Materials Directorate awarded Southwestern Ohio Council for Higher Education (SOCHE) a three plus year Contract. The Contract was for SOCHE to provide 89,960 person hours of short-term intermittent technical support to personnel at the Materials Directorate (MD) and Avionics Laboratory (AA). The award made possible the continuation of activities SOCHE has delivered to the MD since 1986. People and processes were already in place allowing us to dovetail activities and transition to this new Contract activity.

The Student Support Program office located in Kettering, has all the necessary equipment and support to fulfill the Contract requirements. Since the first Contract award in 1986, SOCHE has had the same Director for the Student Support Program. This half-time Director and a full-time Administrative Assistant has delivered 7,401 hours of the 7,849 person hours approved for three administrative/ management tasks. The unused hours are due to two simultaneous contracts operating since July 1992. SOCHE allocated administrative hours and effort to each Contract according to amounts needed to support student task activity.

Systems to monitor and control the budget and individual tasks were developed during a previous Contract. Adjustments were made as needed. This allowed us to readily have all the information necessary to prepare required reports: monthly Performance and Cost Reports, monthly Management Reports, technical reports for individual tasks and quarterly Contract Funds Status and vouchers for billings.

In the first month of Contract activity, SOCHE received and filled 16 student tasks. The number of tasks steadily increased during the next year. Because of the accelerated demand for student workers, MD modified the Contract in 1991 to increase the dollars and student person hours by 25%. The number of active tasks since then has been approximately 45. Student Support hired 89 students to provide support in 108 tasks for 19 technical areas of MD and AA. Student Support Program delivered approximately 94% of the 98,286 approved student person hours.

Approach to Establishing and Maintaining Positive Relationships

Maintaining rapport with the Materials Directorate personnel continued as a priority. This was done in a variety of ways. Before the recruitment process began, the Director consulted the Government Task Leaders to find out the academic and technical background needed to do the task. We sought their opinion in critiquing the technical qualifications of student applicants. The Task Leaders received information throughout the task about anything that could affect the student worker or task performance. Anytime there was a problem, the Director made every effort to resolve the issue to all parties' satisfaction. The staff kept task leaders informed by correspondence, telephone calls and site visits. Thirty-five Government Task Leaders had the opportunity to rate the strengths and weaknesses of program staff and procedures through surveys. (See Attachment 1)

Recruiting and Selecting Students

Since this was our second Student Support Contract, the contacts on campuses were already in place. Liaison persons, previously designated by the college/university presidents, provided Student Support with a point of contact on each campus when the need to recruit students occurred. It also provided students with an accessible place to pick up applications and task descriptions. Student Support provides brochures to appropriate personnel for general program information.

Task requirements ran the gamut in science and engineering - chemistry, computer science, physics and engineering (material science, mechanical, electrical, electro-optics, chemical, etc.). Campus liaison persons, deans, department chairs and individual faculty members received information describing tasks to recruit interested and qualified students. On those campuses that produced newsletters, tasks were published in the newsletters. Campus departments with electronic mail publicized tasks for us also. Students already working in tasks received publicity about openings.

The Director made visits to campuses to generate interest, answer questions and generally keep the program visible. Anytime there was a special event for co-operative education or internships on a local campus, we participated.

Students from 12 institutions submitted credentials for consideration. Credentials consist of an application, course listings/transcripts and resume plus letters of reference if the student wishes. In order to maintain a prospective file of qualified applicants, Student Support encouraged and accepted credentials on a continual basis. In addition to campus referrals, Government Task Leaders would sometimes refer a student they knew. Materials Directorate personnel often teach and do research on campuses. They have occasion to get to know students through these contacts or through other student oriented programs on Base.

Student Support screened credentials to determine academic standing (GPA), citizenship status, previous work experience and academic preparation. Student Support forwarded credentials of those students who seemed to meet the standards to the Government Task Leader. The Government Task

Leader reviewed credentials and decided which students should be invited to see the work setting. Student Support staff contacted those students to set up appointments with MD personnel. Task Leaders were asked to assess the technical qualifications of the students. After conferring with the Task Leader, the Director ranked the student applicants according to their qualifications. The Director negotiated with the most qualified first. If this student did not accept the position, the Director contacted the next student.

After the Director selected the ablest student from those qualified, a written cost estimate (task proposal) was prepared and submitted for government approval. The person hours and schedule of work was determined after conferring with the Task Leader and the student. The 89 students that Student Support hired attended Sinclair Community College, The Ohio State University, University of Cincinnati, University of Dayton, Wright State University and Xavier University.

Recruitment activities were on-going throughout the year. Anytime there was an opportunity to meet with staff or faculty on a campus, it was done. Special events on campuses were also attended. The Director belongs to local and national associations for co-operative education and experiential education. Whenever meetings were held that seem to increase interaction with campus personnel, the Director attended.

Supporting, Directing and Supervising Students

New students hired received an orientation to the Program. Contracts, time sheets, general correspondence and telephone calls all serve to support and direct the students' activity. To assist new students and Task Leaders in setting goals and make expectations clear, Student Support developed a "Handbook of Information" that each received at the beginning of a Task. Students also received a copy of their task description as prepared by the Government Task Leader.

As they completed tasks, students submitted technical reports about their research activity. Part II of this report contains all the technical reports.

Student Support used the technical reports in evaluating the students' performance. Government Task Leaders were asked to evaluate the students' strengths and weaknesses in task performance through written and oral assessments, at least once during a task assignment. They were asked to do this with the student and to allow the student to have input (See Attachment 2 for summary). Only two students were removed from a task assignment for poor performance.

The Director contacted Task Leaders throughout the task to see if things were going well. We encouraged Task Leaders to let us know immediately if problems arose that needed attention.

Predominantly, students worked the 1040 hour tasks in a part-time during the academic year, full-time during the summer schedule. Whenever the Task Leader stated a preference, we selected students who could work full-time, alternating quarters. While the majority of tasks were 1040 hours each for 12 months, we were flexible in providing tasks for summer or other short terms.

Task Assignment
by Materials Directorate Division

FIM - 1

AARI - 2

MLB - 26
  MLBC - 6
  MLBM - 3
  MLBP - 11
  MLBT - 6

MLI - 12
  MLIC - 1
  MLIP - 3
  MLIM - 8

MLL - 27
  MLL - 6
  MLLM - 13
  MLLN - 7
  MLLP - 1

MLP - 36
  MLP - 1
  MLPJ - 25
  MLPO - 10

MLS - 7
  MLS - 2
  MLSA - 2
  MLSE - 3

Other Evaluations

Task assignments have enriched students' academic preparation in a variety of ways: two master's theses completed, a dissertation in progress; senior research projects; co-authorship of articles submitted to professional journals; and co-op credit and internships for career related work experience.

As students completed their tasks, Student Support asked them to complete a questionnaire about all aspects of their

Summary

The Student Support Program was a success based on all the feedback we received. Feedback tended to be more positive than not in all aspects of our surveys.

Credit should be given to those who made it possible and rewarding for us to administer this Contract. Government Task Leaders were always willing to provide information or to cooperate with the Student Support staff whenever asked. Students gained mentors as Task Leaders took an interest in and spent time guiding them. The positive relationship and communication level between the Project Engineer, Contract Negotiators and Director was a key factor in keeping things running smoothly.

Technical Data, as required, was delivered on time almost without exception. The delivery of 94% of approved student person hours rather than 100% caused us to have unexpended funds. One-hundred percent is a goal to strive for. No task exceeded the approved funding level.

This has been a win-win Contract for MD, AA and the students involved. Government Task Leaders had the benefit of on-going efforts by students to support the work of the division. Students brought new ideas and enthusiasm with them and potential workers/colleagues were identified. State-of-the-art work settings allowed students to see real applications of their classroom experiences. The opportunity for hands-on experience related to academic study was the most important reason, a major of the students we surveyed, gave for participating in the Program. Task assignments provided research opportunities for students not readily available from other sources.

We look forward to continuing our efforts on behalf of the Materials Directorate.

Attachment 1
January 1993
35 Surveys Sent
31 Responded

PROGRAM & STAFF EVALUATION RESULTS

We are interested in your comments and suggestions concerning the effectiveness of our services to the Materials Laboratory. Would you please take a few minutes to complete the following:

Rating Scale: A - Outstanding
B - Above Average
C - Average
D - Below Average
F - Not Applicable or No Experience

		A	B	C	D	F
1.	Student selection procedures.	10	15	5	0	1
2.	Match of applicants to Task Assignments.	13	15	3	0	0
3.	Communication of information.	18	11	1	1	0
4.	Timeliness of information.	19	7	5	0	0
5.	Willingness and ability to resolve problems.	16	11	0	0	4
6.	Responsiveness to your inquiries.	22	7	0	0	2

7. What has been the major benefit of our program to you/your organization?

(Please see attachment)

8. How many students have we placed with you?

9. Comments/Suggestions:

(Please see attachment)

Signature: (on file) Organizational Symbol: (on file)

Ext: (on file)

7. Categorized comments of major benefits of our program from the government task leaders:

Valuable asset in keeping research going/ additional manpower.	15
Outstanding coupling of lab interests and student capabilities.	3
High quality student assistants.	16
Recruiting of personnel.	2
Provides ability to pursue research projects there was never time for before.	2
Tremendous increase in output.	1

9. Categorized comments/suggestions:

Top notch students.	2
Excellent program/Keep up the good work.	8
Publish list of students and lab groups.	1
Would like to pay students more.	1
Change rating scale.	1
Full-time alternating quarter students have really worked well.	1

SOCHE
2900 Acosta St., Suite 142
Dayton, OH
513-297-3159

STUDENT SUPPORT PROGRAM

STUDENT EVALUATION

Attachment 2

103 **Surveys Sent**
81 **Responded**

Student: ______
Dates of Employment: ______
Guiding Scientist or Engineer: ______
Organizational Symbol: ______ Extension: ______
Task No: ______ Objective: ______

Performance Evaluation: 1 ___ Outstanding 2. ___ Above Average 3. ___ Average 4. ___ Below Average 5. ___ Not Applicable

	(1)	(2)	(3)	(4)	(5)
1 Leadership characteristics	16	25	16	0	24
2 Sensitivity to problems and ability to resolve	33	43	5	0	0
3 Effective in preparing and organizing work	34	36	11	0	0
4 Takes initiative, self-starter	40	31	9	0	1
5 Demonstrates willingness to accept responsibility	48	25	6	0	2
6 Demonstrates analytical ability	31	42	6	0	2
7 Demonstrates original and creative thinking	17	47	9	0	8
8 Demonstrates accuracy and thoroughness	40	36	5	0	0

	(1)	(2)	(3)	(4)	(5)
9. Exhibits diligence and perseverance	45	30	5	0	1
10. Demonstrates ability to learn	59	21	1	0	0
11. Has healthy attitude toward organization	42	30	5	1	3
12. Is cooperative in working relationships with others	49	28	2	0	2
13. Quality of work performed	39	38	4	0	0
14. Effectiveness in oral communication	24	46	9	0	2
15. Effectiveness in written communication	15	35	15	0	16

Overall Performance:

Outstanding	Above Average	Average	Below Average
44	34	1	0

Attendance: 77 Regular 2 Irregular
Punctuality: 77 Regular 2 Irregular

Comments on potential growth and development for student in major field of study: ______

8

Have you discussed this evaluation with the student? 59 Yes 11 No (9 NOT INDICATED)

Student's Comments: ______

Signatures:

dent (on file)
Guiding Scientist or Engineer (on file) Date (on file)

[illegible]

Attachment 2

Attachment 3
86 Surveys Sent
56 Responded

STUDENT SUPPORT PROGRAM
TASK ASSIGNMENT SURVEY

The following questionnaire is designed to assure complete anonymity. As a student worker, your evaluation of the program is important to us in planning and improving the program, so please be candid.

I. How did you learn about the Student Support Program? Check one.

14 a) Advertisement (flyer, brochure, campus paper)

12 b) Faculty member

7 c) Student worker

4 d) WPAFB employee

19 e) Other (please specify) campus co-op-11 internship-8

II. Please rank your first and second most important reasons for participating in this program.

1st	2nd	
16	28	Part-time employment for pay
3	13	Opportunity to conduct research work (Senior project, graduate thesis, etc.)
2	1	Co-op position for credit
35	14	Opportunity for hands-on experience related to academic study

III. Do you feel you received adequate information and assistance from the Student Support staff in carrying out your Task Assignment?

Yes 53 No 3 If no, please elaborate. No housing help was provided-1

IV. Did your guiding engineer or scientist establish clear expectations about your Task Assignment?

Yes 56 No 0 If no, please elaborate.

V. Did the Task Assignment meet your expectations?

Yes 51 No 5 If no, please elaborate. Any Joe Shmoe could have done it-1

Attachment 3 page 2

A = A LOT
B = SOME
C = A LITTLE
D = NOT AT ALL
E = NOT APPLICABLE

VI. How much were you exposed to each of the following during your Task Assignment? (Circle one letter per line.)

A	B	C	D	E		
16	26	8	3	3	1.	Philosophy of research
17	26	8	3	3	2.	Use of scientific method to solve problems
27	18	5	1	4	3.	Use of experimental checks and controls
27	14	2	0	7	4.	Measurement techniques
17	13	14	5	7	5.	Design of equipment
18	12	15	2	9	6.	Process of design of an experiment
39	8	4	3	2	7.	Data analysis (with or without computer assistance)
18	11	10	9	8	8.	Computer programming
16	21	14	2	3	9.	Acquisition and use of scientific literature (books, audio visual, etc.)
22	12	13	2	7	10.	Identification of new questions as a consequence of scientific exploration
22	14	14	2	4	11.	Teamwork in scientific research
32	12	9	1	2	12.	Use of advanced scientific equipment
12	19	14	7	4	13.	Other students with similar interests and goals
16	24	11	8	4	14.	Scientists working in different areas of research
6	23	19	3	5	15.	Information on scientific careers
					VII.	How much has your experience in this Task contributed to your development in each of the following?
33	15	5	0	3	1.	Working with professionals
32	15	7	2	0	2.	Responsibility on a job
26	19	9	1	1	3.	Understanding of scientific principles
18	24	11	1	2	4.	Scientific vocabulary
12	23	15	3	3	5.	Ability to write a technical report

A	B	C	D	E		
28	19	7	1	1	6.	Understanding of your interests and abilities
22	24	7	1	2	7.	Educational goal setting
16	29	8	1	2	8.	Insights into career opportunities in science
22	26	6	1	1	9.	Professional career development

VIII. To what extent did you benefit from the following?

A	B	C	D	E		
33	17	6	0	0	1.	Explanations of work by guiding scientist or engineer
6	18	8	5	11	2.	Tours of other laboratories or installations
34	15	7	0	0	3.	Informal talks with guiding scientist or engineer
31	20	5	0	0	4.	Discussions with other scientists or engineers
12	19	18	4	3	5.	Interactions with other student workers
5	16	17	11	7	6.	Advise from the Program Director

A = Strongly Agree
B = Agree
C = Disagree
D = Strongly Disagree

IX. How do you feel about your Task Assignment experience?

A	B	C	D		
44	10	2	0	1.	I enjoyed the experience
37	15	4	0	2.	I liked the scientific research
25	27	3	1	3.	I was satisfied with the way I spent my time
41	13	2	0	4.	I learned a lot
32	21	3	0	5.	I feel I contributed to the research results

X. What did you like most about the program?

Chance for career related experience - 18
R&D environment (people and projects) - 30
Flexible schedule/hours - 6
Realistic view of governement work, excessive delays and frustrations - 1
Exposure to laboratory setting - 2
Well organized program - 2

XI. Please include any comments or suggestions for improving the program.

Don't make co-op students pay quarter after quarter	- 1
Would like to tour other labs on and off WPAFB	- 1
Interact with researchers outside our group	- 1
Be careful about job description and work matching	- 2
Provide housing assistance	- 1
Holiday pay would be nice	- 1
Task leader should communicate with student more	- 1

WE ENJOYED WORKING WITH YOU AND HOPE YOU HAD A G-R-E-A-T EXPERIENCE.

Please return your survey to:

Southwestern Ohio Council
for Higher Education
Student Support Program
2900 Acosta Street
Suite 142
Dayton OH 454[illegible]0

(513) 297-3159

Note: As an anonymous voluntary survey, we are not able to do follow-up to increase the response rate

STUDENT SUPPORT PROGRAM

Southwestern Ohio Council For Higher Education

Part II - Final Management Report

Technical Reports of Student Tasks

Computer Assistance in Planning Operations

Task Order No. 2
Student Support Program

Son Hai Nguyen
Wright State University

15 May 1991

Guiding Engineer
Mr. Robert Kincses
WL/MLJ

ACKNOWLEDGMENTS

The knowledge that I have gained during the past year working at the Materials Laboratory is very valuable and important. Mr. Mark Groff and Mr. Robert Kincses have both taught me many aspects of how to perform at a job cite and how to plan things ahead. Mark was very helpful during the first several months of my job. Mr. Kincses has widened my perspective on life in general. He was a great leader and I am very glad I had the opportunity to work with him. The last several months of my job, I had the pleasure of working with Lieutenant Bruce Graw. Bruce has been my information center for any problem I had in writing programs in Pascal.

TASK ASSIGNMENT

Many programs were written in the past year, mainly in Fortran. The first group of programs dealt with computer usage. These programs collect monthly data from four different computer systems (Misvax, Apollo, Ibpl, Zeus). These data consist of CPU time, number of pages printed, elapsed time, average disk-space, etc. of each computer user. These data are then tabulated into a monthly and yearly summation for each user. Graphics portion of these programs, allows the system manager to view the calculated data and determines the needs of the users. The second group of programs dealt with security check. These programs collect data from the login failure files from the four computer systems mentioned above. Similarly to the first group of programs, graphics portion of this group of programs is used by the system manager to observe the number of login failures on each day of the last month. If there exists a significant number of login failures on a particular day, the system manager would investigate further. The last and uncompleted task is the updated program written in Pascal for the Zenith computers. This program collects essential information regarding the system of a particular user and passes it on to a new updated file. This program is designed to run automatically when the user turns on his Zenith computer. This saves the systems manager time needed to manually update each computer.

Optical Radar Pre-Amplifier

Task Order No. 3
Student Support Program

Michael Salisbury
University of Dayton

19 August 1991

Guiding Engineer
Mr. Paul McManamon
WL/AARI-2

ACKNOWLEDGMENTS

This report gives a summary of work accomplished under a contract of Southwestern Ohio Council for Higher Education (SOCHE) at Wright Patterson Air Force Base. The work was done for Wright Laboratories AARI-2. I would like to thank Dr. Paul McManamon, Lt. Scott McCracken at AARI-2, Wanda Vogler and Pamela Douglas of SOCHE for their support during the last year.

TASK ASSIGNMENT

The primary focus of this research was to evaluate a fiber laser preamplifiers potential for use in a coherent laser radar system. One other important related task was assigned. The performance of a liquid crystal beam steering device under development through a contract with Raytheon was characterized as a component of the coherent laser radar system.

The first seven months of the contract were spent developing the laser radar setup. Among the most critical factors of the system were component alignment and beam polarization issues. The fiber optic mixing proved to be the most challenging aspect of the system. Focusing the local oscillator and signal beams into the fibers was tedious and time consuming. The fibers were polarization preserving single mode fibers with elliptical cores. In order for there to be efficient mixing of the two beams it was necessary to ensure that the polarizations of the beams coupled into the fibers were along the major axes of the fibers. An experimental technique using half wave plates, a detector and a Tektronix digitizing signal analyzer was developed to accomplish this.

Once a return signal was detected off a mirror target, measurements were made using diffuse targets and moving targets to determine a procedure to be used to test the liquid crystal beam steerer. A significant amount of time was spent insulating the detector in the setup to minimize electrical interference from the acousto-optic modulator used to frequency shift the local oscillator. It was necessary to insulate the detector to improve the sensitivity of the system for looking at a diffuse target.

The liquid crystal beam steerer was then evaluated in the system. Several characteristics of the steerers were uncovered about their performance in a system that would not have been discovered in a device characterization. A modulation in the return signal was linked to the drive electronics of the steerer. Polarization and reflectance measurements indicated that there was a deterioration of the coatings on the steerer, causing the evaluation work to be temporarily discontinued until another device could be obtained.

Active/Passive Channels with Nonmechanical Shared Aperture

Task Order No. 4
Student Support Program

Lawrence Barnes
University of Dayton

24 October 1991

Guiding Engineer
Mr. Paul McManamon
WL/AARI-2

ACKNOWLEDGMENTS

I would like to thank Paul McManamon for giving me the opportunity to perform this research and for his enthusiasm in the project. I would also like to thank Larry Myers for his tutelage and confidence in my competence to obtain experimental data for him. I would especially like to thank Bill Martin, Rob Fetner and Deanna Won for their help in obtaining equipment, identifying and solving experimental problems, and in general, putting up with me in their lab. Dan Smith's programming efforts greatly assisted in the presentation of the experimental data and he revised it many times to suit our purposes. Finally, I greatly appreciate Wright Lab and AARI for providing a rich intellectual environment for research.

TASK ASSIGNMENT

The broadband beam agility experiment's purpose was to experimentally verify the form and position of energies passed through a beam steering system with a computer program used to model non-mechanical beamsteerers. The form of the energies was determined by profiling the energy in one dimension with line scans obtained from pictures acquired by the imaging system. The experiment verified the ability of the computer model to analytically predict the shape and position of the far field pattern of radiation.

A blazed grating was used to model the non-mechanical beamsteerer steering to a fixed angle. The grating, Bausch & Lomb (serial #1093 162), has 600 lines per millimeter and the blaze angle is 8° 38'at $\lambda_{tuned} = 5{,}000$Å and models an ideal blazed non-mechanical beamsteerer. A tunable Helium-Neon laser was used as a spectral source. The laser (PMS model LSTP-0010) offered the ability to tune to 7 different visible wavelengths (6401Å, 6328Å, 6293Å, 6118Å, 6039Å, 5940Å and 5435Å) individually (except for the 6293Å which would only lase with the detuned 6328Å) and pairs (6401Å & 6328Å, 6328Å & 6293Å, 6328Å & 6118Å, or 6118Å & 6039Å) of some wavelengths simultaneously. This allowed for experimentation in the effects of the spectral signature of the target.

The theodolite setup shown as in Figure 1 was used to measure angular positions of the energies utilizing retro-reflection for alignment. These measurements allowed a "sanity check" of the computer program to see if it accurately predicted the location of the energies and also were needed to handover angular measurements to the imaging system. The results from the theodolite data are listed with error analysis is shown in Figure 3. The imaging system was setup as shown in Figure 2 with the blazed grating used to model the non-mechanical beam steerer steering to a fixed angle. The target for the system was the spatial filter (pinhole) of the collimator and was set, using a collimating lens, to be an infinite distance away from the beam steering system. Neutral density filters used in pairs prevented saturation of the camera for profiling the main lobe and controlled the saturation (prevented over saturation) for profiling the side lobes of the image. The laser lines were the spectral signature of the point target and there was no background radiation about the point target. The slit was the limiting aperture for the optical beamsteering system and the lens and camera were the imaging optics of the system.

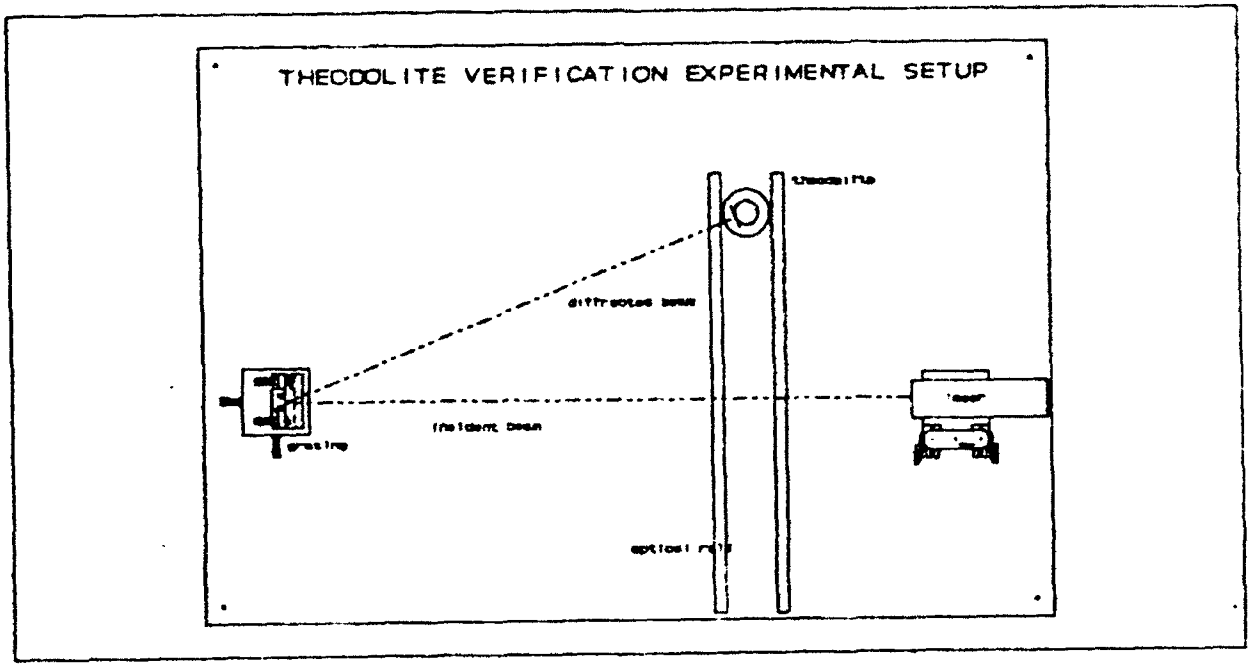

Figure 1

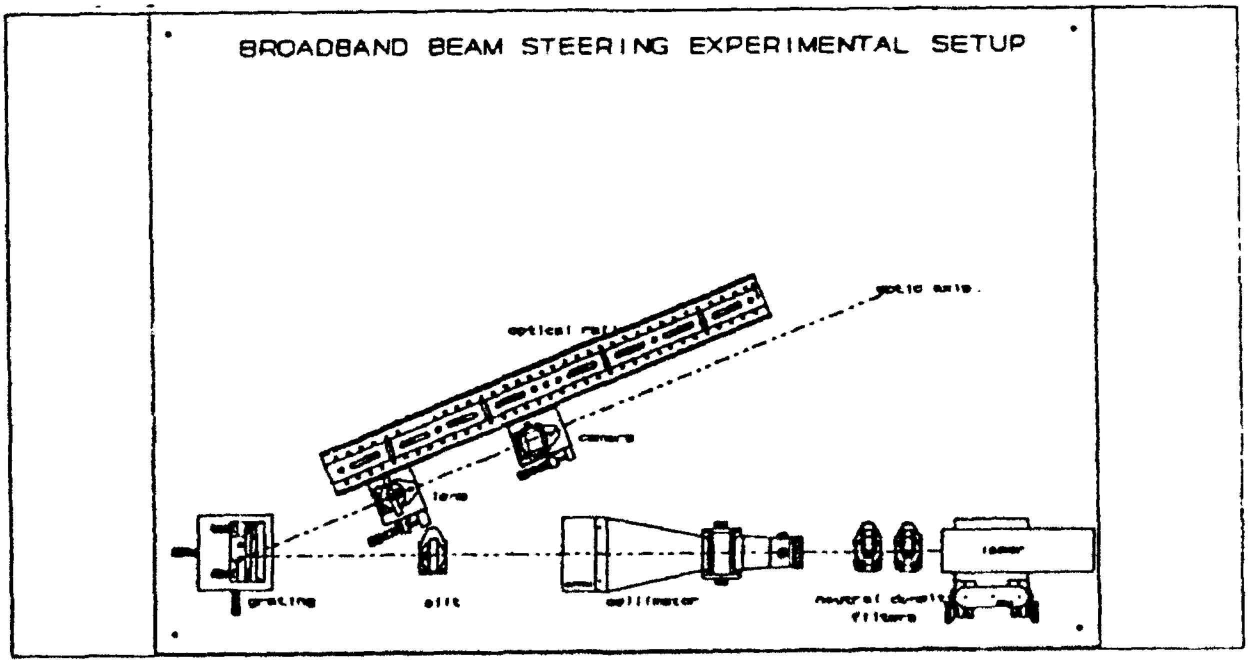

Figure 2

The theoretical form and position of the energies passed through the system were calculated using a computer model developed by Lawrence Meyers[1]. The experimental data was compared to the theoretical data, shown in Figure 3, and errors were observed. The observed errors were believed to by systematic, though attempts were made to account for this error through the quantification of random errors with uncertainty analysis also shown in Figure 3.

This systematic error was possibly caused by barrel distortion in the imaging lens and could be quantified using a grid or other array as the limiting aperture. The image observed at the camera plane would then be the Fourier Transform of the array (which would be another array) and the amount of barrel distortion in the lens could be determined through careful measurement of the distortion of the array.

Experimental data was obtained using Image Action software and a frame grabbing board. The Spiricon Laser Beam Analyzer was used for alignment purposes and does have the ability to measure distances and determine shapes in real time, but the ratio of the resolution (pixel size) to the field of view was approximately four times poorer than that of the Itex Image Action software. The data obtained with the camera was then incorporated into a data spreadsheet program (Axum) through the use of a custom computer written by Dan Smith.

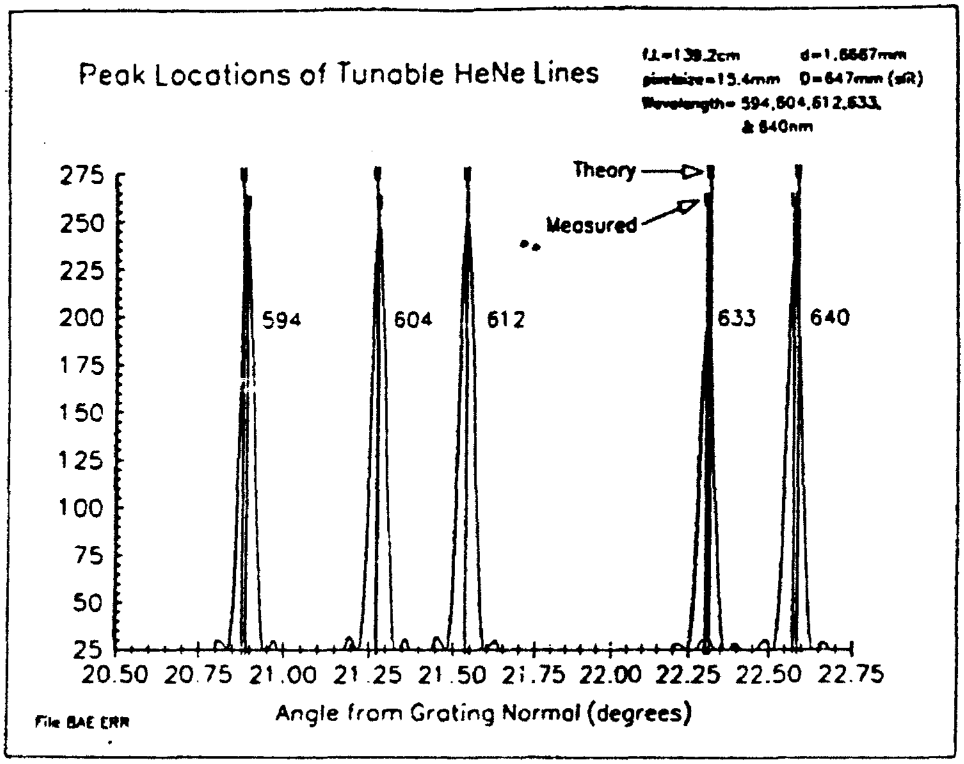

Figure 3

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[1] Lawrence Myers. *Broadband Beamsteering with Optical Arrays*. MS Thesis, Graduate Engineering and Research, School of Engineering, University of Dayton

The program selected and displayed the profile of the horizontal line with the greatest sum and converted the pixel number to an angle in the line scan chosen. The conversion from pixel number to angular position was achieved through Equation 1.

$$(\theta-\theta_{ref}) = \arcsin\left(\frac{(Pixel\ num - Pixel\ ref) * Pixelsize}{Focal\ length}\right) \qquad (1)$$

Where a paraxial approximation of the slope of the ray with respect to a reference axes is used. The invariance of Equation 1 to the distance from the beamsteerer to the lens was investigated.

The feasibility of using a non-mechanical beamsteerer in a broadband imaging system for a passive acquisition channel for LIDAR is greatly diminished by chromatic aberrations introduced by the grating structure of the beamsteerer. Figure 3 shows that the diffraction limited spot for a single target is spread in the image as a function of wavelength. Where ideally a single profile is desired, the profiles for each wavelength indicates the magnitude of the aberration. The use of holographic optical elements could correct for this aberration optically or a digital deconvolution scheme could be used to electronically correct the aberration, but the aberration must be at least partially corrected for the passive acquisition channel to function properly.

High Temperature Ceramic Composites Laboratory Aide

Task Order No. 5
Student Support Program

Brian Hornback
Wright State University

15 May 1991

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Dr. Ronald Kerans and Dr. Hay for their guidance in completing my Task assignment. I would also like to thank Mr. Ed Hermes and any other scientists who assisted me in my responsibilities.

TASK ASSIGNMENT

My tasks at the Materials Laboratory have been many and varied. During the summer of 1990, I worked with Dr. Hay as well as with a visiting scientist. Dr. Hay and the visiting scientist were concerned with studying various types of sols to be used for coating ceramic fibers. My task was to synthesize one of these sols, a gallia sol. The project required a fair amount of research in the technical library, looking for previous work on the synthesis of a gallia sol. Fortunately, an article was found and the sol was successfully, synthesized in the chemistry laboratory.

A second major project I have been concerned with is the production of a high temperature furnace (1700 C) for the coating of ceramics fibers. The task involved not only the production of the furnace itself, working from raw materials but also the creation of a control panel for the furnace. The furnace and control panel have proven to be quite useful for the tasks for which they were developed.

The remainder of my time has been primarily spent preparing samples for transmission electron microscope examination. This task involves cutting, grinding, and polishing ceramic samples to a thickness of about 30 microns and to a fine polish. The samples are then viewed with an electron microscope.

High Temperature Ceramic Composites

Task Order No. 6
Student Support Program

Heather Walls
Wright State University

7 May 1991

Guiding Engineer
Dr. Ron Kerans
WL/MLLM

ACKNOWLEDGMENTS

My deepest appreciation and thanks goes to Dr. Kerans for all of his insight and guidance. The knowledge he shared with me is unobtainable in school, and I am grateful for it.

TASK ASSIGNMENT

General duties included the winding of fiber tapes that were used to fabricate composites. Glass powder and fiber tapes were consolidation processed. The specimens were then sectioned, polished, examined optically in a microscope, tested and the data obtained was analyzed. General laboratory maintenance was also performed.

Specific research included indenter type push-out tests performed on three Beta-SiC* / potassium borosilicate# glass matrix composites using both diamond pyramid and flat bottomed probe indenters. The indenter push-out test is one of the most common methods used to characterize the interfacial properties of ceramic matrix composites. Fibers aligned perpendicular to the surface of the composite are loaded using a small probe until they debond and displace a small distance.

The objective was to determine whether the geometry difference between the two indenters had any effect on the push-out behavior of the composite system due to the fact that both types of loading probes have been used in the past to perform the test.

The glass compositions were chosen so that the coefficient of thermal expansion (CTE) of the glasses varied from slightly less to slightly greater than that of the fiber.

Composites were fabricated by hot pressing in vacuum and argon atmospheres at a temperature of 850°c and 500 psi pressure for 20 minutes. Specimens were cut into four different thicknesses and hand polished to a one micron finish. Testing was done using a modified metallurgical hardness tester mounted to a universal testing machine with computer data acquisition. The data recorded and analyzed was the push-out load as a function of time.

The residual stress state of the composites were examined before and after testing using transmitted cross polarized light. Fibers that were damaged could be excluded from testing.

* SCS-6 Textron Specialty Materials, Lowell, MA
\# Corning Glass Works, Corning, NY

It was found that the diamond pyramid permanently damaged the fiber by chipping and splitting and in some cases induced matrix cracking. The flat bottomed probe, on the other hand, did not damage either the fiber or the matrix.

In spite of the damage done by the diamond pyramid, the peak loads for the two probes were the same and the loading curves were similar.

High Temperature Ceramic Composites

Task Order No. 7
Student Support Program

Jennifer Reid
University of Dayton

19 June 1991

Guiding Engineer
Dr. Ron Kerans
WL/MLLM

ACKNOWLEDGMENTS

As I complete my final days with WL/MLLM, I would like to recognize those who enriched my research experience. Larry Matson, my guiding engineer, was very helpful and a pleasure to work with. In addition to my guiding engineer, I benefited from the working relationships I developed with Dr. Ron Kerans, Kristi Keller (UES), Mike Scott (UES, Metlab), Brian Hornback (SOCHE student) and Heather Walls (SOCHE student). In final I would like to thank the SOCHE staff for their efforts in my research experience.

TASK ASSIGNMENT

Task Order #7, which consisted of five main objectives, provided a great opportunity to explore the research involved with high temperature ceramic composites. The first objective was the fabrication of composite materials. Although I was able to regularly view the fabrication of YAG (Yitria Alumina Garnet, a ceramic composite), I did not have a chance to prepare a ceramic composite. The preparation of samples for optical and electro-optical evaluation was the second objective. I was able to actively pursue this particular objective as my participating engineer provided a large quantity of ceramic composites to be cut, mounted and polished for testing. The third objective was to gain experience in the areas of composite preparation and testing. Once again my cutting, mounting and polishing was utilized in the fulfillment of this objective. I was also able to test some composites for their composition by means of X-Ray Diffraction. The final two duties were data reduction and laboratory upkeep. I devoted more time to laboratory care than data reduction. The students were in charge of keeping the labs clean and orderly.

The majority of my work with WL/MLLM was performed in the Metlab within building 655. I was able to cut, mount and polish ceramic composite specimens in the Metlab. To cut the samples a diamond blade was used on a buehler saw which was lubricated with kerosene. In most instances the cut of ceramic 1" thick took between fifteen minutes and one-half hour, depending on the saw speed. After the sample was cut, it could be mounted or polished directly depending on the purpose of the sample. If the sample was mounted in epomet, it could be polished by hand or with the maximet automatic polisher. The automatic polisher was used for a quantity of four or more samples. To get a fine polish several grades of diamond paste or slurry must be used. It was common to start with 45 um and work down to 1/2 um. Hand polishing was generally utilized for the preparation of TEM thin foils. To prepare a foil, the sample must first be glued to a slide with heat sensitive glue. Next the sample was cut so that a thin layer remained on the slide for polishing. With a slide holder, the slide with the sample was polished starting on diamond wheels to get a thin foil of approximately 50 um in thickness. In final the foil was polished with 45 um, 15 um, 9 um, 6 um and 1 um diamond paste.

Another area which I was able to explore was X-Ray diffraction. After I received a radiation badge and training on the Rigaku X-Ray equipment I was able to determine the composition of two ceramic composites. Initially I was given a sample of YAP/A1203 to analyze. The components consisted of YAP and A1203. Next I heat treated the YAP/A1203 with the DTA/TGA at approximately 1450 C for one hour. The resulting ceramic, YAG, was then tested for composition with X-Ray diffraction. The resulting composition was YAP, A1203 and Y3A15012.

As a result of my research experience with WL/MLLM I was able to broaden my knowledge about the opportunities in materials research. Prior to my work in ceramics, I worked with polymers with another branch of the material labs. Upon my completion of this task, I will begin research for a masters degree in the area of structural composites in another branch of the material labs.

NDSANDS Computer Code Consolidation

Task Order No. 8
Student Support Program

Hugh J. Cullin
University of Dayton

3 May 1991

Guiding Engineer
Dr. Nicholas Pagano
WL/MLBM

ACKNOWLEDGMENTS

A thanks to Dr. Nicholas Pagano for putting in all the time necessary for all the government paperwork. Without his work, this task could not have been possible. A debt of gratitude to David Chu for his writing of several subroutines, without which the NDSANDS program would be at a standstill. Also a thanks to Tobey Cordell. Tobey helped to show practical applications of how NDSANDS would be useful by showing some practical applications for composites throughout the Non-Metallic materials lab. And finally a thanks to Dr. G. P. Tandon. Without his day-to-day instruction, helpful problem analysis skill, and overall project guidance, this task would not have been able to even begin.

TASK ASSIGNMENT

The task, which began back in March of 1990, consists of taking a program, NDSANDS, which already worked on MS-DOS (IBM compatible) machines, and converting the program to a more user-friendly form on the Macintosh. Along the way, the program, which was not quite complete, was also to be finished. Many obstacles had to be overcome.

First, a general background of Fortran on the Macintosh, Mactran, had to be studied. The next step was to look at a user's guide to the NDSANDS program itself, for MS-DOS machines. This program was thoroughly analyzed. Using Mactran, a dummy program was set up to create all of the windows that appeared in the user's guide for the actual program. The already written parts of the program had to be adjusted so they would agree with Mactran. The methodology used to make sure that the old parts agreed with Mactran was to set up small dummy programs. By using these small programs, an error could easily be identified. Many problems with loops were solved in this manner.

With Mactran, unlike the Fortran used by DOS machines, a number can not be read as a number, it is taken as a character. This was a difficult problem to overcome. David Chu, an expert with Mactran, wrote a subroutine to help overcome this problem. The other parts of the program still had to be developed. Again, dummy programs were used to solve this problem. Bit by bit, the new parts began to fall into place. As one small problem was finished, another would spring up. Dr. Tandon helped a great deal with these problems, as well as providing guidance in the general direction the newly created parts took the project.

Now, only a small portion of the program is working in its finished form. However, this is the most vital part of the program, as the other subroutines all refer to this subroutine as their basis for data. The other parts will be much easier to finish due to the subroutine written by David

Chu and the new subroutine that has already been finished. After the program is finished, it will be demonstrated to those scientists who are going to use it frequently, as well as Dr. Pagano.

NDSANDS stands for N-Directional Stiffness and Strength. The program NDSANDS deals with materials called composites. Composites are nothing more than a combination of materials. A composite is created by using fibers, coatings, particles, and matrices. There are many other factors involved in the process as well, but these four are the types of materials used in the process. Composites are used for making many things, such as airplane wings and computer boards. This task is concerning the actual program, not the number manipulation involved. A general background of what the program is going to do has helped, but was not really necessary to complete the task. The Macintosh Plus, the Macintosh II, Fortran textbook, Mactran guide, NDSANDS user guide, and other programs written by G. P. Tandon were the materials used to help with this task. Writing small dummy programs, getting subroutines from David Chu, and help in all areas from Dr. G. P. Tandon were also vital to the task.

Dynamic Oxygen Absorption Study

Task Order No. 9
Student Support Program

Mark Roselius
Wright State University

15 May 1991

Guiding Engineer
Dr. Harvey Paige
WL/MLBT

ACKNOWLEDGMENTS

I would like to express my gratitude to Dr. Harvey Paige, Mrs. Lois Gschwender and Mr. Ed Snyder for their guidance and assistance with the oxygen absorption studies. I would also like to thank Dr. Paige and Dr. James Liang for their guidance and assistance with infrared analysis. Also, I would like to express my appreciation to Dr. Kent Eisentraut, Dr. Ken Davis, Mr. Al Sicree, Mr. Vince Vidoni, Mr. Shashi Sharma and Mr. George Fultz for support provided in solving various equipment and computer problems that occasionally appeared over the previous year. Finally, I would like to thank Dr. Arthur Krawetz of Phoenix Chemical Co. for his continued support of the OXAB device.

TASK ASSIGNMENT

The task assignment is to study the resistance to oxidation of fluids and additives in military lubricants. The lubricants involved are synthetic base fluids with or without antioxdants. These materials were studied using the oxygen absorption apparatus, gas chromatography and infrared spectroscopy. Useful ranges for various parameters of the oxygen absorption apparatus or OXAB were established.

Oxygen Absorption Studies

Polyphenylethers Evaluation of the oxidation rate of 5 phenyl-4-ether or 5P4E is presently being conducted. These tests are being run on the Oxygen Absorption apparatus developed by Phoenix Chemical Co. and MLBT. The conditions used for 5P4E are a flowrate of 50ml/min, test temperature of 300C and a pressure of 733 to 744 torr, depending on the run. The duration of the tests presently range from 10 to 14 days. Phoenix Chemical is currently running tests of the same material for comparison.

Concentration Determination Absorbance Spectrometry is commonly used to find the quantity or concentration of an analyte in a solution or other matrix. One technique frequently used is to run a set of standards of known concentrations in a spectrometer and measure the Absorbance. By using Beer's Law:

$$A=ebc$$

where **A** is Absorbance, **e** is absorptivity constant, **b** is path length and **c** is concentration; the absorptivity constant for an analyte can be determined by plotting Absorbance vs concentration with the slope being **e**. From there it is a simple matter to find the concentration of the unknown using the same path length. Generally, this technique is accomplished using light in the visible or UV regions of the electromagnetic spectrum. In the following cases, infrared light was used since the species of interest absorbed strongly in that portion of the electromagnetic spectrum. The key element to the employment of this technique is to maintain a response where **A < 1**. Above **A=1** the response is no longer linear. This technique provided conclusive results in the determination of hydrogen containing molecules in compounds not expected to have them such as **Krytox AC, Krytox AZ** and a number of proprietary model compounds. In addition, it was used to find the concentration of tricresol phosphate (TCP) in polyphenylethers. The technique produced less satisfactory results in the determination of ketones present in perfluorinated compounds and in measuring concentrations of alkenes in silahydrocarbons. These measurements were made on a Perkin-Elmer Model 1750 FTIR except for the TCP spectra which was obtained with a Beckman 4250 dual beam IR. Development and refinement of this method is continuing.

GC/FTIR Several studies involving Gas Chromatography and FTIR have either been concluded or are presently underway. GC is used to separate the individual components of a by material differential vapor pressure or differential interaction with the column. Using an FTIR as the detector allows for IR spectra of the components to be produced. For this purpose, GC/FTIR was used to obtain the spectra for perfluoro-1,3-dioxolane, oxidation products from OXAB studies of polyphenylethers, $C_2F_4X_2$ and $C_2F_iX_jX'_k$ model compounds, and perfluoro-1,2-dimethoxypropane and perfluorodimethoxymethane. Virtually all this work was confined to qualitative analysis of spectra to assign frequencies to functional groups of interest.

Far infrared Spectral analysis of $C_2F_4X_2$ and $C_2F_iX_jX'_k$ model compounds was performed using a Nicolet far IR instrument. Far IR ranges from 500cm-1 to 50cm-1, a region where halogenated compounds are more easily distinguished from one another. As with GC/FTIR, the results obtained are qualitative and not quantitative in nature.

Microscope/FTIR MLBT acquired a Spectratech^R microscope attachment for use with the Perkin-Elmer FTIR. This instrument was attached to the IR and an alignment technique was developed. Preliminary studies of lubricant additive-to-metal bonding on the surface of test sample balls from four ball dynamic wear tests have been done. Also studied were metal coupons with organic materials applied.

Results

Oxygen Absorption Rates

Test Run #	tMAX	MAR
9109	220 hr	0.43 uM/g/min
9110	240 hr	0.46 uM/g/min
9111	208 hr	0.51 uM/g/min

Notes:

tMAX: time to maximum absorption rate
MAR: maximum absorption rate

Runs 9101 - 9108 were a series of equipment checkout runs required after a number of repairs. During these repairs a number of subsystems were given a thorough cleaning after two years of use. Some improvements such as a more reliable pump, furnace alignment block and glassware modifications to aid durability were added at this time. The test apparatus was also moved to an enclosed fume hood to control vapors given off during shutdown and cleaning. The computer that supports the apparatus also required repair and is in the process of being expanded in its capability to store data.

Optical Physics Laboratory Assistant

Task Order No. 10
Student Support Program

Edward A. Isaac
Wright State University

11 June 1991

Guiding Engineer
Mr. Marc Martin
WL/MLPJ

ACKNOWLEDGMENTS

I wish to thank Marc Martin for his support and encouragement throughout the task assignment. Several laboratory personnel were also very generous with their time and knowledge. I wish to extend my thanks and gratitude to those people; Nils Fernelius, WL/MLPJ; Tim Pottenger, WL/MLPJ; the staff of S.A.I.C.; and countless others which gave a helping hand.

The research atmosphere of the Materials Lab will continue to be an enjoyable and edifying place to work with great reverence to these people.

TASK ASSIGNMENT

The non-linear optic labs in the Materials Building at Wright Patterson Airforce Base have taken form. The primary objective of the lab and its staff is to test and isolate possible materials for use in protecting Air Force assets. This quest has brought our team to the edge of non-linear optical research. Scanning the spectrum, the scientist are developing methods which will protect Air force systems and personnel from laser attacks. Newer materials are being sought out and tested daily with the hope that from deep within these crystalline fretworks a signal may appear leading to further study and eventual understanding of this process.

Infrared laser systems pierce into oblivion trying to exhume characteristics which will help define a process known as phase conjugation. Our most recent plunge into deciphering non-linear properties is a new technique using waveguides. The non-linear property known as n2 is governed by the equation

$$n_2 = \frac{n - n_0}{I}$$

where n0 is the linear refractive index and I is the intensity of the laser energy. Defined by the equation above you will see that n2 is the intensity dependant refractive index of the targeted material. The waveguiding technique will determine the magnitude and sign of n2.

The principle involved is very similar to the theory utilized in fiber optics. Two different refractive indices "channel" the energy down through a selected zone. This "guiding" of the energy will exhibit predetermined characteristics which will be monitored and compared to other known results. The phenomenon which we are seeking must be sifted out of other effects such as thermal and electrical non-linearities.

Millions of data points represent many hours of thorough research and development. These points are sorted into hundreds of categories (ie: least squares fit, cubic spline fit, background noise, second surface signals, nfgs fit, ect.) for further analysis to determine which are the results of intensity variations or of known origins.

The faction which has gathered in the materials lab is diversified in many disciplines; this creates a sophisticated and demanding atmosphere in which research is of the utmost importance. The yearning to become the best in the world will continue to drive this elite force onward until scientists understand this phenomenon worldwide. Papers, lectures, conferences, and countless other activities are scheduled to help bring this knowledge forward and into practicality.

Division Computer Support

Task Order No. 11
Student Support Program

Keith Andrews
Wright State University

June 10, 1991

Guiding Engineer
Mr. Marc Martin
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Marc Martin for his help and encouragement during my task at Wright-Patterson AFB Materials Laboratory. Mr. Martin has taught me a great deal about what will be expected of me as a Computer Engineer and has helped me realize the importance of my studies at WSU with respect to how they relate to my future career.

TASK ASSIGNMENT

The task of Division Computer Support has a main objective of providing services to division personnel by the selection and implementation of appropriate software and hardware to accomplish their task based on future needs and current inventory of computer resources. This task encompasses the job of System Manager, Software Support and Design, Hardware Maintenance, Software Analysis, and Product Acquisitions Advising. Each of these jobs will be described in following paragraphs to help define this task in an orderly manner.

Within the laboratory of SAIC's Laser Phase Conjugation experiment, is a MicroVax 3/GPX known on the Materials Lab Network as Spock. This MicroVax is crucial to the experiment because of its speed in acquiring data from instruments through an IEEE/GPIB Board. This system requires for security purposes that each user must have an identification phrase and a password. Thus all new users must be entered into the computer authorization program to give them a new account. This is one of the jobs of System Manager. Others include Routine Security Checks, Weekly Backups of the Hard Drive, Maintenance and Recovery from Drive Errors of System Crashes, and Installation of New Software and System Upgrades.

The MicroVax must have a set of rules to determine which instrument it is accessing, what type of data it wishes to retrieve from the device, and where to store the data acquired. Thus the job of Software Support and Design is crucial to the results of the experiment. This part of the overall Computer Support Task has included learning the Fortran Programming Language, Writing and Designing the Data Acquisitions Programs, Debugging such programs, and Organizing the Output Data Files. The current project is to take all the previously written programs and combine them into one. Each of these was designed to obtain very similar objectives but had minor differences based on the mathematical functions performed on the data.

The section of Materials Lab known as MLPJ uses several Macintosh Computers for office work and presentation development. The job of Hardware Maintenance at MLPJ mostly deals with these computers and their peripherals. Such work has included the installation of 73 new computers throughout the Materials Laboratory. This consisted of connecting monitors, installing memory and peripheral boards, installing software, and running system diagnostic programs. Another part of the Hardware Maintenance job has included the stringing of cable between these computers to setup a network. This Local Area Network (LAN) now allows multiple users to access the same printer, mail to be sent from one computer to all others, and the transfer of data and programs from one computer to another.

The job of Software Analysis and Product Acquisitions Advising are very similar. They both require that a program be thoroughly tested or researched before it is considered a valuable addition to the current computer resources. For each software package tested a report must be submitted to Mr. Martin or designated personnel on the purpose of the program, how well it achieves its goal, what are the restrictions of the program, who else has tested the product and include quotes from them, and your overall conclusion of the product.

CONCLUSION

I believe that the Task of Division Computer Support has given me a great opportunity to explore the expectations of my future career. It has been a wonderful learning experience that has improved my knowledge of current computer systems and has enhanced my ability to work with others and communicate within the scientific community. This task has allowed me to learn the Fortran Programming Language, become more familiar with VAX computers and their operating system (VMS), enhanced my knowledge of laser technology, and re-confirmed my belief in the scientific method as the correct procedure to obtain an experimental objective. I have enjoyed my work at MLPJ and hope to continue the tasks I am currently engaged in.

Thin Film Design Software

Task Order No. 12
Student Support Program

Cooper C. Faessler
University of Dayton

11 June 1991

Guiding Engineer
Mr. Walter Johnson
WL/MLPJ

ACKNOWLEDGMENTS

I would first like to thank Mr. Johnson for the opportunity to help in his project. He has given me an insight into a field I had not considered before, optics. I have gained much experience and knowledge working on this task and he is the one most responsible.

TASK ASSIGNMENT

This task consisted of a multitude of little jobs, mostly running programs. I have spent the majority of my time on a Macintosh II computer using programs such as Microsoft word, cricket graph, kaleidagraph, and power point. There are many other programs that I sometimes use. The main aspect of my task was thin film optical disks. My usual work consisted of taking these disks and running them through the Lambda 9 spectrometer. If I were not using a disk I was using a program called Megaline, which was an optical disk simulation program, to gather similar data. I would then take the data and transfer it to the apple computer. Once there, the data was turned into graphs. The reason I transferred the data was that the programs on the apple computer made the graphs more appealing and easier to put emphasis on what was needed than the original programs the data came from.

Once graphed, the information could be used as viewgraphs, which is what Mr. Johnson did. He would take the compiled information to his speeches and talks to explain to the audience what optical disks were, what the practical uses are for them, and how far along their progress is.

The main pieces of equipment I used were the Lambda 9 Spectrometer, the Apple II and a Mitsubishi MP286- and IBM compatible computer which has DOS. I would run the Megaline program on the MP286, the optical disks on the Lambda 9, and everything else was done on the Apple II.

The data was analyzed mainly by comparison and contrast. I would obtain graphs of samples I had run and then compare them with each other or I would run Megaline and compare those results with the actual sample.

I have learned a great deal about optical disks, something I had never heard of before this task. I have found a whole new dimension in my major of electrical engineering that I feel is very intriguing. What I can see from this job is that there is a great deal more to be done with these optical disks and this field in general, and I hope to have some part of it in my future.

Optical Molecules Software Development

Task Order No. 13
Student Support Program

Teresa Wilson
Wright State University

11 June 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Zbigniew Tokarski for his help and guidance during my contract. I would also like to thank Dr. Robert Crane, Mr. Tim Bunning and Dr. Tom Cooper for their support and advice.

TASK ASSIGNMENT

The object of my task was to assist in the programming and networking of computer hardware with the analytical instruments of the Material Laboratory's Biotechnology Lab, which will aide in the research and development of nonlinear optical material.

One of the main jobs during the task was to assist in the preparation of technical documents produced by the laboratory personnel. A Macintosh computer was used to accumulate data from different instruments and then change it into a format that could be easily understood and manipulated for later use in articles and publications. Many transformation applications were needed in order to transfer the different formats of incoming data into a master format which could produce a consistent set of output graphs and tables.

Another main job was to produce executable programs using the Pascal language. These programs take in different data values collected by the scientists and give output according to certain rules governing the calculations. The output is used by the scientists for the recognition and characterization of different optical materials.

I am very thankful for the opportunity to work in a research environment where I can put the skills I have learned to good use. I have learned about so many different aspects of computing that I have not encountered in my studies. I feel this learning experience will be very helpful together with the rest of my education and my future career.

IR Absorption and Photoconductivity of Semiconductor Materials

Task Order No. 14
Student Support Program

Ann Maack
University of Dayton

29 April 1991

Guiding Engineer
Gail Brown
WL/MLPO

ACKNOWLEDGMENTS

The people I would like to thank, who helped me with my task assignment are: Gail Brown, Dave Zelmon, Dave Fischer and Kevin Maute.

TASK ASSIGNMENT

During my task assignment I worked on gallium arsenide samples. My task was to determine the infrared optical properties of the samples by performing qualitative and quantitative measurements of defects, dopants and impurities in semiconductor electronic materials. The two types of optical measurements that are performed on semiconductor materials in MLPO are infrared (IR) absorption and photoconductivity. My particular task was assisting in the photoconductivity measurements. In doing this, I was aiding in the investigation of the defect/impurity levels in semi-insulating gallium arsenide, an important microwave device material.

In the course of my task, I mounted samples on a coldhead which was part of a CTI-Cryogenic closed cycle refrigeration system. A vacuum pump was used to reduce the atmospheric pressure in the system and then the interior temperature of the system was brought down to approximately 8 Kelvin. The coldhead system was placed in a Digilab FTS-20V Spectrometer and after the temperature was reduced and steady, I took data using a Digilab 3240 computer system. The data was collected at different temperatures to study the temperature dependence of the sample. One of the things I looked for in the spectra was changes in the intensity of the defect/impurity levels with respect to temperature or secondary illumination. In addition, a reference file was needed, with the reference file being a room temperature scan of the cesium iodide window through which the sample is viewed by the spectrometer. The reference file was used to correct the photoconductive response spectrum for the optical characteristics of the spectrometer.

In the process of doing my task, I learned how to prepare samples for mounting and how to properly mount the samples on the coldhead. I learned how to take data using the computer system and how to manipulate the data into a more presentable form. The types of samples I looked at were semi-insulating, n-type and p-type gallium arsenide. I collected and plotted spectral scans at different temperatures.

I was also the system administrator of both the Digilab 3200 and the Digilab 3240 computer systems. When necessary I removed files from the computer systems and stored these files on floppy disks and/or streamer tape. I installed software upgrades to the computer systems and performed system recoveries when problems arose on either computer system.

I trained Captain G. Seeger so that he would be able to perform my duties on the days I was not working and so that he would be able to take over for me after I leave the SOCHE program. I developed a guide that listed specific duties I performed and how to continue them in a consistent manner.

This year I completed the construction of an optical light pipe between the Digilab FTS-60V spectrometer and a superconductive magnet dewar. Using the data I collected with this system, I finished my thesis. With the presentation of my thesis to Dr. Perry Yaney, Dr. Roger Becker and Dr. Gordon Little (as well as those that attended the discussion), I completed the Electro-Optics Masters Program. With their help, I was able to write my thesis in a form that was consistent with those presented by other students in this program. Their patience and inspiration was greatly needed and appreciated.

I would like to thank Wanda Vogler and Gail Brown for the opportunity that allowed me to receive my masters degree and for all the help I received from those at SOCHE and at the base. I shall miss everyone that I have been associated with over the past few years.

Organic Matrix Composites Analysis

Task Order No. 15
Student Support Program

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19 December 1990

Guiding Engineer
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ACKNOWLEDGMENTS

I would like to thank the following University of Dayton Research Institute on-site personnel: Ron Cornwall, Bill Price, Bill Ragland and Brian Rice. I would also like to thank the following WL/MLBC personnel: Capt. Dave Curliss. They contributed help and instruction throughout this investigation.

TASK ASSIGNMENT

This investigation of the kinetics of resin systems used in composites was undertaken to find out the best possible cure cycle which can be used to create a composites. Our goal was to be able to analyze the various resins for their kinetics by looking at changes in their peaks under ultraviolet spectroscopy (UV) at various states of cure. The results from the ultraviolet spectroscopy showed that different resins when cured in composites had different types of cures which should be employed to yield a more completely cured composite. With this knowledge, better cure cycles can be employed to create a better composite.

The experimental portion of the experiment was divided into three phases, 1) Resin Specimen Fabrication, 2) Ultraviolet Spectroscopy, and 3) Spectral Analysis.

1. Resin Specimen Fabrication

Resin was extracted from commercial prepreg by use of acetone and then the solvent was allowed to vaporize off of the extracted resin for a day. Resin samples were then placed on glass slides and exposed to a vacuum to rid the resin of gas. After the samples were degassed, they were cured at various temperatures for different lengths of time.

2. Ultraviolet Spectroscopy

The specimen samples which were created at different times and temperatures were then analyzed by ultraviolet spectroscopy for any changes in their characteristic peaks. All of the peaks recorded were assigned a wave number of the use of peak pick which is incorporated into the Perk and Elmer UV/Vis interfaced computer. The various peaks were then compared with the peaks in different samples which were cured for a longer time. All of the samples peaks which were compared to others were cured at the same temperature, but the cure times were different.

3. Spectral Analysis

The ultraviolet spectral analysis was done on the different resin samples. The resulting spectra were then analyzed and the peaks wave numbers were identified.

Various resins were analyzed for their kinetics by looking at changes in their peaks under ultraviolet spectroscopy at various states of cure. This showed that different resins, when cured in composites, had different types of times and temperature which should be employed in their cure cycles to yield a more completely cured composite. With this knowledge, better cure cycles can be employed to create a better composite. Ongoing research will be investigating other resins for similar properties.

Organic Matrix Composites

Task Order No. 15a
Student Support Program

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15 June 1990

Guiding Engineer
Mr. Tobey Cordell
WL/MLBC

ACKNOWLEDGMENTS

The author would first like to sincerely thank Ms. Frances Abrams (Wright-Patterson Air Force Base) for her help, advice, and patience in teaching the necessary laboratory procedures for performing this task. She has demonstrated her expert scientific/technical knowledge of organic composites and brought forth new insights that, the author believes will be very beneficial in her future engineering endeavors. Secondly, the author wishes to acknowledge Mr. Ronald Cornwell and Mr. William Ragland (both of University of Dayton Research Institute) for all of their laboratory skills assistance. Lastly, the author wants to give much gratitude and thanks to Mr. Tobey Cordell for making this task assignment and previous task assignments possible.

TASK ASSIGNMENT

Organic composites have always had a problem of warpage called spring-back during processing (curing). Warpage can be attributed to the strain that the composites undergo upon curing due to the anisotropy of the thermal expansion in the composite layers. The objective of this project was to minimize this spring-back effect in graphite/epoxy/kevlar composites by manipulating both the gelation (solidification) and curing (processing or cross-linking) temperatures of the cure cycle and by using a non-uniform orientational arrangement for the lay-up.

The graphite/epoxy/kevlar composites utilized in this task were laid up of kevlar tape 1734, graphite woven 133/134 (weave-graphite cloth), and graphite tape 1634, and contain 47 plies with various orientations and thicknesses. The stacked laminates were wrapped around a black-pipe-steel-cylindrical mold (approximately 5.6 inches in diameter) and then cured on the mold in an autoclave. Three different cure cycles were employed to compare the warpage effects; i.e., to determine which cycle produced the least amount of spring-back.

This task was based on work previously performed that used the same composites, but a different cure cycle from the cycles run. The cure cycle that was run before this task is a control or standard cure cycle for graphite/epoxy composites, where the gelation and cure temperatures are each 350 degrees F. It yielded a spring-back of about 0.108 inch. Thus, the goal of this project was to reduce this 0.108 inch warpage by altering the control cure cycle.

As stated above, three different cure cycles were applied to the graphite/epoxy/kevlar composites using an autoclave. The first cycle had both gelation and curing temperatures of about 250 degrees F. Cure cycle 2 was a post cure of cure cycle 1. During the post cure, the mold was not used and the curing temperature was approximately 350 degrees F. The third cure cycle had gelation and curing temperatures of around 250 degrees F and 350 degrees F, respectively. The latter cure cycle employed the steel mold for the entire cycle. A dielectric measurement device with sensors, where the sensors were placed in the laminates during each cycle, were used to (1) detect when gelation (vitrification) had occurred and (2) determine the time required for both gelation and curing. The dielectric measurement device was also connected to a compute containing a stand-alone computer program, Qualitative Process Analysis (QPA), which converted the conductivities from the device to temperature readings.

Orientation is a major contributor to anisotropy in thermal expansion and, consequently, causes process induced stresses. The lay-up arrangement for this project contained several ninety-degree plies near the top (opposite of mold side) and the center and bottom portions of the laminates were fairly balanced in orientations. Thus, within those 90 degree plies, the orientation of the filaments (graphite) were to be taken as ninety degrees to the laminate and the matrix (epoxy resin) effects dominated in the zero degree direction of the laminate. The thermal expansion coefficient of the epoxy resin is greater than the thermal expansion coefficients of graphite and kevlar, and, because of this, expansion probably occurred in the matrix or, in other words, the zero-degree direction of the 90 degree plies. This type of warpage is known as outward spring-back and is characterized by the sides of the composite warping outward (diameter increases on sides). Inward or natural composite spring-back, on the other hand, always occurs in processing composites and is where the composite's sides contract inwards (diameter decreases). Inward spring-back was the type of warpage exhibited by the composite from the previous cure cycle already performed prior to this task. It was believed, then, that the spring-back was to be reduced because of (1) this task's lay-up arrangement (probably caused outward spring-back plus the natural composite or inward spring-back and (2) the altering cure cycles.

Lastly, it was to be determined from this project whether warpage can or cannot be reduced by manipulating the control cure cycle. If the spring-back was constant with varying gelation and/or curing temperatures, then it was path independent and cannot be reduced. On the other hand, the strain was path dependent if it could be altered by changing the cure cycle.

RESULTS/DISCUSSION/CONCLUSIONS

Cure cycles 1, 2 (post cure), and 3 reduced the spring-back in the organic composites of this experiment. The warpage decreased from 0.108" (inward) for the control cure cycle to 0.079" (inward) for cure cycle 3 to 0.019" (outward) for cure cycles 1 and 2 (See Attachment for Spring-back Calculations). The composite from cure cycles 1 and 2 had the greatest reduction in spring-back. It has been hypothesized by the author that this can be attributed to the post cure (cure cycle 2). The post cure cycle performed as an annealing process, where the process induced stresses were relieved. This stress relief caused a slight sagging in the top portion of the laminate, since there was no mold used in the post cure to retain the original top dimensions. To compensate for this sagging effect, the composite incurred a net outward spring-back, where the sides of the laminate warped outward (outward spring-back). However, to prove this hypothesis, cure cycles 1 and 2 would have to be rerun and the warpage incurred compared to the -0.019" value.

This "Spring-back Effect of Graphite/Epoxy/Kevlar Composite Laminates" task has indicated that reduction in warpage (spring-back or strain) can be achieved by manipulating the control cure cycle, or, in other words, spring-back is path dependent.

Advanced Composites

Task Order No. 15b
Student Support Program

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15 May 1991

Guiding Engineer
Capt. David Curliss
WL/MLBC

ACKNOWLEDGMENTS

I would like to thank WL/MLB personnel, Capt. Dave Curliss, as well as, the following University of Dayton Research Institute on-site personnel; Marlene Hougz, Brian Rice, Jim Lute, Bill Price and Ron Cornwall for their timely and helpful instruction and guidance throughout this investigation.

TASK ASSIGNMENT

This investigation involved the analysis and characterization of a BASF NARMCO bismaleimide resin; 5250-4 using infrared and UV/Vis spectrophotometers. The analysis involved locating any significant peak changes in the UV/Vis spectra, correlate them to structural changes in the resin and compare them to non-changing peaks as a possible measure of degree of cure. The characterization involved the study of infrared spectra for identifiable peaks and comparison of both UV/Vis and infrared spectra to existing spectra for similar bismaleimide resins.

Samples used for analysis were made using resin extracted from prepreg. For extraction, approximately 8" by 8" strips of prepreg were cut from a prepreg roll and placed over an aluminum pan. Acetone poured over these strips separated the carbon fibers from the resin, which collected in liquid form in the pan. The resin was further dissolved by the addition of 50-100 mls of acetone to the pan which was set overnight to allow the acetone to evaporate. The carbon fibers were heated in a 100^{o} F oven overnight then thrown away. Approximately two tablespoons of the resin is then dissolved in a minimum amount of acetone (approx. 10mls) and placed dropwise (approx. 7 drops) onto 2" by 2" squares of previously coated kapton. The acetone is evaporated then the application is replaced a second time.

The prepared samples are placed in a 50^{o} F vacuum oven and degased under 30" Hg vacuum for a minimum of 20 minutes or until no gas bubbles are visible. Once degased, the oven temperature is raised to 350^{o} F and samples are removed starting at 50 minutes, then every 5 minutes after that up to 90 minutes. One sample is cured overnight to use as a comparison to a fully cured sample.

UV/Vis and infrared analysis was performed using a lambda 9 spectrophotometer and a Digilab "Bio-rad" FTS-7 spectrophotometer, respectively. UV/Vis spectra plotted absorbance vs. wavelength with a range of 185nm-2600nm. Infrared spectra plotted percent transmittance vs. wavenumbers (cm^{-1}) with a range of $500cm^{-1}$ - $2200cm^{-1}$.

UV/Vis spectra were obtained for three repeated series of samples run at 350^{o} F. Peak changes were somewhat reproducible, but were complicated by differing sample thickness and a varying baseline due to increases in general sample absorbance corresponding to the darkening of the resin over time. Two peaks were observed to undergo noticeable changes throughout the 50min-90 min cure cycle including peaks at 1834.2nm and 2060.1nm. Infra-red spectra were obtained for one series of samples and showed characteristic peaks for known components of 5250-4 including; C=O at 1719.2 cm^{-1}, CH_2CH_2 at 2927.1-2972.8cm^{-1}, free N-H bending at 1520.7 - 1519.7cm^{-1} and aromatic C=C stretching at 3100cm^{-1}.

Current analysis involves identification of a non-changing peak in the UV/Vis spectra with which to do a peak-ration study, as well as, attempts to identify significant characteristic UV/Vis peaks.

Mathematical compensation for a drifting baseline and methods to standardize sample thickness are necessary before a useful peak ratio analysis of the UV/Vis spectra of 5250-4 can be done. Such adjustments are currently being attempted. However, at this point, the spectroscopic analysis of 5250-4 demonstrates that changes in the resin during the cure cycle can be studied using a UV/Vis spectrophotometer and suggests a UV/Vis fiberoptics probe would be useful in such an analysis.

SPRING-BACK CALCULATIONS

OUTER DIAMETER OF CYLINDRICAL MOLD = 5.597"

(1) CONTROL CURING CYCLE - PROJECT'S BASIS

- On Mold
- Gel & Cure Temps of 350°F Each
- Spring-Back (Warpage) =Outer Dia Mold-Inner Dia Part
  =5.597"-5.489"=0.108" (Inward)

(2) CURING CYCLES 1 AND 2

CURE CYCLE 1	CURE CYCLE 2
- On Mold	- Free Standing Off Mold
- Gel & Cure Temps of 250°F Each	- Post Cure, Cure Temp of 350°F

- Spring-Back (Warpage)=5.597"-5.616"=-0.019" (Outward)

(3) CURING CYCLE 3

- On Mold
- Gel Temp of 250°F
- Cure Temp of 350°F
- Spring-Back (Warpage) =5.597"-5.518"-0.079" (Inward)

High Temperature Materials Modeling

Task Order No. 16
Student Support Program

Dave Diller
University of Dayton

31 August 1990

Guiding Engineer
Dennis Dimiduk
WRDC/MLLM

ACKNOWLEDGMENTS

I would like to thank Dennis Dimiduk, Dan Miracle, T.A. Parthasarthy, Chris Woodward, Wanda Vogler and Thersia Carter for this opportunity. Over the past year, they have given me a great deal of their time and effort and have shown me a lot of patience. Because of them, this task has been both enjoyable and educational.

TASK ASSIGNMENT

The task has been centered on the computer simulation of imperfections in various materials. The largest portion of this task was spent working with two programs received from Sandia National laboratories: Creator and Dyn52. Initially, Creator was designed to create a perfect crystal lattice. After debugging, routines were added to Creator which could create the following imperfections in a crystal lattice: stacking faults, anti-phase boundaries, and dislocation using both isotropic and anisotropic elastic solutions.

The second of these programs, Dyn52, is designed to allow the atoms in a lattice created by Creator to relax to their equilibrium positions. Using this program, moderate success was achieved simulating edge dislocations in nickel, screw dislocations in nickel, stacking faults in nickel, and anti-phase boundaries in nickel three aluminum.

Another portion of this task was spent developing graphics software to provide graphic display of some of the information, such as positions and energies, generated by Dyn52 and Creator. Most of the graphics programming was done using the GKS standard.

Most experience came from using the computers. The VMS operating system and the UNIX operating system, Fortran language, and the GKS standard for graphics programming were learned. Also, a very basic understanding of materials science especially as it pertains to dislocations and the computer simulation of dislocations, was achieved.

Advanced Composites

Task Order No. 18
Student Support Program

Karen Thompson
Wright State University

13 September 1991

Guiding Engineer
Ms. Frances Abrams
WL/MLBC

ACKNOWLEDGMENTS

I would like to thank the following people for their assistance in helping me complete my task assignment. Frances Abrams, Kris Kliner and Major Joe Hager were very instrumental in assisting me with both the theoretical and experimental aspects of the assignment. Kris Kliner, Charles Hill, Jim Lute, Ron Cornwell and Bill Ragland provided the hands on training necessary for the completion of this task assignment.

TASK ASSIGNMENT

The objective of this design project was to develop a methodology for the reduction of defects resulting from gas evolution of volatiles in cured phenolic composites. The applied pressure and bagging procedure of the current cure cycle (Materials Lab, WPAFB) were altered to reduce the vaporization of volatiles in the thermosetting matrix resin and in turn a reduction in defects. The primary goals of the assignment was to reduce the evolution of vapor volatiles and produce a void free composite (or one with minimal voids) with good mechanical properties. Upon the completion of the task assignment, the autoclave cured carbon/phenolic composites will be carbonized to produce carbon/carbon composites (beyond the scope of the assignment).

Phenol, methanol and water are the primary sources of gas evolution in the carbon/phenolic system. A pressure-temperature diagram was constructed to determine the vapor-liquid transition curves for each volatile, and in turn, the magnitude of applied pressure necessary to prevent vaporization. Preliminary tests were performed on the neat resin and prepreg to determine if frothing occurs (no frothing was considered to indicate gas evolution had been minimized). Composites were cured in the autoclave and evaluated for defects.

The Macintosh II computer was used to perform the pressure calculations for a given temperature (using the clausius-clapeyron equation) to plot of the vapor-liquid transition curves for phenol, methanol and water. The preliminary tests were performed on the neat resin and prepreg in the Parr pressure reactor. The carbon/phenolic composites were cured at 350 degrees F in an autoclave (two at 200 psi and two at 100 psi). The Olympus microscope was used to view specimens taken from each composites and to make photomicrographs of each specimen.

Each composite was analyzed to determine if the defects had actually been reduced. The analyses consisted of visual (photomicrographs) and physical detection (acid digestion, thickness and weight measurements) of defects. While the severity and distribution of the defects (delaminations, voids or cracks) were determined by ultrasonic C-scan analysis. These analyses were used to evaluate the overall success of the project.

The combined results of the evaluation tests indicate that no void free composites were produced by the method used in this design project. The experimental data is inconclusive as to the origin of the defects, and therefore the occurrence of gas evolution is uncertain. The no-bag cure method employed for the composites did not provide enough compaction pressure. An 8x8 inch laminate would require more than 940 pounds of mass to achieve a compaction pressure of 14.7 psi. The gas evolution theory could still possibly be used to produce a void free laminate, but a different bagging procedure would have to be employed to allow better compaction of the composite.

Plasma Modification of OP Films

Task Order No. 19
Student Support Program

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10 June 1991

Guiding Engineer
Capt. David Curliss
WL/MLB

ACKNOWLEDGMENTS

I would like to thank the following persons for their guidance and support in the execution of this study: Capt. David Curliss, William Ragland, Bill Price, Gwen Steele and John Russell. Without their assistance I would not have been able to finish this task in a timely manner.

TASK ASSIGNMENT

This task has been divided into three different projects, these being: (1) the study of short-term plasma treatment of advanced composite materials, particularly OP films, (2) measuring the specific gravity of several composite materials, and (3) examining the sensitivity of several advanced composite materials exposure to military jet fuel, JP-4.

EXPERIMENTAL PROCEDURES AND RESULTS

I. Plasma Treatment of Modern Composites (OP Films)

The main objective of this research was to examine how to utilize several different gas plasmas to etch composite specimens. Etching cleans the specimens surface of microscopic impurities, namely dust, that obscure the viewing of the specimen under an electron microscope. An improved method of cleaning the specimens surface was desired.

Research into several recent articles and text determined that the best cleaning plasmas would be obtained using either Argon or Nitrogen gases, with an etching time varying from 15 minutes up to an hour. Unfortunately, the labs plasma generator could not sustain the required vacuum thus halting any further progress on this project. At this time, the plasma generator is under repair. Future work on this project may take place during July of 1991.

II. Specific Gravity Measurements

The purpose of this study was to measure the changes of the specific gravity for many advanced composite materials under varying temperatures and pressures. These changes in specific gravity are of particular interest to the Air Force in their pursuit to develop stronger and more flexible materials to be used in aircraft research and design.

The device used to measure the specific gravity changes, (known as a dilatometer), was purchased and installed by the materials lab. Numerous calibration runs were executed in order to obtain proper instrumentation of the dilatometer. This device is currently being used to measure the specific gravity of common composite materials, namely APC2 and HTA.

III. JP-4 Exposure on Advanced Composites

The sensitivity of two advanced composite materials, (APC2 and HTA), exposure to military jet fuel, JP-4, was studied. This was a carry over study from last year's task. The previous objective was to investigate how commonly used plastics would be affected after continual exposure to jet-fuel. This study focussed on examining the materials for fuel absorption and any significant changes in their mechanical properties. The present study is being conducted in an attempt to reproduce the previous study's results. At the present time, the materials have been processed into (+/-45)2s laminates and cut into standard geometry coupons. The coupons were exposed to JP-4 @ 180F for a total of 1680 hours and gained the predicted amount of fuel, less than 1% weight gain. The coupons are presently waiting to be mechanically tested.

CONCLUSIONS

Further work will be conducted on measuring the specific gravity changes for various advanced composite materials in order to produce enough results for a comparison study. The jet fuel soaked coupons are awaiting mechanical testing. Upon testing completion, the results will be compared to those obtained from the previous study. At the present time, any further progress on the plasma treatment on composites is dependent on repairing the plasma generators vacuum system.

Optically Active Molecules

Task Order No. 20
Student Support Program

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17 June 1991

Guiding Engineer
Dr. Robert Crane
WRDC/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Robert Crane and Dr. Tokarski for all their help and guidance while I worked on my task.

TASK ASSIGNMENT

The objective of this task was to determine the differences in the extent to which various dyes differ in absorbency of light due to the solvent that the dye is combined with and a comparison of these differences which may or may not be observed qualitatively.

Although the project has not yet been completed, the dyes that have been completed such as the 4-dimethylamino-4-nitro stilbene, for example, were used with chloroform and exposed to ultraviolet light for difference amounts of time and then compared to the combination of DMF and other solvents which cause the spectrum to shift in wavelength.

The conclusion so far is that certain substances are solvent dependent and that the wavelength at which the maximum absorbency occurs may be greatly affected by the solvent in which it is dissolved. This difference may be great enough to cause a complete color change. When a spectrum is taken, this can be seen as a definite shift in the wavelength at which the maximum occurs.

More dyes are to be observed in various solvents and studied as to which combinations produce the greatest and the least deviations in spectra.

Optically Active Molecules

Task Order No. 20a
Student Support Program

Elizabeth A. Reno
Wright State University

17 June 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

My deepest appreciation and thanks goes to Dr. Tokarski for all of his insight and guidance. The monthly reports and presentations were very helpful in conditioning myself for discussing my work in a business environment, which I will most likely find myself. I also appreciate the advice he was able to impart as to the particulars of conducting myself in an industrial setting. This is knowledge unobtainable in school, and I am grateful for it.

TASK ASSIGNMENT

My particular task was involved in studying the chromophore, Disperse Red 1. It was looked at as an optically active molecule suitable for attachment to both a cyclic and a linear siloxane compound which will maintain liquid crystalline properties desired. This material will then be formed into a thin film and tested with a Nd.YAG laser and an Argon laser for nonlinear optical properties. If these nonlinear optical properties exist, it is then possible to use the material as an optical filter for use in cockpit canopies and visors on the helmets of pilots.

The entire process was a four step synthesis beginning the benzoic acid and ending with the cholesteric chromophore. Each synthesis, except for the silanization, was completed within a day or two. However, it was found the silanization step took approximately one week. These reactions were monitored using Fourier Transform InfraRed spectroscopy. The presence or absence of peaks as bonds were broken and reformed was used as an indicator of the completion of the reaction.

After a reaction was completed, the product was recovered through solvent-nonsolvent precipitation. This product was then tested for purity using FTIR, UV-Vis spectroscopy, Thin Layer Chromotography and Elemental Analysis. The data showed that the first three synthesis steps went as expected. The silanization step was more difficult to determine. Skepticism arose because of the one week time needed to complete the reaction. Previous research done on similar compounds required about two days for completion. It was also discovered the linear siloxane compounds purchased were not the molecular weights first thought.

This made it difficult to know how many sites were substituted on the siloxane compound. Even with all this difficulty, the elemental analysis showed the silanization step to be somewhat successful.

More data needs to be collected on the final products and the possible use of High Pressure Liquid Chromotography to determine how many compounds are being synthesized in the last step will give a greater understanding to what exactly is being produced. At this point, Disperse Red 1 is promising.

Optically Active Molecules

Task Order No. 20b
Student Support Program

Laura McGivern
University of Dayton

17 June 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Zbigniew Tokarski for his time and effort and his expert advice in guiding me towards the specific goals of my task assignment. He was very helpful in explaining the meaning and the purpose of the work I was doing. He also helped me learn how to use much of the laboratory equipment. I would also like to thank all of the other student employees in the Biotechnology lab at Wright Laboratory who helped me achieve preliminary goals in preparation for my final goals of each major project.

TASK ASSIGNMENT

I ran a total of twenty-four different solubility tests with twelve different solvents and two different dyes. This was done in order to learn the degree of solubility of the two dyes in twelve different solvents. The results were then used by a fellow lab worker in running certain tests and reactions.

I also helped Dr. Tokarski synthesize four different substances to be used in a silanization reaction. I utilized basic chemical laboratory techniques such as refluxing, distillation, filtering and recrystallising.

The same method was used for all twenty-four solubility tests. A small amount of the dye, either Sudan IV, or Hoescht 33258, was dissolved in a particular solvent to the point of super-saturation. An even smaller amount of each solution, approximately one milliliter, was then extracted from a small vial with a syringe, injected into a smaller vial, placed under a hood until partially dry and then placed in a vacuum oven at a temperature between 40 and 50 degrees C until completely dry of the solvent. The vial was weighed alone, with the solvent-dye mixture, and with the dye alone to determine the solubility of the dye in each solvent in terms of grams of dye per gram of mixture.

The reactions done with Dr. Tokarski were done using three-necked round bottom flasks, distillation columns, heating mantles and magnetic stirrers. The necessary reactants were placed in a reaction flask, hooked-up to the condenser under a hood, and allowed to reflux for eight or forty-eight hours to ensure that the reaction went. The majority of the time spent on this project was spent on purification steps. I filtered each product warm to get rid of insoluble impurities, placed the filtrate in a refrigerator to cool, filtered it cool, evaporated half the solvent, cooled it and filtered again. The last several times two of the products were also purified by liquid/liquid extraction using a separatory funnel. All the products were analyzed using the FTIR spectrophotometer and comparing the spectra to standards.

It was learned that the Hoescht 33258 dye is nearly insoluble in all twelve solvents, that an acid/water solution takes an extremely long time to evaporate, and that the order of solubility of Sudan IV in the twelve solvents from most soluble to least soluble is as follows:
CHC13 > THF > DEC > Dioxane > Toluene > MeOH > Acetone > Basic H20 > DMF > Hexanes > Isopropanol, and Acid H20 is yet unknown due to its long evaporation time.

From the synthesis I learned that the longest, most involved process of synthesis is purification and not the reaction itself. So far three of the four reactions indicate at least forty to fifty percent yield of product, and the fourth product yield has yet to be determined.

Polysiloxane Liquid Crystals

Task Order No. 21
Student Support Program

Jennifer Daugherty
Wright State University

10 January 91

Guiding Engineer
Dr. Robert Crane
WRDC/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Zbigniew Tokarski, Dr. Thomas Cooper and Dr. Robert Crane for allowing me to work in the Biotechnology Laboratory at WPAFB.

TASK ASSIGNMENT

The work completed in this task can be summarized by the following description:

A study was conducted on the guest-host relationship between three organic molecules (B-carotene, hematoporphyrin "IX" and disperse red-1) and poly(v-benzyl-L-glutamate)(PBLG) for use as nonlinear optical materials. The major issue addressed was the incorporation of the guest into the host. Two methods of incorporation were used in this study: the physical-doping approach and the covalent attachment approach. In the physical-doping approach, the instability of the organic molecules to UV light and heat, and the orientation of PBLG in thin films were factors to be considered. The sensitivity of the three organic molecules was determined by UV/Visible/IR spectroscopy, thermal gravimetric analysis, gas chromatography, and differential scanning calorimetry analysis with respect to the amount of exposure time the samples may have received. Films of PBLG which were mechanically aligned by shear were found to have orientation factors as high as 1.1, with typical values between 0.6-0.9. Values of 0 and 1 correspond to no and complete orientation, respectively. Electric field alignment of PBLG in solution has also been achieved. In the covalent attachment approach, the reaction method and conditions, the percent substitution which could be achieved, and the effect on PBLG were all factors to be considered. The reaction method used in this study was a transesterification reaction between the hematoporphyrin or disperse red-1 nonlinear optical materials and the PBLG backbone. The mole percent substitution was determined by visible spectroscopy and the effect on PBLG was monitored by IR spectroscopy.

Lanthanide Complexes

Task Order No. 22
Student Support Program

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10 July 1991

Guiding Engineer
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WL/MLPJ

ACKNOWLEDGMENTS

Special thanks are extended to Dr. Thomas Cooper, Dr. Ruth Pachter and Dr. Zbig Tokarski for their technical support and patience throughout the year and especially for the opportunity afforded to me at the Materials Laboratory.

TASK ASSIGNMENT

The last year was spent under the primary guidance of Dr. Thomas Cooper of the Biotechnology Group at the WL/MLPJ branch of the Materials Laboratory. The goal of this branch is to develop laser hardened materials for national defense purposes. In particular, I have been assigned to the Peptide Laboratory where natural and synthetic proteins and peptides are investigated as laser hardened materials.

The projects I have been involved with deal with the synthesis, characterization, purification and modelling of peptides. The synthesis of peptides has been carried out using the Merrifield solid-phase method in conjunction with the FMOC amino protecting group chemistry. Using an Applied Chemtech automated synthesizer and FMOC chemistry, a seven amino acid peptide was constructed. The homogeneity of peptide synthesis product was determined by HPCE (high performance capillary electrophoresis) and HPLC (high performance liquid chromatography). These analytical tools distinguished three major products which were subsequently separated using preperative HPLC. Additional characterization of the separated peptide fractions was performed using a JASCO Circular Dichroism Spectrometer. The CD spectra obtained displayed typical random coil conformations using TFE (trifluoroethanol) as solvent.

Separately, computer modelling of a different peptide has been initiated under the guidance of Dr. Ruth Pachter. The initial structure was determined using the program Protean II available on the VAX computer system. Additional energy and dynamic calculations will be performed on Quanta software using an Iris workstation.

Future work will involve derivatizing the side chains of the heptamer peptide with a suitable dye. The appropriate dye will absorb light of a particular wavelength, thus inducing a conformational change. This change will be studied using the CD spectrometer at various temperatures and pH values.

During this year, state of the art equipment has been utilized to synthesize, purify and model peptides. Among the equipment used has been a peptide synthesizer, HPLC, HPCE, computers (VAX, Macintosh & IBM). Additionally, the use of UV/VIS, IR and CD spectrometers has been extensive. These skills are indispensable for the modern day peptide chemist who is interested in the relationship between the primary structure and three dimensional orientation of amino acids joined to form peptides and small proteins.

Laser Hardened Materials

Task Order No. 23
Student Support Program

David Flora
Wright State University

10 June 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to recognize the following people for their assistance with my research at WL/MLPJ: Dr. Greg Young (thesis advisor), Dr. Tom Cooper, Dr. L. V. Natarajan, Dr. Zibgnew Tokarski; fellow students: Teresa Wilson, Tim Bunning and Kelly Menster.

TASK ASSIGNMENT

Task assignment number 23 involves the synthesis and characterization of polyamino acids (peptides) for possible use as matrices for optically active compounds.

WORK TO DATE: In previous work our group focused on aligning NLO molecules by droping them into liquid crystal matricies. Recent work has focused on the direct attachment of NLO molecules to amino acid side chains of peptides.

To this end, various methods of peptide synthesis and characterization have been investigated. This has led to the purchase of an automated peptide synthesizer, and the synthesis of several peptides to be used in NLO attachment experiments.

RESEARCH: Synthesis: The method chosen to synthesize peptides is that developed by E. Atherton and R.C. Sheppard at Cambridge University, England. Their system is known as the Fmoc-Polyamide Continuous Flow Method.

It is based on an orthogonal protection scheme (to prevent side reactions among the various amino acid residues) using 9-Fluorenylmethoxycarbonyl as the base sensitive alpha amino blocking agent, and tert-butyl based side chain blocking groups. Some amino acids require more elaborate side chain protection, for more information see SOLID PHASE PEPTIDE SYNTHESIS a practical approach, by E. Atherton, and R.C. Sheppard, IRL Press, 1989. The tert-butyl based blocking groups are acid sensitive, and in combination with the Fmoc group, enables the preferential removal of Fmoc groups during synthesis. The result is a reduction of side reactions (deletion of an amino acid from the sequence) that occur during synthesis. The peptide bond formation is facilitated through the activation of the carbonyl carbon with pentafluorophenol and the catalyst 1-hydroxbenzotriazole.

The Polyamide is a polymeric support on which the peptide molecule is built. The advantage conferred by its use lies in the ease with which the growing peptide can be separated from synthesis reagents.

Characterization: The methods for amino acid, peptide, and protein characterization have been well established for many years, and are: (1) compositional amino acid analysis, (2) reversed-phase high pressure liquid chromatography (RP-HPLC), (3) ion-exchange chromatography, (4) gel filtration, and (5) amino acid sequence determination.

Compositional amino acid analysis provides data on the number of each residue incorporated into the peptide. This is compared to a predicted ratio to help determine the heterogeneity of product. Reversed Phase HPLC is another effective method of determining product heterogeneity as well as a method of purification. Differences as small as one amino acid may be detected under proper conditions. Ion-exchange chromatography separates components based on over all charge at a specific pH, and gel filtration separates components based on size (i.e. molecular weight). Sequence analysis allows one to specifically determine which amino acid residues are attached to one another (moving from the N to C-termini).

RESULTS: To date several synthesis have been completed. Specifically, KLKE, KLKLK, EEEEE, and HPEYFKNA.

Reactions were monitored by the Quantitative Kaiser (ninhydrin) color tests, and each acylation was terminated upon negative color production. Total acylation run time was generally one and a half hours.

Results of RP-HPLC are currently used to evaluate the homogeneity of the crude synthesis product. Compositional amino acid analysis and sequence analysis will be available shortly. This will allow detailed analysis of synthesis products.

The lab is still in the stage of determining which model peptides to synthesize, and optimizing conditions to that end. Once the peptides of interest are determined, and the conditions needed for their synthesis optimized, this lab will have the ability to examine various NLO molecules attached to various peptides.

Polyamino Acids

Task Order No. 24
Student Support Program

Kelly Menster
Wright State University

10 June 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I wish to thank Dr. Zbigniew Tokarski for his guidance in selecting a research topic and his help in giving focus on the specific goals of the project. I would also like to thank Mr. Tim Bunning whose knowledge and input has been a tremendous source of information and guidance.

TASK ASSIGNMENT

Various types of polymers have been tested for optical activity. Polyamino acids and polysiloxanes may be promising. The synthesis and characterization of these materials must therefore take place to uncover their significance. The project entails the synthesis and characterization of liquid crystalline polysiloxanes and comparison of these materials with cyclic siloxanes.

The liquid crystalline mesogens were synthesized using the DCCI coupling reaction. Hydroxbiphenyl or cholesterol is attached to 4-allyloxybenzoate in the presence of DCCI and DMAP (catalyst). The resulting liquid crystal is then attached to the siloxane backbone by a hydrosilation reaction. A ten percent excess of the liquid crystal is added to the siloxane using a platinum catalyst. The reaction is completed when the Si-H band in the infrared spectrum disappears. This is the most common way of monitoring these reactions. However, when the reaction is carried out using a cyclic siloxane this bank does not appear in the IR spectrum. Consequently, another method of monitoring this reaction was needed. The reaction was found to be over when the absorbance ratios of the 930cm-1 bank to the 1730cm-1 band leveled off.

Products were characterized using DSC to determine phase transition temperatures. There was little difference in thermal properties between the linear polysiloxanes and their cyclic analogs. These products were also analyzed by hot stage microscopy to determine the type of liquid crystalline phases present (smectic, nematic, cholesteric).

The liquid crystalline polysiloxanes seem to exhibit the same behavior in DSC and microscopic analysis. However, the polymer is much more viscous than the ring structure. The liquid crystalline polymers may also be promising for the separation of PAH isomers when used as stationary phases in capillary columns for gas chromatography.

Electrical and Magnetic Properties of High Temperature Superconductors

Task Order No. 25
Student Support Program

Jon T. Keller
Wright State University

10 June 1991

Guiding Engineer
Mr. Tim Peterson
WL/MLPO

ACKNOWLEDGMENTS

Special thanks and acknowledgment to Tim Peterson for his guidance and instruction provided during the last year.

TASK ASSIGNMENT

During this task, several variances on the same theme were performed. The basic idea was to measure different variables of a material, and determine its superconductivity. We start with the sample, which is usually a thin film, but sometimes we test bulk samples as well. The thin films we work with are produced here, but in a different lab, and are usually on the order of microns thick, a centimeter long, and about half that wide. We take this film and place contacts on it, which allow a current source and a voltage measuring device to be attached to the film. The film is then immersed in a system where liquid helium is circulated, thus allowing the sample to be cooled down to as low as 4 Kelvin or -273 degrees Celsius. Current is put thru the sample and the voltage across the voltage contacts is measured as the temperature is slowly lowered. At higher temperatures, the sample acts as a normally resistive material, and this produces a voltage drop across the contacts (since voltage and resistance are directly related). As the temperature decreases, the voltage will begin to decrease, until at some point it is approximately zero, i.e. the resistance is zero and the sample has just gone superconductive, meaning current flows through the sample totally unhindered. The temperature at which that particular sample goes superconductive is called its critical temperature, and this is one of the values we are looking for in testing these thin film samples. The other value we are looking at is called the critical current density. We test to see how much current we can put through the sample at various temperatures and still have the sample be superconductive. At temperature far below the critical temperature, the critical current density is very high, but as we move towards the critical temperature, the closer to that temperature we get, the less current the sample can handle before breaking down and becoming non-superconductive.

From this macroscopic information, i.e. critical current density and critical temperature, we hope to learn something of the microscopic structure of the superconductive materials. Several papers have been published by other labs, describing their theories and the experimental results which supposedly support these theories, and one of the goals of this task is to verify the validity of the experiments performed by other experiments, and thereby, verify their corresponding theories, or develop our own theories. Several attempts have been made at this verification, but data consistency has been a problem lately. Currently, part of my job is to aid in the standardization and dehumanization of the data and subsequent conclusions. All instruments used in the data acquisition, such as voltmeters and ammeters, are represented by virtual instruments on the Macintosh computer, and all subsequent data manipulation and analyzation are also performed on the Macintosh. The virtual instruments can be manipulated just like the real thing, and since the computer and the instruments are connected, this allows the computer to control the instruments. Another goal of the program is to improve the quality and ability of the various films. Although I am not directly involved in making the samples themselves, the tests I perform enable the sample makers to know if what they made is good or bad, and if their steps towards improvement are taking them forward or backwards.

Electromagnetic Materials Experiment Support

Task Order No. 26
Student Support Program

Daniel J. Murphy
University of Dayton

10 June 1991

Guiding Engineer
Mr. Thomas Kensky
WL/MLPO

ACKNOWLEDGMENTS

Special thanks and acknowledgment to Tom Kensky and Ron Perrin for the guidance and instruction they provided during the last year; it has proven to be invaluable toward my career development.

TASK ASSIGNMENT

The general objective of this task was to provide support in the design of electronic circuitry for use by in-house researchers. In addition, software design was also required for the purpose of data acquisition.

Specifically, the work done consisted of a myriad of tasks. These tasks are described more thoroughly in the following section.

A large percentage of the summer hours were spent on the design and testing of an "automatic" crystal-growth system. This was delegated to me to provide a means for the scientist to grow crystalline substances in a relatively automated manner - i.e. simply add the substance for growth into a beaker, enter some needed data into the computer, and push a button. The system would then, over a two week period "grow" a crystal made of the added substance. These crystals could be made from any of a wide variety of substances to be used to test the material properties. These could include the ability of the crystal to perform "frequency doubling". Frequency doubling is the ability of the substance to output a light of twice the frequency of the light which was input. Often times this light is not visible upon entering the crystal, but is visible upon exiting it. The system resulted in the use of a Z-100 PC for inputing of data and for the control applications of the system. Also utilized were numerous hardware technologies, such as 8-bit precision analog-to-digital conversion, thermal sensing, motor drive circuitry, and the use of solenoid/valves for automatic fill-up of the crystal bath. This project served as no other has, in opening-up new technologies and ideas to my knowledge base. As an engineer I feel this project was an invaluable vehicle in exposing me to essential and practical technologies for future needs.

The other project which consumed much of my work hours, was the completion of a 68000 microprocessor-based microcomputer. This microcomputer was assigned to me last spring, and it was my responsibility to design, test, construct and implement it. The design stage encompassed several months work on the Macintosh's Douglas CAD/CAM, and paper and pencils. About July of last summer the "breadboard" stage began; this required another month of hand-wiring the design onto a temporary "testing" board. After the "bugs" were worked out, and all aspects of the system design were tested, the project then moved into the final stage-construction. A simple software monitor was written in menu format to provide the user easy use. At present, the system is not being utilized for any specific function, but was developed as an open-ended unit so as to be functional for a variety of applications. These could include software development, user terminal for RS-232 communications, or a controller for practically any application requiring serial/parallel ports or extensive RAM space.

Also, throughout the year many short term projects, repairs, computer network maintenance and installations (cable routing) were performed, which consumed many hours when taken together. These were typically linked to assisting a scientist in a specific want or need in order for him to fulfill his experiment or goal. Often times these "little" tasks were a surprise in what they sometimes offered in means of new ideas and/or remedial knowledges. For example, when a scientist needed a simple circuit designed for amplifying a voltage signal, it gave me a chance to re-discover and strengthen my knowledge of operational amplifiers and the design of circuits utilizing them.

The last year while employed with SOCHE via MLPO, has given me numerous opportunities to investigate new engineering ideas, to further my knowledge level as applied to both my profession and as a co-worker, and to be better prepared for a successful career in engineering. I feel the Student Support Program provides an excellent service to not only the students who are employed, but also to the employer, namely Wright Patterson Air Force Base, due to the aid the students provide the scientists and engineers. And perhaps most of all, the program is good for the community and nation, in order to keep up with technology and the world.

Computer Application In Electromagnetic Materials Research

Task Order No. 27
Student Support Program

Richard A. Sorg
University of Dayton

10 June 1991

Guiding Engineer
Mr. Ron Perrin
WL/MLPO

ACKNOWLEDGMENTS

I would like to thank both Ron Perrin and Tom Kensky for their help and guidance over the past year. Both were very helpful and knowledgeable on many subjects which allowed me the chance to learn more than I would have thought possible while working at the Materials Laboratory.

TASK ASSIGNMENT

I was tasked to support research being conducted in electronic, electromagnetic, and optical materials. The focus of my work was centered around computer support. Most of the experiments being conducted in the Materials Laboratory are controlled in some form with the use of computers. The data taken from these experiments is also analyzed and interpreted with the help of computers. A large part of my work experience was centered around developing the software needed to control these experiments and analyze data. One project that I was actively involved in was to write part of a program in Fortran that would extract data about the mobility content of a super conducting sample material. The program generated quite a bit of data and I was responsible for writing another program to interpret this data and make it more meaningful to the researchers. Most of the software written was done in Fortran using the VAX computer system. Other software projects included modifying an existing d base program and writing software in a graphics package call RPL. RPL was used to generate graphical plots of data taken from some of the experiments being conducted.

I was also involved in developing and constructing hardware that was used by in-house researchers. In order to carry out their work, the scientists and physicists often have a need for custom built electronic devices. One such device was needed to control a stirring motor and the water level of a tank as part of a crystal growth experiment.

Other tasks included developing graphs, flow charts and spread sheets on the Macintosh computer system. Numerous time was also spent stringing cables as part of the installation of an apple talk local area network and ensuring computer communication was always up when offices and computers were moved to difference rooms.

My time spent working at the Materials Laboratory was a very valuable experience in obtaining hands-on training that I was able to incorporate with my studies at the University of Dayton.

Computer Applications in Electromagnetic Materials

Task Order No. 27a
Student Support Program

Larry Brunson
Wright State University

10 June 1991

Guiding Engineer
Mr. Ron Perrin
WL/MLPO

ACKNOWLEDGMENTS

I would like to thank Mr. Ron Perrin especially for recommending me for this position and for entrusting certain tasks to me. Thanks also to Mr. Tom Kensky who took time to offer help and answer question.

TASK ASSIGNMENT

Except for a few minor jobs as soldering computer plugs onto cables, stringing new computer cable overhead, and performing routine maintenance to a laser printer, the task was largely given to two projects. The first was to find a way to send intelligible data from a Varian DS-15 data station out its serial port. This computer collects data from a spectrophotometer and stores it on disks formatted incompatible with either IBM or Macintosh. For this reason to use the data for plotting or other manipulations it must first be transferred to an IBM format, hence the need to send the disk's contents out through RS-232.

This sounds like a simple task, except that the data is coded on the disks and there's no access to the decoding program used by the Varian that allows the data to be viewed on its screen, that by making a few simple changes, one might be able to send this intelligible data out the serial port. (Among the given Varian programs there was one to send the disks' contents out through RS-232, but the gibberish seen on the receiving terminal bore clear witness to the fact that somehow the data had to be decoded first.)

Work was started on this project May 1, by first becoming familiar with the machine and the Basic programming language. Three work days later with no answer in sight, Mr. Perrin gave me another week to try. The next work day the problem was solved with the Varian program; it was just a matter of altering one keystroke in the program's execution.

The second project from Mr. Perrin was to become familiar with the Douglas CAD/CAM package in order that to use it to design some printed circuit boards for one of the scientists. It was not possible to finish this before the contract expired.

As a result of this task I have noticeably grown in computer knowledge, not only because of the two main projects, but also because I had the opportunity to observe Ron disassemble a computer and pinpoint a bad chip causing it to send gibberish to the printer. I look forward to the coming work year and the learning that college can't provide.

Electronic Failure Analysis Support

Task Order No. 28
Student Support Program

Mark Rose
Wright State Univestity

15 July 1991

Guiding Engineer
Mr. George Slenski
WRDC/MLSA

ACKNOWLEDGMENTS

I wish to express my thanks and appreciation to those individuals for whom I worked, to those with whom I worked, and to all who helped me to do my job: Mr. Ron Williams, Mr. George Slenski, Mr. Eddie White, Mr. Mike Marchese, Mr. Dale Hart, Mr. Rick Reibel and Mr. John Ziegenhagen.

TASK ASSIGNMENT

The job of electronic failure analysis support is a wide ranging one. It consists of aiding the failure analysis engineer in determining the cause of failure for an electronic system or component. The performance of this mission requires the application of many skills. Additionally, the supporting student must be capable of working with minimal guidance in a methodical and meticulous manner. In particular, my first project, a printed wiring board assembly, demonstrated the need for all of these skills and traits.

A printed wiring board assembly had been referred to the electronic failure analysis group for determination of the cause of its failure and necessary corrective actions. The assembly was part of an aircraft communication system, and while it did not pose an immediate danger to pilots, it meant the aircraft could not operate as it should. As the problem seemed to be common to a large number of the same type of assemblies, affecting possibly hundreds of aircraft, a solution was urgently desired.

In consultation with engineers and technicians, a process of failure analysis was agreed upon in advance. The first step in the process was to review the preliminary information provided with the assembly. It appeared that failure was caused by open circuits resulting from failed solder joints. Photographs of the received printed wiring board (PWB) were obtained in conjunction with a thorough visual inspection using both the unaided eye and optical microscopes. Next, real-time x-ray radiographs were taken, followed by electrical testing of the PWB to confirm and document the extent of the failure. This initial process involved only non-destructive evaluation of the PWB.

At this point, it was decided that several representative solder joints should be obtained from the PWB and cross-sectioned. Inspection of the cross-sectioned solder joints with optical microscopes and scanning electron microscopes at high magnification revealed two major failure modes: cracks and voids in the solder, and separation of solder from copper barrels in plated-through holes. Based upon the available evidence, and in consultation with a metallurgical expert, several design and manufacturing flaws were hypothesized, including: excessive thermal mass due to two copper ground planes, inadequate pre-tinning of component leads leading to gold contamination in the solder joints, and insufficient etchback of the PWB laminate prior to application of the copper plating.

X-ray elemental analysis was able to confirm the presence of gold in the solder joints in unacceptable quantities. A visit to the manufacturer of the PWB by project engineers confirmed the lack of etchback and the lack of quality control over the pre-tinning process. The presence of gold contamination was determined to be the single greatest contributing factor to the failure of the PWBs. Gold causes solder to become brittle, leading to cracking and giving the solder a dull, uneven finish.

Also, the excessive thermal mass of the ground planes required the application of too much heat, contributing to the poor appearance of the solder and to the separation of the copper barrels from the solder. The separation was further compounded by the lack of etchback prior to plating with copper. The final recommendations to the manufacturer included: redesigning the PWB to eliminate the two ground planes, changing the pre-tinning solder baths more frequently, monitoring the solder baths closely for contamination, and etching back the PWB laminate prior to application of the copper plating.

A great deal of technical information was learned about soldering, manufacturing processes, operation of various equipments, and the failure analysis process. However, some lessons learned were more general in their application. For example, it is extremely important to document every step of the failure analysis process in detail as each step is performed. Sometimes, a seemingly trivial observation may prove to be important later on. Also, one must not jump to conclusions during an investigation. Premature conclusions can lead an investigator to pursue phantom leads or overlook crucial evidence. In short, one must be thorough, observant, and open-minded while conducting a failure analysis investigation.

Structure Failure Analysis Support

Task Order No. 28a
Student Support Program

Kuo-Chu Colin Huang
University of Cincinnati

15 July 1991

Guiding Engineer
Mr. Ron Williams
WL/MLSA

ACKNOWLEDGMENTS

I would like to take this opportunity to thank the following people for helping me throughout this quarter:

Technicians:	Engineers:
Andy Logue	Ron Williams
Tom Dusz	Larry Perkins
John Ziegenhagen	Michael Oliver
	Neal Ontko

TASK ASSIGNMENT

A corrosion project was handed to me after an initial proposal was made. The subject of this program is to compare the new developed alloy (2090) to the other (7075) in many standard tests before it is schedule to be used for military purpose. Both alloys are aluminum base. The 2090 alloy is being introduced into military aircraft because of its light weight (density).

The sheet samples are purchased from Alcoa and machined into required testing specimen sizes and some are anodized with special coating. All the tests are performed according to American Society for Testing Materials (ASTM). Test specimens are cleaned with degreaser and dimensioned and weighted before the beginning of experiments. Some samples need to be coated with special paint/primer and treatment. Several tests are time consuming and waiting to be done. At this time, two standard tests are in progress for a period of time: atmosphere test and alternating immersion.

The 45 degree angled rack was already built for atmosphere test. Specimens are fastened with nylon bolts and nuts and observed weekly for the next three months. So far, there are no signs of corrosion on specimens. By using the knowledge of siphon, equipment was designed for alternating immersion test with, two equally sized tanks filled with salt water and a timer to control the rate. The specimens are to remain in one of the tanks for at least 30 days. Weekly checks to maintain the fluid level are required. To date, the bare metals are showing more severe corrosion than the anodized metals. The 7075 bares are much worse than the 2090 bares.

Recorded data are generated using computer hardware and software. Also, photos are taken to verify the existence of project. The photos record the condition of specimens prior to testing, during the experiment and after experiment.

At this early stage, the 2090 alloy performs no worse than the 7075 in mechanical properties and corrosion resistant. However, conclusions can not be drawn until this experiment is concluded.

Characterization of Monomers and Polymers

Task Order No. 29
Student Support Program

David Carpenter
Wright State University

25 June 1991

Guiding Engineer
Dr. Fred E. Arnold
WL/MLBP

ACKNOWLEDGMENTS

I would like to thank everyone at the Materials Laboratory Branch for their friendliness and cooperation. Also, I would especially like to thank Dr. Fred Arnold and Dr. Thuy Dang whose guidance made this possible.

TASK ASSIGNMENT

My assignment at the polymer research branch was to maintain the laboratory equipment and supplies, and learn preliminary techniques for classifying monomers and polymers.

The first step was to learn the various types of glassware. There are hundreds of different kinds of glassware and each has its own purpose. I learned the names of them by experience, mostly by helping Dr. Thuy set up the apparatus for whatever duty that needed to be performed. These duties were running reactions, purifying monomers, distilling or drying compounds. In addition to knowing the glassware by name, I had to know how to clean them. Cleaning glassware was a major duty of mine, because it was a never ending process. Chemistry research dirties quite a lot of dishes and clean glassware is a must for reactions to perform correctly.

An ultrasonic bath was my biggest help. This device did most of the work that would normally require scrubbing by hand. It vibrates and heats the glassware which is also bathed in an ammonium water solution. Most glass can be cleaned this way, but not all. Funnels which have plates that separate compounds from solutions are the hardest to clean. They require soaking in a sulfuric acid and potassium dichromate mixture which sometimes needs to be repeated over and over before it is clean. Whatever the ultrasonic bath can't clean, good old-fashioned scrubbing with scouring powder will. Sometimes it is more cost effective to throw the glassware away than it is to clean it, because the solvents used to clean them costs more the glass itself. Acetone (the solvent which gives finger nail polish its scent) is the most used solvent. It is used to rinse all glassware after it is clean to prevent water spots. After a great deal of experience, I developed my own techniques to clean glass, which I felt was best.

The other part of my task order gradually started. I learned the different methods for classifying monomers and polymers after I became adjusted to my other duties. Melting points, viscosities and IR's were the three main classification methods I learned. Melting points are taken for most monomers (the building blocks of polymers) to see how pure they are. Usually the shorter the range of temperatures for which the compound melts, the purer it is. Experience tells you when a monomer should melt and when it is actually melting however, sometimes it is difficult to tell. IR's (infrared spectrum) are taken for unknown compounds. Certain elements and element groups appear as peaks on the IR graphic printout. This is a job done mostly by the computer, but the chemist can interpret the output by using books which help them identify the makeup of the compound. My job was to make the mixture for the computer to read.

Viscosities are measurements to determine how good a polymer is. Usually, the thicker the polymer is, the better. A liquid sample is placed in a special type of glass called a viscometer, which measures the time it takes for the liquid to fall from one mark to the next. Along with some mathematical calculations a viscosity can be measured. The higher the number the better. This is the high point of polymer research.

Characterization of Monomers and Polymers

Task Order No. 30
Student Support Program

Melissa M. Epperson
Wright State University

1 July 1991

Guiding Engineer
Dr. Fred Arnold
WL/MLBP

ACKNOWLEDGMENTS

I wish to thank Dr. L.S. Tan of the Materials Laboratory, Wright Patterson Air Force Base, who served as my guiding scientist. I also wish to thank Mrs. Sharon R. Simko of the University of Dayton Research Institute, who worked with me under the direction of Dr. Tan.

TASK ASSIGNMENT

The main component of my job was maintaining laboratory equipment, supplies and glassware. I washed glassware almost daily and used the building vacuum with special apparatus to clean the fritted filters and NMR tubes. When glassware would not come clean, I used the ultrasound bath and, sometimes, potentially hazardous solvents. The waste from these solvents requires special disposal, for which I was responsible. I had to complete government forms on the contents of the bottles of waste with their Environmental Protection Agency (EPA) codes and then properly label and deposit them in the proper location. I was also responsible for ordering our supplies through the University of Dayton Research Institute (UDRI) and the federal government. The UDRI orders required only contacting the companies for prices and completing a simple order form. The government forms are much more complicated and time-consuming. They require special government numbers and copies of the catalog pictures of the items being ordered. They also required I write justifications for all electrically-powered equipment. In addition, I kept records of our expenses for both UDRI and government orders.

Another big part of my job was monitoring organic reactions and doing preliminary characterization of the newly-created polymers. Organic (carbon-based) reactions commonly take hours or days to complete. When Dr. Tan left for meetings, I sometimes assisted by keeping chromatography tubes filled, and I helped Mrs. Simko by checking the Thin Layer Chromatography and doing reactions and filtrations when she had hand injuries. I frequently performed intrinsic viscosities, melting points and infrared spectroscopy (FTIR), which I did on both the old Beckman machine and the new BioRad computer. Although I performed these procedures, I do not have the training to analyze the results. Dr. Tan never expected me to interpret the results, do any research projects or use any equipment beyond the computers, MP, FTIR and dishwashing apparatus.

By maintaining the laboratory and doing characterization, I freed Dr. Tan to do his research. I also helped him in other ways. I frequently copied technical journal articles, and I once prepared a graphic display for his presentation at the American Chemical Society (ACS) conference in Atlanta, GA. I spent much time on the

Macintosh computer preparing slides of Dr. Tan's work for that conference and for his publications. Several of the charts that I created appeared in Polymer Preprints. I also kept records on characterization samples that needed to be sent to other labs. Anyone requiring the services of those labs brought their samples to me. I recorded and delivered the samples and, when they were done, I picked up and returned them.

I learned on this job that scientists do not suddenly make great discoveries: research is a long, tedious process. Many compounds are made, studied and discarded before any one shows real promise. Dr. Tan is possibly the most productive researcher in the Polymer Branch. I felt instrumental in the operation of my laboratory because I provided the organizational and technical support that allows Dr. Tan to devote most of his time to research.

High Temperature Superconductivity

Task Order No. 31
Student Support Program

Benjamin Smith
Wright State University

19 June 1991

Guiding Engineer
Mr. Tim Peterson
WL/MLPO

ACKNOWLEDGMENTS

Tim Peterson, my mentor, was extremely helpful and seemed to realize that I was more interested in why something happens and not just satisfied with knowing what buttons to push to retrieve the data.

TASK ASSIGNMENT

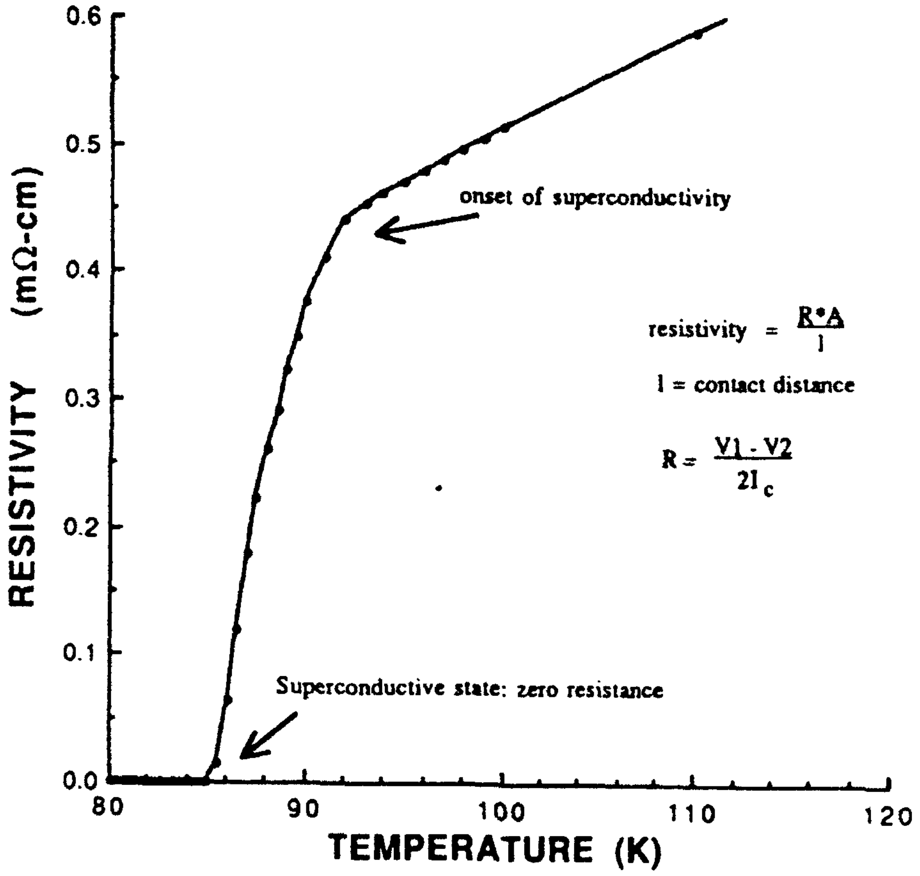
The mission of the Laser and Optical Materials Branch (MLPO) of the Materials Lab is to provide the Air Force with new materials. In support of the Air Force space and aircraft application needs, basic research and exploratory development work is performed in the areas of infrared detectors, nonlinear optical materials, microwave materials, and high temperature superconductor ceramics (HTSC). I am directly involved with the high temperature superconductivity activities.

The purpose of the Materials Laboratory HTSC activities is to maintain a presence within the field so that the Laboratory can utilize improvements in material growth and processing techniques. This will aid in development of the HTSC material technologies applicable to devices and systems which are necessary for the advancement of the Air Force capability.

The characterization techniques currently being used include AC magnetic susceptibility, resistivity versus temperature, critical current density and photoconductivity. My involvement in the HTSC activities is the characterization of bulk and thin film materials by determining resistivity and critical current density of a variety of HTSC samples from either in-house sources or contractors.

The type of samples that have been tested during the past term were small bulk materials and thin films. Samples of this type are tested at low currents(10^{-6} or 10^{-1} ampere range).
Because small test currents are used on samples with vanishing low resistances, the voltages measured are extremely low. System accuracy when measuring these low voltages is extremely important for dependable and repeatable results. Noise from electrical or magnetic fields can reduce the sensitivity of these measurements.

The low values of resistance require a four (4) wire measurement technique to eliminate lead resistance. Thermally induced voltages from the contacts can still cause errors in the measurements and these voltages must be accounted for. Beginning at low temperatures, approximately 70 K, a voltage measurement (V1) is taken with the current in a positive direction; the current is then reversed and another measurement (V2) is taken before thermal gradients change. The thermal voltages therefore cancel in the final calculation. Current is kept constant during the measurement of the resistance of the samples. Temperature is increased gradually to room temperature (300 K). The resistance (R), measured in Ω is a function of the material as well as of the sample size and shape. Volume resistivity (Ω-cm), however, if a function only of the material. It is valid to compare resistance between different samples as long as the sample shape and size are the same. Below is a familiar curve of a superconductive sample plotted resistivity versus temperature.



Gathering the data for resistivity measurements was long and tedious. Thanks to Ron Perrin and Tom Kensky of the engineering staff, the system is now automated with the use of a Macintosh II software system. More samples can now be analyzed with much better accuracy.

Critical current measurements can also be taken at a constant temperature, usually submerged in liquid nitrogen (77 K), voltages are measured at each value of current in the positive and negative directions. Again, this is to correct for the thermally induced voltages. The average of the two voltages, V1 and V2, is then computed. If the voltage drop is zero, the material is said to be in the superconductive state. Current then is increased gradually until a non-zero voltage drop across the sample is measured. The measured current when this transition to the normal state begins is the critical current of the sample. To obtain the critical current density (J_c) of the sample, the critical current is divided by the cross-sectional area of the sample. The critical current density is measured in amps per cm^2.

In future work with the Materials Branch, I will gain experience in measurement of the critical field of the sample as a function of temperature, and the critical current as a function of both temperature and magnetic field. This will complete the three critical values which describe the transition to the superconductive state in terms of the actual temperature, the magnetic field, and the current to which the material is exposed.

At the present time I have not taken any related academic courses that would be helpful in performing this job. I feel courses in quantum mechanics and solid state will be extremely helpful. I think that my lack of knowledge may have been to my advantage because I was open to new ideas without a set way of thinking. At the beginning I did not know if I would be interested in the behavior of electrons in solids, or basic solid-state physics, but now I find the subject intriguing and I am eager to learn more. One of my learning objectives was to take on the responsibility of researching on my own, the nature of my work and I hope to keep up with this objective during the academic terms so that I do not fall behind in this fast changing field.

Synthesis of Anthracene Containing Model Compounds for Evaluation as Nonlinear Optical Materials

Task Order No. 32
Student Support Program

Paul Roderer
Wright State University

19 June 1991

Guiding Engineer
Mr. Bruce Reinhardt
WL/MLBP

ACKNOWLEDGMENTS

I would like to extend my thanks and gratitude to Capt. Cady Coleman, Gibby Dillard and Marilyn Unroe for their patience, guidance and support - and basically for "putting-up" with me. My very special and sincere thanks to Mr. Bruce Reinhardt for lending me his divine wisdom and guidance and for turning me into a chemist. He has been a very patient and understanding boss, an outstanding chemist and educator and a friend. Thank you.

TASK ASSIGNMENT

Development of Non Linear Optical compounds (NLO) has profound industrial and technological implications. NLO compounds have applications in fiber-optic technology, satellite communications, Star-Wars defense programs and computer circuitry. Since industrial competition and international competition is high in these areas successful development of these compounds presents a rather high priority to numerous aforementioned organizations.

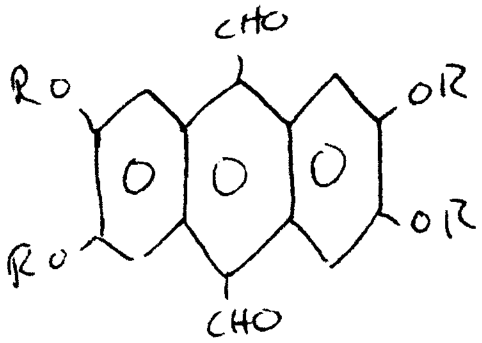
Synthesis of a specific class of NLO organic compounds was the object of this task. These compounds are classed X 3 compounds and are distinguished by the ability to control their NLO properties by another electromagnetic radiation source such as a laser. These are a particularly desired class for construction since refraction control can be very rapid with a light source. Compounds showing a high degree of second order hyperpolarizability display the greatest degree of promise for this class. These compounds also must exhibit extreme optical clarity in order to transmit to the extreme demands of these applications. Currently, the best X 3 NLO organic compounds are not efficient enough for practical applications. Creation of compounds that contain a large amount of conjugation and therefore potential for high hyperpolarizability was the main objective of this task.

There were three major projects attempted during this task. They were the synthesis of compounds 1, 2 and 3 as shown on the included figure at the end of this report. Generally, library research was needed to obtain possible synthesis methods for these compounds. These compounds have never before been synthesized and recorded so design of a synthesis route was a major complication. Compound 1 was the first attempted synthesis. Three months was spent attempting to synthesize this compound by different methods and an eventual snag ceased research of this compound. At the time of completion of this task both compounds 2 & 3 were being attempted and some very promising results have been accumulated. Neither compounds have been fully synthesized, but some of the more difficult steps have been successfully completed in relatively high and pure yields.

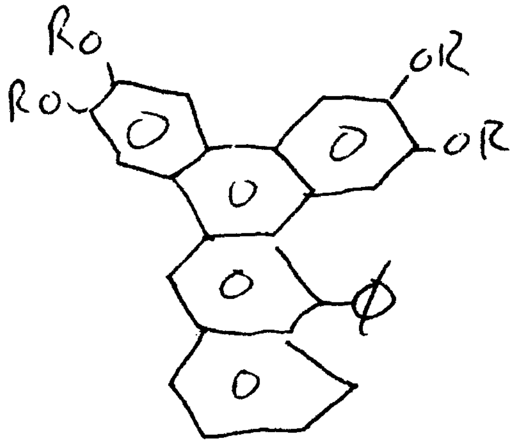
Research was initiated by Mr. Reinhardt and often began with a library research assignment. After a synthesis route was proposed (usually also by Mr. Reinhardt) experimentation began. This typically consisted of attempting to synthesize the desired compound via the proposed route. Product purification methods included column chromatography, recrystallizations and mechanical filtrations. Product structure identification was assisted by proton NMR spectroscopy, IR spectroscopy, TLC results, melting point determination, elemental analysis and mass spectroscopy.

While none of the proposed NLO structures were fully synthesized, some very important steps have been successfully completed. Organic synthesis of this type is a very slow and tedious process and often many set-backs can be expected. Important steps in synthesis of compound 2 suggested that complete synthesis of this compound is very close.

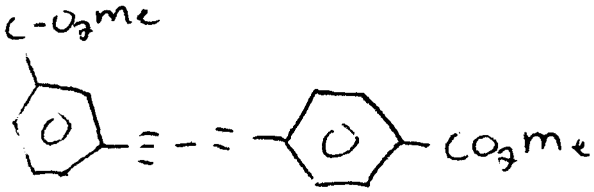
As a student of chemistry at Wright State I have personally learned a lot of theoretical chemistry. At Wright Laboratories, under Bruce's guidance, I have put this knowledge to work and learned to think like a chemist. This background has been easily as beneficial as my education to understanding and developing my scientific abilities. At the same time I feel that I have made contributions to, and benefited these aforementioned research goals. NLO synthesis was an area of chemistry that, before this task, I had never encountered and now has become my main area of interest in the chemistry field.



Compound # 1



Compound # 2



Compound # 3

Fig one

Attempted potential NLO Compounds

$R = C_{10}H_{21}$

NLO Materials for Optical Wavelength Conversion

Task Order No. 33
Student Support Program

Anne Cunningham
Xavier University

10 June 1991

Guiding Engineer
F. K. Hopkins
WL/MLPO

ACKNOWLEDGMENTS

I would like to thank Ken Hopkins, Mayan Shah and John Westwood for all the assistance and wisdom they shared with me.

TASK ASSIGNMENT

I assisted with the beginning of experimental work to demonstrate the suitability of certain nonlinear optical materials for optical wavelength conversion. To this end, I wrote computer code to model phase-matching diagrams based on the Sellmeir predictions and to draw Greninger Charts to be used with Laue X-ray diffraction of various distances. Additionally, I studied lithium triborate (LBO) and determined its structure based on information from journal articles. Laue X-ray diffraction was performed on the LBO crystal to determine the crystal's orientation and optical axes. I worked on a project team, including a mechanical engineer, to design an experiment to measure the second harmonic generation phase-matching of LBO at different wavelengths. The experiment is currently in the prototype phase of production. The first design is completed and the needed equipment will soon be built from the plans by the machine shop at Wright-Patterson. The computer programs work satisfactorily and are self-documented. It has been shown, both theoretically and experimentally, that a spherical LBO can provide meaningful Laue pictures and may oriented on the basis of these pictures once the orienting equipment is built.

Description of Research:

Since this was only the first three months of a two year projects and since the actual experiments required custom-designed equipment, the majority of my work was in background reading, journal searches and experiment design work. I did, however, perform Laue X-ray diffraction for crystal orientation.

The X-ray source used a copper target without a filter and was set at a DC bias of 40 kV, with a current of 30 mA. The Laue camera used type 57 Polariod High Speed Instant Film Sheets of 4" X 5" and an exposure time ranging from one minute to one minute and 30 seconds. The resultant pictures were transferred onto a transparent Greninger map overlaid. From the Greninger map, they were transferred to a Wulf net. The Wulf net diagram, with practice, could be read in a manner to determine the crystal orientation, provided one knew the crystal structure and had calculated the inter-planar angles. The crystal orientation is essential to determine the optical axes and from them the phase-matching angles.

Results:

I learned the skill of crystal orientation by Laue diffraction. The first prototype of the optical experiment was designed and certain parts for it were ordered. It was found possible to get meaningful Laue pictures of a small spherical crystal, where this had previously been in doubt. Since the project has scarcely begun, there are no other real results, other than the computer programs that I wrote, which work well. I am unable to make conclusions at this point due to a great lack of results.

Thermal Fatigue in Titanium Aluminide Composites

Task Order No. 34
Student Support Program

Ty Olmstead
Wright State University

10 June 1991

Guiding Engineer
Dr. Ted Nicholas
WL/MLLN

ACKNOWLEDGMENTS

I would like to give thanks to all the engineers who assisted me in my Task assignment, with a special thanks to Capt. Larry Butkus, Capt. John Pernot and Larry Zawada.

TASK ASSIGNMENT

The current task was to help Larry Zawada, Capt. Larry Butkus and John Pernot in their research of Ceramic Matrix Composites (CMCs). These composites are being considered for Air Force aerospace applications, such as the Advanced Tactical Fighter (ATF) and National Aerospace Plane (NASP). The CMCs are superior to their metal counterparts in their capabilities to maintain strength at elevated temperature, with respect to their lower density. Air Force research in CMCs is aimed at doubling performance of present day jet turbine engines. However, before the CMCs can be "bill of material" for a turbine engine, the reliability of these materials must first be characterized to determine if they are suitable candidates to replace the current metal components. One of the many groups testing these CMCs is the Materials Behavior Branch in the Materials Laboratory at WPAFB. This group characterizes the CMCs in an in-house program under the lead of Larry Zawada. The team's main focus is in the mechanical behavior, the thermal behavior and the estimation of the life expectancy of the CMCs being tested.

Specialized equipment used by the in-house group includes computer controlled Horizontal Servo-Hydraulic fatigue machines, a Schenk thermal-loading machine and an acoustic emission set-up. This equipment allows the engineers to test the CMCs with great accuracy.

To evaluate a material the engineers first conduct tension tests at room temperature and at elevated temperatures to determine the maximum strength of the ceramic or glass composite they are investigating. After the test is completed, the tensile data is analyzed to find the most interesting peculiarities (ex. the stress where extensive micro-cracking leads to failure of the specimen). The specimen is then mechanically fatigued at and near this stress level. After testing, the data, which is collected by computer sampling, is transferred to a Micro VAX computer system and converted into the proper engineering units. The data is then sent to Macintosh computers and plotted. Engineers examine these graphs to determine if any prevalent patterns or trends are present.

Only a few trial thermomechanical fatigue tests have been run to date and they are still continuing. This issue, therefore, will not be addressed at this time.

Control Systems Programming Support

Task Order No. 35
Student Support Program

David Tucker
Wright State University

27 January 1992

Guiding Engineer
Dr. Jim Malas
WL/MLIM

ACKNOWLEDGMENTS

I would like to thank the following for their help and support: Dr. Walter M. Griffith, Chief, Manufacturing Research Branch, Integration and Operations Division, Dr. Steven LeClair, Visiting Scientist, Dr. Jim Malas, Materials Research Engineer, Mr. Oliver Patterson, Visiting Scientist and Capt. Pete Vanden Bosch. They guided my work and gave advice in both scientific applications and personal development. Also to be thanked is Mr. Sam Laube, a graduate student at the University of Cincinnati, for his guidance on hardware and bus technology.

I would like to express my appreciation for this student work program. It has greatly increased my professional skills. I have enjoyed this work very much. It has influenced and confirmed my career plans. At this time I want to thank Ms. Vogler and the staff of the SOCHE organization for giving me this opportunity and for their support of this Task. This experience has been a high point of my education.

TASK ASSIGNMENT

The PLD process is used to place a thin coating of a solid lubricating material onto the surface of an item, called the substrate. An example would be to coat a metal ball bearing (the substrate) with a low friction material such as molybdenum disulfide. A sample of the low friction material is placed in the path of a laser that vaporizes it. This vapor travels through a vacuum and is deposited on the substrate. The laser beam is guided by mirrors, which move in order for the laser beam to contact the sample in different places as the sample rotates in a vacuum chamber. The thickness of the deposit can be measured and calibrated by a sensor called the XTC. The extent of the vacuum in which this is done is measured by a device called the ion sensor gauge.

It is very important to remove the operator from the lab for safety reasons. Though protective equipment is worn, there is danger of exposure to ultraviolet radiation produced by the laser. The operator records the XTC and/or ion gauge data every 30 seconds by hand while the process is in operation. Therefore the PDL process needs a computer for remote data acquisition and process monitoring instead of manual performance of these operations.

It was necessary to learn to use a Macintosh computer system. The LabVIEW 2 software package that was used to communicate with the sensors and the think C system for later data analysis. The parameters of the process, some of which were to be programed first and others that are to be programed at a later time, were studied with the scientific theory of the PLD process itself and the research goals of the PLD team. It was necessary to work with Mr. Laube on how the data was to be recorded and with the equipment operator to design a satisfactory visual interface with which the Process would be monitored.

The first items programed were equation analysis programs that were used to test a theory based upon data taken from previous experiments. After sufficient hardware arrived, data from the XTC and ion sensor gauge were recorded by computer and graphed. A user interface for the operator to monitor the process was done at the same time.

The operator is now removed from the lab and monitors the process with the computer. The operator's exposure to ultraviolet radiation has been greatly reduced. Data is recorded and stored automatically at time intervals chosen by the operator, and this stored data can be graphed by another computer application. This has spared the operator from having to take data by hand for hours and then manually enter it for analysis.

Processing and Properties of Molecular Composites

Task Order No. 36
Student Support Program

Morley Stone
Wright State University

1 July 1991

Guiding Engineer
Dr. Charles Lee
WL/MLBP

ACKNOWLEDGMENTS

I would like to thank Dr. Charles Lee (WL/MLBP) for his continued help and support within the processing and properties group of the Polymer Branch, Wright-Patterson A.F.B. I would also like to thank Dr. Chyi-Shan Wang of the University of Dayton Research Institute for allowing me to work on his project and for the trust and confidence he displayed in my abilities. His friendship and guidance were greatly appreciated.

TASK ASSIGNMENT

This task concentrated on the processing of polymer films suitable for conductivity research. The goal was to find the best candidate polymer material as compared to previous ladder polymer studies. The films were prepared one of two methods: either aggregated or cast film method. Four different polymers were candidates: PBT, PBO, di-hydroxy PBT and BBL. Upon completion of the processing of a particular film, the film will have its conductivity measured on a Keithly/cryogenic test system. Alternatively, the film might be ion-implanted prior to conductivity determination as to investigate the effects of ion-implantation on polymer/electrical properties. Two different processing methods were used so as to determine which produced the higher quality film and whether or not one method intrinsically had a better conductivity associated with its ultimate film structure.

The Air Force has been investigating these particular polymeric materials for many years as far as structural applications are concerned. The current investigation of these materials for conductive purposes and for non-linear optical purposes dramatizes the Air Force's quest for alternative materials in modern and future aircraft. The multiple-role use of materials, like molecular composites, in future aircraft shows the Air Force's Ultimate goal of improving strength and performance while reducing weights and material failure.

The methodology for this task was varied depending on the type of film desired. For an aggregated film, a 0.1% solution of polymer dissolved in methane sulfonic acid (M.S.A.) was prepared. This solution was then precipitated, dropwise, in distilled water rapidly vortexing. This rapid vortex spread the droplet of polymer into a thin sheet. This resulting thin sheet of polymer greatly facilitated aggregate film formation. This precipitated polymer was then transferred to a 600 ml. Frit filter with ~2 micrometer fine pore size where the polymer was rinsed with distilled water and methanol under house vacuum. The separate droplets of polymer coalesced into a homologous, uniform film after appropriate vacuum drying.

The cast film preparation used a 1-2% polymer concentration in M.S.A. This solution was then poured into a flat-bottom casting dish and placed into a sublimator. The sublimator sat in a 60°C, temperature controlled bath. Vacuum was applied and dry ice served as the cryogenic to condense, by freezing, the resulting acid vapor which was pulled out of the solution. This procedure normally took a week to completely draw out all of the acid. Upon completion of acid extraction, the resulting film was then washed in a distilled water bath.

Pieces of the films produced by these methods will be mounted in order to determine conductivity. The polymer will be mounted across the pins of a semi-conductor socket. A silica-rubber mounting strip is used to insulate as well as hold the film sample in place. This mounting will then be subjected to electrical potential (V) and current (I) from which resistivity can be determined. Finally, the inverse of this resistivity will prove to be the conductivity of the polymer film.

One of the goals of this experiment was to be able to prepare an aggregated film whose quality approached that of a cast film. A "good" quality aggregated film was defined as having a smooth and uniform thickness while also being compact and tight in nature. Success was obtained when some minor adjustments were made in the procedure. Primarily, the use of a larger volume water bath (>3000 ml.) during precipitation and increased neutralization via rinsing with greater volumes of water and methanol improved film quality. These steps lowered the degree of acidity which was believed to be hampering earlier film processing efforts. Therefore, it was finally possible to produce aggregated films which possessed the qualities defined above.

The cast film preparation was not as successful, but errors were equipment related. Initially, film samples were processed using a conical-top sublimator with a circulating, refrigerated alcohol as the cryogenic. The design of this sublimator allowed impurities condensed on its top to fall back onto the film. Also, the setup made it very difficult to position the casting dish so that it was level. To correct this situation a new sublimator was purchased which used a central dry ice chamber as its self-contained cryogenic. The compact design also made the leveling of the casting dish in the chamber easier. However, an error in the design of this sublimation unit also effected film quality. Specifically, the top and bottom pieces of the sublimator did not fit tightly. As a result, water vapor in the air was drawn inside the sublimator, condensed on the inside of the chamber and contaminated the film with moisture. To correct the problem in future films, the top and bottom pieces of the sublimator will be ground down to provide the much needed air-tight fit.

Until now, our conductivity measurements have been limited to various heat-treated BBL fiber samples. Upon good cast film sample production, comparison of conductive properties between the two methods can commence. Until then, only theory can predict the behavior of these films under current (Figure 2).

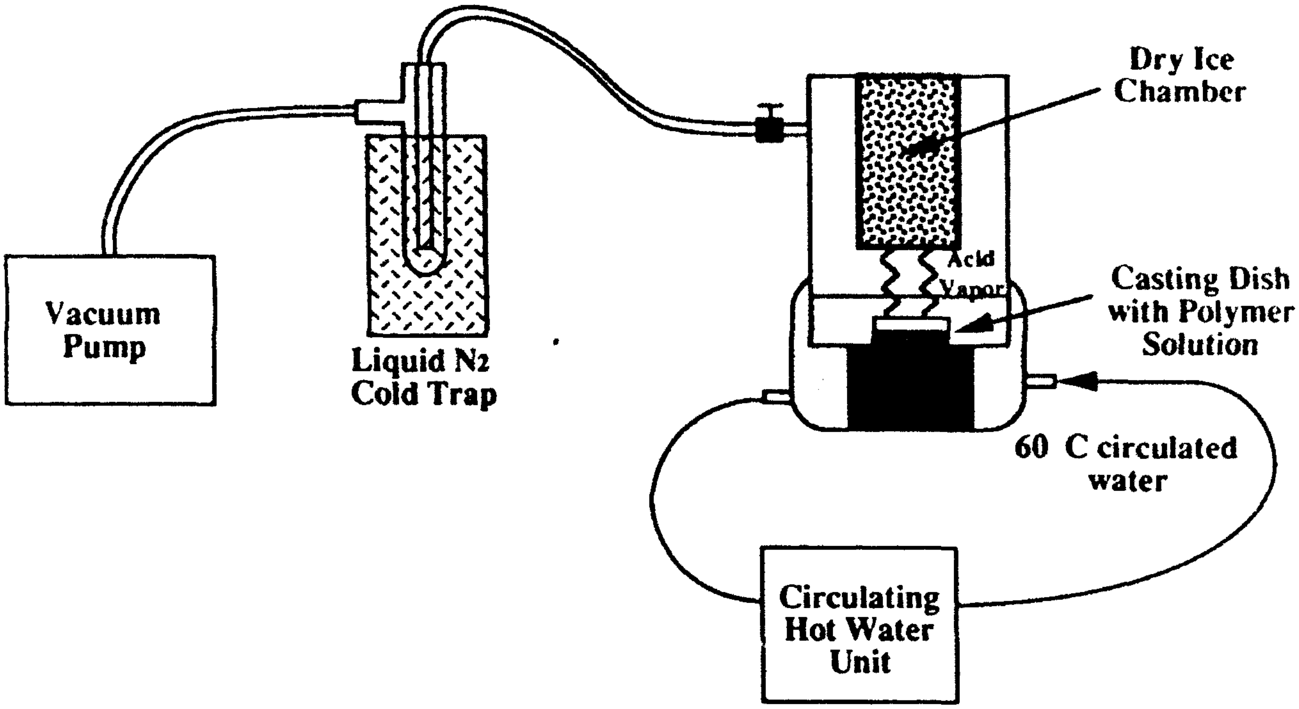


Figure 1 : Sublimation Chamber Set-Up For Cast Film Processing

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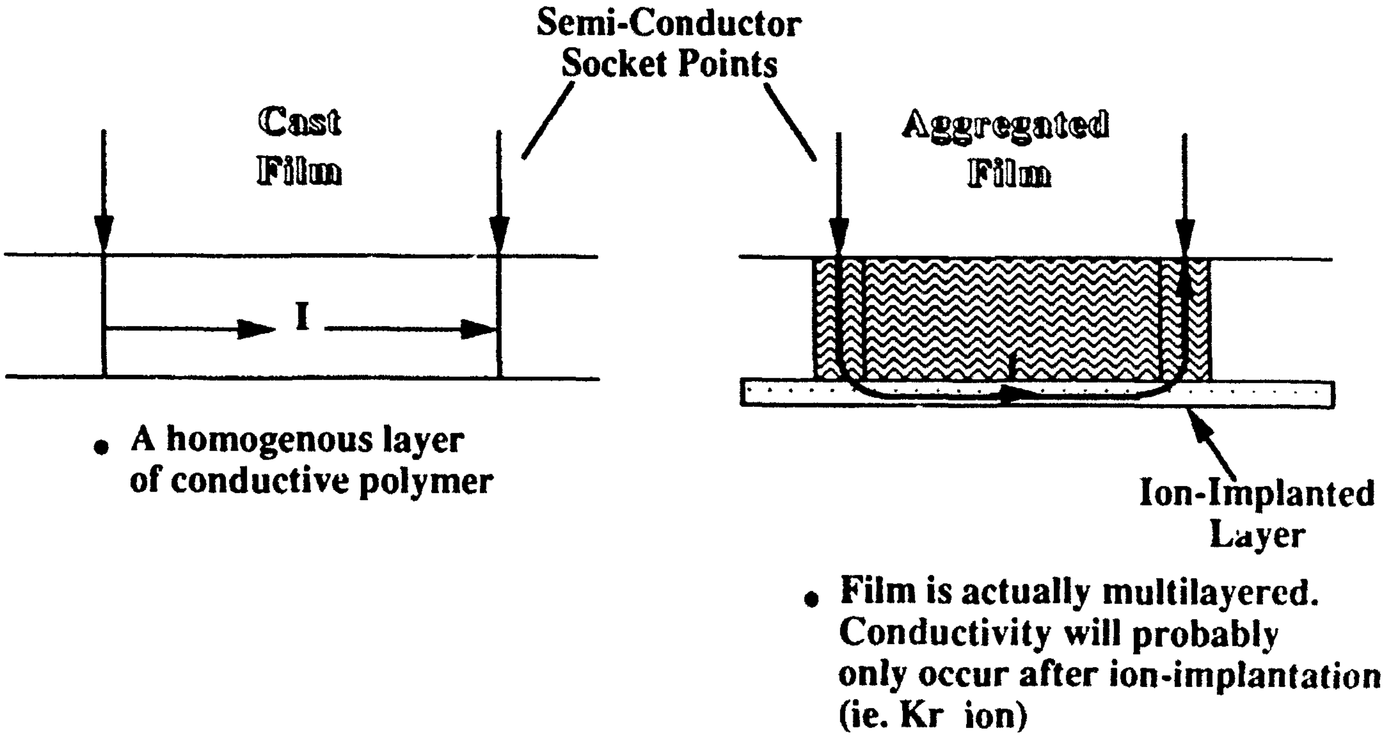


Figure 2 : Theorized Film Properties

Synthesis and Characterization of Polymers

Task Order No. 37
Student Support Program

Sungmee Yoon
Wright State University

28 October 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to recognize Dr. Tom Cooper, Dr. L.V. Natarajan, Dr. Zibgnew Tokarski, Tim Bunning, Kelly Menster and Dave Flora for their assistance with my research at the WL/MLPJ.

TASK ASSIGNMENT

The Task Assignment Number 37 involves the synthesis and characterization of mesogenic units (a component of a molecule that includes a mesomorphic or liquid crystalline phase) and leader groups which have photochromic and NLO (Non Linear Optical) properties.

Synthesis: Various carbon length leader groups, allyloxybenzoic acid (3C leader group), penteneoxybenzoic acid (5C leader group), and octeneoxybenzoic acid (8C leader group) were synthesized by following a procedure modified by Tim Bunning from reference literature 167. Dicyclohexylcarbodiimide (DCCI) coupling reaction was used with a catalyst, dimethylaminopyridine (DMAP), to attach hydroxybiphenyl and also to cholesterol. Different solvent systems were used to purify the products in trying to find the best solvent systems (cost and toxicity).

DCCI/DMAP coupling reaction was also used to attach 1'-hydroxyethyl-3',3'-dimethyl-6-nitroindolinospiropyran (hydroxyethyl BIPS) to 3C leader, Isobutryic acid, 3-(2-thienyl) acrylic acid and poly-L-glutamic acid. More DCCI reaction was conducted to couple 3C leader and N-4-nitrophenyl-(L)-prolinol, cholesterol and carboxy ethyl BIPS, hydroxy nitro BIPS and PGA H+ with pyrrolidino pyridine catalyst. A silanization reaction was conducted using 45:45:10 ration of the hydroxybiphenyl allyloxy benzoic acid: cholesterol allyloxy benzoic acid: "T" BIPS allyloxy benzoic acid, respectively. A four step reaction was done to synthesize 5-hydroxy-1',3',3'-trimethyl-6-nitro-BIPS and 1,4-diiodohexane was reacted with 2,3,3-trimethyl-(3H)-indole to ultimately synthesize iodohexyl BIPS. These various reaction products were purified by recrystallizations, TLC (Thin Layer Chromatography), column chromatography and flash column chromatography which were analyzed by either FTIR, Lambda 9 Spectrophotometer, NMR and/or elemental analysis.

Results: Various different ratios of solution mixture in purifications were found. Some combination characteristics of the chemicals were learned such as the hygroscopicity of cholesterol absorbing water from the atmosphere and DCCI will more readily react with water than cholesterol to inhibit the reaction process. One of the elemental analysis result of the product synthesized following a literature was far better than the published article's result and also to the exact theoretical value which indicate better product purity and completeness of the reaction.

Systems Support-Materials Behavior and Evaluation

Task Order No. 38
Student Support Program

Joseph Hunter
Wright State University

22 September 1991

Guiding Engineer
Mr. Bob Urzi
WL/MLSE

ACKNOWLEDGMENTS

My thanks goes to William Purcell, Mark Forte, James Mazza, and Mr. Robert Urzi for their help and guidance throughout my task.

TASK ASSIGNMENT

I worked in the Systems Support Division at WPAFB Materials Laboratories. The lab's main concern was in the area of adhesives and composites. A couple of overall objectives included analyzing existing adhesives for structural repair of aircrafts as well as studying various methods in which to field repair the body of aircraft using composites. Several projects relating to these objectives were conducted including: "Environmental Conditioning Comparison" (engineer: William Purcell), "Wet Lay-up Repair Evaluation" (engineer: Mark Forte:) and "Adhesive Processing Study" (engineer: James Mazza) to name a few.

In the "Environmental Conditioning Comparison" project the effects of water and humidity on cracks in aircraft were studied. To do this 200 wedge specimens using two different surface preparations and two different adhesives with a consistent primer were prepared. The specimens, consisting of two pieces of aluminum 2024-T3 bare and an adhesive, required a great deal of preparation. A huge metal sheet was cut into 1 ft in. pieces. The pieces were put into a vapor degreaser which removed grease and dirt using a methylethylketone base. Scotch-Brite and Alconox were used for another cleaning phase. Half of the metal sheets were then etched (three pieces at a time) for twenty-two minutes using a phosphoric acid anodizing solution along with a 10 volt current supply through the acid. The remaining sheets received a Pasajell surface treatment. Etching is basically a controlled corrosion process. After rinsing (15 min.) and drying 15 min.) approximately 0.4 mils. of BR-127 primer was sprayed on one side of each piece of aluminum. The primer adds about 50% adhesive strength to the specimen. One of two film adhesives is now used to adhere two pieces of metal. Half of the specimens are cured by vacuum pressure and half by direct pressure, both methods requiring 250° F for 90 minutes. The plate specimens were cut into 200 testing specimens using a bandsaw. The specimens were then machined so that the glue-line thickness readings could be taken. They were then ready to test. Wedges were put in one end of each specimen, the initial crack length created was measured and the specimens were placed in either water or a humidity cabinet at 70°, 120°, 140°, 160° and 180° F temperatures. Crack readings were then taken after 1, 4, 24, and 144 hours of conditioning. After testing, crack length vs. time were plotted and then analyzed which adhesive and/or surface preparation performed best and at which temperature water conditioning was more severe than humidity. In all instances

PAA surface treatment outperformed Pasajell, and also crack length did increase in step with temperature as was expected.

The purpose of the "Wet Lay-up Repair Evaluation" project was to validate current repair procedures using new adhesives made by Hysol Corporation. Our statistical data along with that of the flight Dynamics Lab would help determine the usefulness of the adhesives for use on the B1-B.

The following is a detailed lay-up procedure. A 24 x 36 in. piece of graphite fabric was cut and placed in a nylon bagging film. Then EA 9396 resin was mixed, adding 50% excess resin, and impregnated the fabric by applying resin into 10 pieces, they were placed on top of each other, and the panel was cured under 26 in. Hg for 45 minutes at 200° F/min. heat up rate. A non-destructive evaluation of the panel was conducted and the specimens were tested using four-point loading and compression to obtain strength data. The whole procedure was conducted many times using different impregnation techniques to find out which method produced less voids. It was found that pouring all the resin on one or two bottom pieces of fabric and then piling the remaining dry pieces on top was the best way to decrease voids. This method allowed the air in the resin to escape through a porous teflon filter before reaching the remaining graphite plys.

The purpose of the "Adhesive Processing Study" project was to establish a vacuum pressure range for the curing of Hysol EA 9394 adhesive over all temperature ranges. Seven standard lap shear panels were fabricated using 2024-T3 bare aluminum as the adherents. The aluminum surface preparation was phosphoric acid anodize followed by priming with American Cyanamid's BR-127 adhesive primer. After mixing the hardener and resin the adhesive was applied to both adherents. The thickness of each panel was controlled by using glass beads in the resin. The panels were cured at 200° F for 90 minutes at a heat-up rate of 5-7° F per minute. The pressure during cure was provided by vacuum and varied for each of the seven panels (14 to 29 in. Hg). After fabricating, the panels were cut and machined into specimens. The bond area and bond line thickness for each specimen were measured and recorded prior to tensile testing. A viscosity and pressure graph was then tabulated to obtain the results. It was found that when too high of a pressure was used the panel foamed at the bond line, but when the pressure was too low the panel simply came out too thick. Finding the optimal pressure at which to cure is an important procedure when testing new adhesives.

Materials Behavior and Evaluation

Task Order No. 39
Student Support Program

Cathleen Gustafson
Wright State University

31 December 1991

Guiding Engineer
Mr. Robert Urzi
WL/MLSE

ACKNOWLEDGMENTS

This Task was completed with the help and guidance of Mr. Jim McKiernan and Mst. Sgt. Bryan Cramer. Mr. Robert Urzi, the Technical Area Manager was very supportive. The engineers in the section, especially, Mr. Jim Mazza, Mr. Mark Forte and Mr. Bill Purcell were very helpful and willing to answer any question.

TASK ASSIGNMENT

In Composite Supportability, the work done is centered on testing composites and adhesives. Testing is done mainly on different types of adhesive systems including; film adhesives, two-part paste adhesives and resin systems. The tests are done by making test specimens, including but not limited to, lap-shear panels, blister panels, composite panels for four-point flex-shear and compression tests and plate shear specimens.

All the panels have to be cured either in a press, autoclave or zip-vac system. Different specimens are prepared different ways for the actual testing. The panels have to be cut and machined to the correct size. Exact measurements are taken to be used in the final calculations. The actual testing is usually done on a tensile machine.

The Air Force is continually looking for better, faster and less expensive ways of doing things. One reason for these tests is to determine the best product. Companies send samples of their products hoping the Air Force will determine that their product is the one to use. Some of these products are tested by groups. They also try to develop better ways of doing things like repairing battle damage. This group will also double check other peoples findings, if the results could have an impact on the Air Force.

The results of the tests can have direct and immediate impact on how things are done. If a new method is developed to make patches for repairing battle damage, the new method will be used. Also, the better performing adhesives will be used. The results are usually published so others can benefit from the research.

An engineer first has to submit a project plan before anything can be done. Once a plan has been approved and assigned a number, the project engineer reviews it with the technician. The technician is then responsible for seeing that the project is completed.

The first thing done is to make sure that all the supplies required are available and are still within their shelf life. The next step varies depending on the types of testing that is going to be accomplished. Metal to metal panels (i.e. lap-shear) have to be cut to bonding size, the surfaces prepped by either the Phosphoric Acid Anodized (PAA) or Pasa-Gel 105 method and sprayed with primer before they can be bonded. Composite panels are made by a procedure called a wet lay-up. The adhesive for the set lay-up is usually an epoxy resin that is spread evenly on the composite cloth. The cloth is then cut to the correct panel size. Both types of panels, metal and composite, are cured according to the test plan. Different curing methods yield different results. Next, specimens have to be cut from the panels, the metal ones using a band saw and machined smooth on a milling machine and the composite panels with a cutting wheel. It is important to get specimens uniform in size and shape because they directly affect the test results.

The type of test run is determined by what specifications are needed. Most of the tests are run on a tensile machine. The tensile machine can be configured for various types of tests. A plot of the test results can be obtained by connecting the tensile machine to a plotter.

The results of the tests are compiled by the technician and given to the engineer. The engineer then analyzes the results and determines if additional testing needs to be accomplished. If everything is complete, the engineer writes the final report indicating the results of the tests.

This Task provides many opportunities to apply knowledge gained in school. The practical experience obtained in this Task will aid in the understanding of several topics in future classes. Also, understanding the types of machines implemented and their capabilities will be a valuable asset as an engineer. The Task also helps to determine if this is the career field of choice.

Tribological Studies On Research Fluids

Task Order No. 40
Student Support Program

Scott Long
Wright State University

31 December 1991

Guiding Engineer
Dr. Shashi Sharma
WL/MLBT

ACKNOWLEDGMENTS

I would like to direct my thanks to Dr. Shashi K. Sharma, Mr. Bruce Schreiber and Mr. Ron Mortimer for their time and effort in assisting me with my Task. They were patient and understand-ing when I did not have the necessary knowledge of the material I was working with.

TASK ASSIGNMENT

While working on this Task, a variety of jobs was performed. For the first ten weeks, an extensive and detailed report of the WL/MLBT Pump Tests performed from 1980 through 1991 was created. This consisted of thirty-two tests that ran from 100 hours to 930 hours. For example, there were data sheets, metal analysis reports, pictures, data charts and lab notes. The data was organized into a database by using the Apple Macintosh II system and the Zenith IBM compatible lap system. Specifically the "Excel," Macdraw II" and "Microsoft Word" software were utilized. All this information will be used to create the final report at a later time.

Second, the Cruise Missile Grease Bearing Experiment was run. This consisted of running tests on bearings that have been stored in extreme conditions in a humidity chamber. The chamber represents the condition the bearing will go through while sitting in the missile. Two practice tests and only nine true tests were completed. The tests had to be aborted because of the continuing failures. The Amoco/Pope Spindle bearing tester was used throughout the test. Each test involved analyzing the test bearings, disassembling the spindle, reassembling it and monitoring the test. The final process involved analyzing the data and determining why the failure occurred.

Next, an Eight Channel Data Acquisition Filtration Box was designed and constructed. A circuit had to be designed to perform this job. Resistors, capacitors and other electrical parts were used. The two boxes made will be used on a Tribology machine and Traction Disk machine.

The next procedure that took place was "moth balling" the Hydraulic Fluid Pump Stand, which involves cleaning the stand. Everything was taken apart and cleaned thoroughly. All the components of the stand were put back together to keep it clean until the next test. Overall, there were two stands that underwent this procedure.

Finally, a RMS-to-DC converter was devised. Again, circuits were drawn-up to perform this task. The converter will be used on the Traction Disk and Tribology machines.

There is an oscilloscope and data acquisition output on the box. To construct this, resistors, capacitors and zeener diodes were used.

I have learned a great deal about electronics and fluids during my Task. I became familiar with analyzing data and constructing circuits. I have also learned to work as hard as I can and not be content with anything less than doing my best. I put one-hundred percent into everything I did and was rewarded by my accomplishments. I am very glad that I had this opportunity to gain more knowledge about a variety of details. I hope that I will be able to further my knowledge in the future.

Automated Optical Characterization of NLO Materials

Task Order No. 41
Student Support Program

Linda Wood
Wright State University

16 December 1991

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank the following individuals for whom I have worked and who have helped me in my Task Assignment: Dr. R. L. Crane, Dr. T. M. Cooper, Dr. Z. Tokarski, Mr. Marc Martin and Ms. Teresa Wilson.

TASK ASSIGNMENT

Integration of computer hardware with existing lab computer systems was examined to improve efficiency of lab work. As a result, a CD-Rom was added in order to utilize effectively large quantities of information available for lab safety, specifically, MSDS sheets.

Investigations were made into possible applications of computer software to increase lab operation effectiveness and efficiency. As a result, many programs were investigated and this knowledge has been used to tutor individuals on usage as well as assisting individuals with questions and problems in using the software.

Logical developments were used in the analysis and study of materials and equipment desired to be utilized. The main resources for study were library materials, manuals and individuals available in and around the base.

Operation of many different pieces of hardware including; Macintosh II, Vax, Perkin Elmer and IBM computers was learned. Data analysis, transfers and translations were all accomplished with existing computer software and equipment.

Much was learned about operational systems. It seems the importance of skilled individuals and those who practice continued learning is invaluable to the scientific research field. Staying informed of new discoveries and equipment and the latest methods and accomplishments in research is essential. The advances made in computer systems and operations comprise a large segment of this area. They are not only used for receiving current information, but are continually becoming more important to aide the effectiveness, speed and efficiency of analysis as well.

I believe continued experience that I gain from working at Wright Labs will prove invaluable to my desired career in the environmental research and development field. I also believe that my contribution to the lab is beneficial in maintaining a smooth operation.

Interfacial Properties in Ceramic Matrix Composites

Task Order No. 42
Student Support Program

Cynthia Kurek
Wright State University

27 January 1992

Guiding Engineer
Dr. Ron Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Larry Matson, Ron Kerans and Randy Hay for their guidance and direction in the projects. My gratitude also goes to Mike Scott and Eric Fletcher for their assistance in metallography and to Cameron Begg for his help on the SEM.

TASK ASSIGNMENT

The Task was divided into two parts. The first part was performed during the summer and involved TEM (transmission electron microscope) foils of YAG (Yttria-Alumina-Garnet) - Alumina eutectic fibers. This was related to Larry Matson's research on the YAG-Alumina interface. This is important as an Alumina-matrix/YAG-fiber composite is under investigation for possible high-temperature use.

The second part of the Task began in the fall when work was started with Randy Hay. He is investigating the kinetics of the YAP (Yttria-Alumina-Perovskite) - Alumina eutectic transformation to polycrystalline YAG. This reaction is very interesting as it is accompanied by an 11% volume increase. This leads to possibilities for a fiber material that would experience large compressive stresses, which are desirable in composite design.

During the summer a systematic method for producing fiber foils was developed through trial-and-error. The final product involved mounting the fibers in epoxy and polishing one surface. The surface was then glued to a glass slide, the excess mount was cut off and the backside of the fibers was polished to a thickness of 20-40 microns. The fibers were then cut into foils and ion milled until they were ready for study in the TEM.

More recent work has involved heat treating small pieces of YAP-Alumina eutectic for known times and temperatures, looking at the samples in the SEM (scanning electron microscope) and preparing them for the TEM. Cubes of the eutectic were heated in a furnace between 1200° and 1400°C for 6 to 2000 minutes. The samples were then cut in half and mounted. The specimens with large amounts of reaction product were looked at in the JEOL 840X scanning electron microscope. Photographs were taken, showing where and how much of the material had transformed.

All specimens, whether they were looked at in the SEM or not, were made into foils for TEM inspection. This again involved gluing the polished surface to a glass slide, cutting off the excess mount and polishing the backside of the specimen to a thickness of 20 microns. The specimen was then covered with a copper grid, cut into 3 mm. disks and ion milled. The foils are then studied in the TEM and photographs are taken from which desired data can be acquired.

The work on the YAP-Alumina project is not yet complete. Several heat treatments remain to be performed. Many specimens also need to be analyzed in the TEM. After this is done, the photographs must be studied to find a relationship between the amount of reaction, time and temperature. Results from this study are to be presented at the annual Ceramic Society meeting to be held in Minneapolis in April.

Interfacial Properties in Ceramic Matrix Composites

Task Order No. 42a
Student Support Program

Robert Strevell
Wright State University

27 January 1992

Guiding Engineer
Dr. Robert Kerans
WL/MLLM

ACKNOWLEDGMENTS

My thanks go to Dr. P. Jero and Dr. Robert Kerans for their help and guidance throughout my task.

TASK ASSIGNMENT

The purpose of this project was to show that moisture will affect the interface of an SCS-6/glass composite by altering the interfacial frictional stress. Three borosilicate glasses were used (D, E & F), which varied the residual stress state of the samples from tension, in the case of D glass, to compression in the case of F glass. These three samples were in turn cut to three thicknesses. The results were obtained by pushing out a set of fibers and then "re-pushing" those same fibers after controlled exposure times in dry, moist and wet environments. Thus, it was demonstrated that environmental considerations are important criterion for design of these composites.

The results of this experiment show that samples exposed to moisture require an increased peak load to push-out the fiber once debonding has occurred, and that with increased exposure time and sample thickness this effect is more pronounced. From this it can be seen that exposure to moisture causes an increase in the interfacial frictional stress, and that this effect is a function of exposure time and interfacial area.

In conjunction with this project, a second experiment was performed to determine the effect of a "wet" environment on the debonding characteristics of the same SCS-6/glass system. In this case separate groups of fibers were pushed out at 24 and 48 hour intervals. The effect of a wet environment on the initial debonding of the fibers was then observed.

The results of this experiment failed to show any significant change in the initial debonding stress. However, in most cases, taking into account the fact that the samples are exposed to air during the testing procedure, there is an increase in the initial debonding load with increased exposure to air during testing after being immersed in water.

Composites are replacing other structural materials to a much greater extent today than aluminum and steel alloys, due to their higher strength to weight ratios. How useful ceramic composites will be depends on the ability to understand and engineer the fiber/matrix interface (1, 2). The properties of importance in this interface include the interfacial frictional stress, which affects the load carrying ability and pull-out of the fibers after debonding, and shear strength which controls the debonding. Single fiber push-out tests are one way to measure the frictional stress. However, little attention has been given to the factors that contribute to frictional stress.

Once a composite has been designed and put into service it is important to know if and how it's properties will change during it's life. It is expected that moisture, which is present in most environments, will have some effect on these properties. Assuming this is true, environmental considerations are important design criterion.

Experimental specimens with matrices of varying CTE were prepared and cut to three thicknesses. CTE refers to the coefficient of thermal expansion. These samples were then tested dry, in the presence of water vapor and wet. For the first experiment, testing consisted of flat-bottom probe push-outs with re-pushes at 0 hour (immediately), 1 hour, 4 hour and 24 hour intervals. The results were recorded as crosshead displacement vs. load diagrams. The second experiment consisted of the same push-out procedure, but was conducted by pushing out one fiber as a control, then pushing out two groups of fibers exposed to the wet environment for 24 and 48 hour periods.

The fiber used in this experiment is SCS-6 from Textron Specialty Materials. SCS-6 is a silconcarbide fiber. The SCS-6 fiber (≈142 micron dia.) uses SCS-O as a core, which is a siliconcarbide fiber with no coating, with a complex carbon-rich coating that is 4 microns thick and varies radially in the ratio of carbon to silicon. Details of the chemistry and structure are given in DiCarlo (3).

The three glasses used are Corning Glass Works (CGW) D, E & F, each having a different coefficient of thermal expansion (CTE). The CTE is responsible for the mechanical gripping between the fiber and the matrix (4).

The fibers were held in place by tape while being wound into tapes with 0.56 mm fiber spacing. These tapes were then cut into lay-ups for loading into the graphite dies. Glass frit (≈8 grams) was packed into the die one gram at a time with a layer (7 total) of fibers between each gram. The charge was then heated under a vacuum (refer to Appendix A). Pressure was applied once the softening temperature of the glass was reached (≈850 degrees C) and held for approximately 20 minutes under argon. The samples were allowed to cool in the press.

Samples were then cut, perpendicular to the fiber direction, to three thicknesses ranging from 0.8-3.0 mm. Each sample was then stored in a desicator to prevent premature exposure to moisture. Next, both faces of the sample were polished to 6 μm finish on diamond wheels to reduce the affect of surface roughness. Kerosene was used as the lubricant. Each sample was examined under polarized light to confirm the tensile, neutral and compressive stress states of the D, E & F glasses respectively, and to select the fibers to be tested.

Push-out tests were then performed using the Instron Universal testing machine. Next, the polished surface of the glass was cemented to a metal mount with a slot in it, so that the row of fibers selected to be pushed were centered over the slot. The mount, gripped in a sine-vise, was aligned with the load train using a traveling microscope. Load vs. crosshead displacement was then recorded during the push-out procedure.

For each sample tested, the fibers were initially pushed-out then unloaded and immediately reloaded and pushed again or "re-pushed". This initial push and re-push were performed directly after removal from the desicator. Successive re-pushes were performed after 1, 4 & 24 hour periods of exposure to dry, moist and wet environments. Dry samples were kept in the desicator, moist next to a water vapor source (in this case a wet sponge) and wet samples completely immersed in water.

From the first experiment it can be seen that for the SCS-6/D, E & F glass systems the interfacial frictional stress is affected by exposure to moisture as a function of exposure time and interfacial area. The interfacial frictional stress apparently increases with both increased exposure time and increased interfacial area. Some of the results are inconsistent with the explanation offered, possibly due to inconsistencies in fabrication and preparation, but these inconsistencies were minimized as much as possible. These results warrant further investigation, including collection of more data and SEM characterization of the interface itself.

From the second experiment it can be seen that after exposure to water the load required for debonding increases as well, but only as a function of the time of exposure to air after removal from the water. This effect is much less pronounced in the case of fibers which have not yet been debonded, as is the case in the previous experiment. This suggests that the interface is less vulnerable to the effect of moisture in this state.

The first experiment shows conclusively that moisture has a definite effect on the interfacial frictional stress of previously debonded fibers and that this effect shows a possible increase in frictional stress as a function of exposure time and interfacial area.

The second experiment shows much less conclusively an effect on the initial debonding stress with exposure to water. This effect however seems to be a function of exposure to air after removal from water since the specimens show an increase in initial debonding stress with increased exposure to air.

APPENDIX A

Fabrication of Specimens

Fiber: Textron SCS-6 SiC, 142 µm Dia.

Matrices: 3 K_2O-B_2O_3-SiO_2 glasses:

	Modulus (GPa)	CTE (x 10^6, /°C)*
D	63.0	3.66
E	69.0	3.95
F	67.6	4.25
SCS-6	410	3.99

* RT-500°C

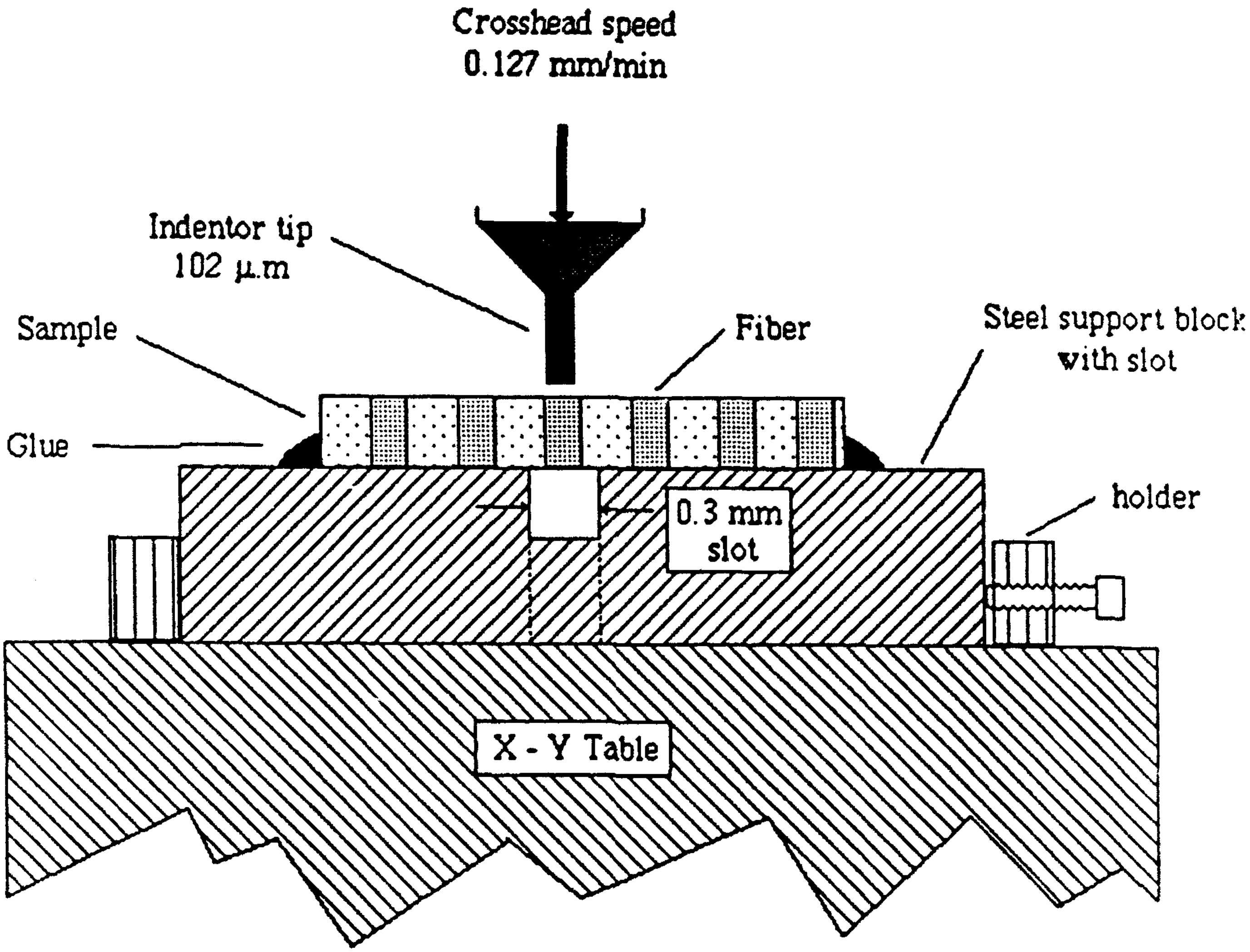
Compositions: (wt %)

Glass	SiO_2	B_2O_3	K_2O
D	79	16	5
E	77.5	16	6.5
F	76	16	8

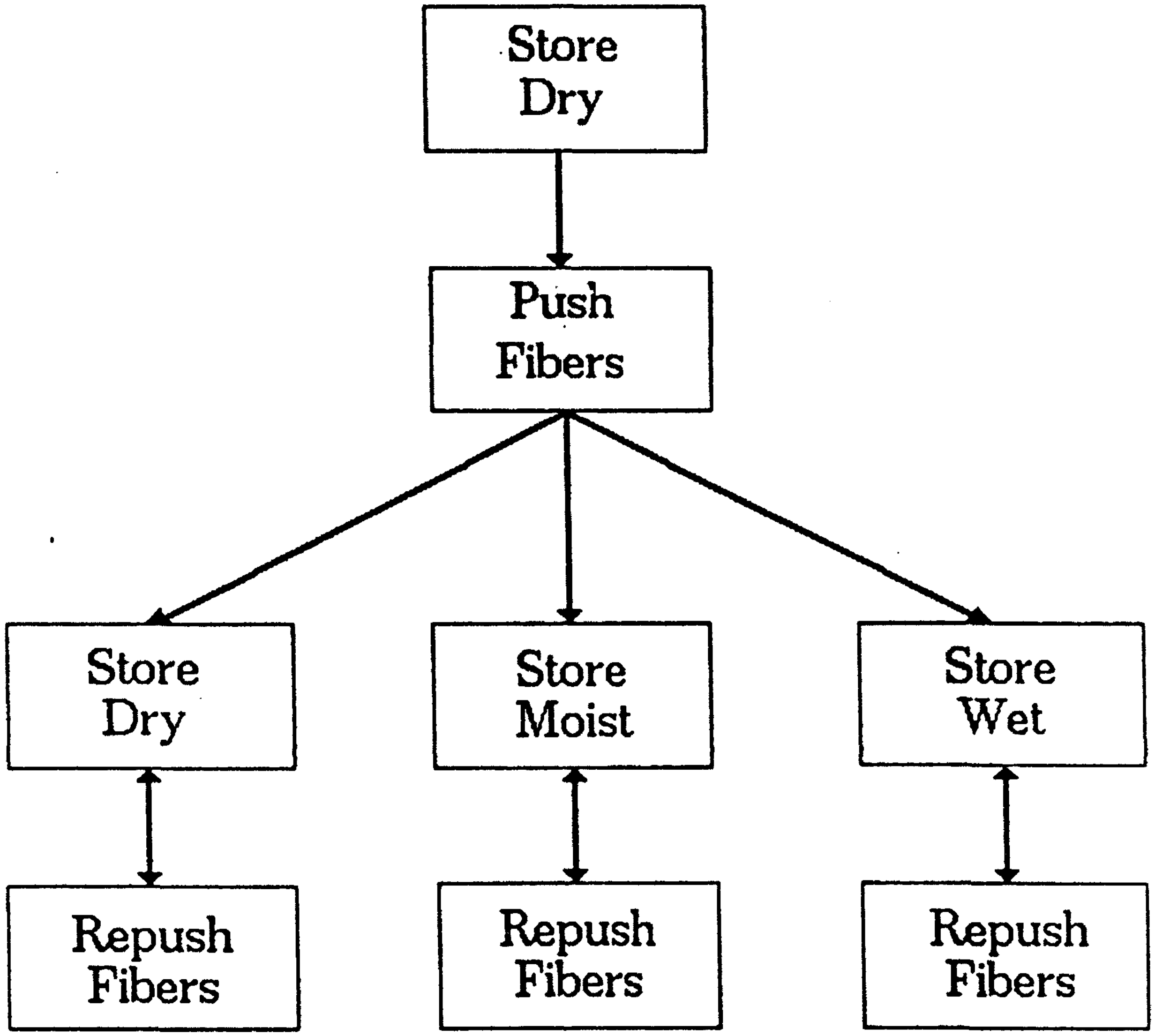
Sample thicknesses:

~0.3, 1.2, 2.0 mm

Processing:	Wind tapes
	-0.56 mm fiber spacing
	Cut & lay-up tapes with glass frit
	-unidirectional
	-6 fiber layers
	Vacuum hot press in graphite dies
	-850°C, 20 min, 500 PSI
Preparation:	Cut slices perpendicular to fibers
	-store in desiccator
	Polish both faces to 6 μm finish
	-diamond wheels / kerosene lubricant
	Examine/photograph specimens
	-confirm stress state
	-select fibers to push


Crosshead speed
0.127 mm/min
Indentor tip
102 µ.m
Sample
Fiber
Steel support block
with slot
Glue
0.3 mm
slot
holder
X - Y Table

Test Matrix: Sliding Friction



Store Dry

Push Fibers

Store Dry

Store Moist

Store Wet

Repush Fibers

Repush Fibers

Repush Fibers

<u>Repush after:</u>

0 hr (immediate)

1 hr

4 hr

~24 hr

extended

High Temperature Mechanical Behavior of Ceramic Composites

Task Order No. 43
Student Support Program

Douglas Barlage
Wright State University

23 June 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank T. A. Parthasarathy (UES), S. Sambasivan (Systran Visiting Scientist) and Dr. Kerans for their guidance during my Task assignment.

TASK ASSIGNMENT

The objective of the Task was to identify the mechanisms that control the high temperature failure of ceramic composites. The primary mechanism explored was the behavior of the fiber matrix interface. Assistance was also provided in creep property testing through sample preparation. Another primary activity was assistance in free energy measurements of YAG. The bulk of all these activities consisted of hot pressing, creep sample preparation, and micrprobe sample preparation all involving considerable metallurgical activity. One primary exception was a large deal of work into the push out testing of a model composite. Here activity was also focused on testing and the analysis of the results.

The start of the Task involved preparing compression specimens for creep testing. These specimens are typically 3X3X6mm in size. A high polish was required on three faces of the box shaped structure. Achieving this size and polish with any regularity and little waste is not a trivial assignment. With guidance a hand held mount was designed and constructed to be used in conjunction with existing polishing and grinding devices to expedite processing of these specimens.

Shortly after, considerable hot press work needed to be done to create sintered ceramics for various testing. Training in the hot press technique was given along with the responsibility of producing several ceramic slugs to be used in the free energy of YAG study. These slugs were then heat treated. After this, small specimens for testing in a microprobe were prepared. The most directly related of these hot pressing assignments for interfacial studies was the dopant migration in an alumina-YAG composite. Here, a highly polished YAG single crystal slug was hot pressed in alumina powder doped with different metals. The composite was then heat treated and tested to see if the dopent diffused to the interface between the alumina and the YAG. The composite interface was then tested to see if indeed the interface became weak with the addition of dopant.

During the entire assignment, another project that involved push out testing was worked on. It has become evident that interfacial roughness plays a large role in the behavior of the interface of ceramic composites. There was a need to gain insight into how these effects influenced push out behavior. To do this, it was suggested that a model composite be created that had large rods that acted as fibers. It was relatively unimportant what material this was constructed of. Ease of construction then became a primary factor in the design of this model composite. Plexiglass rods were chosen to be imbedded into an epoxy matrix. Several different epoxies were tested with the same plexiglass rods. It turned out that during setting, the epoxy bonded to the plexiglass rods. This voided the possibility of push out testing. The plexiglass rods deformed before they were able to be pushed out of the epoxy matrix. Different coatings of the fibers were then examined to prevent this bonding. A teflon type substance was found to coat the fibers evenly and sufficiently enough to prevent bonding in a consistent manor. The next large problem to be overcome was controlling the geometry of the fibers to be imbedded in the matrix. A good deal of machining skills had to be learned to create molds with tight enough tolerances to provide consistent results.

After a sufficient model composite had been created and tested for viability in push out testing, the rods were then manipulated to provide different interfacial roughness. Four large groups of model composites were then created, as received (0 micron) and 15 micron, 25 micron and 48 micron applied roughness. These were then tested for the push out and push back sliding frictional load. This load was then normalized with respect to thickness and plotted vs. the assumed amplitude of the roughness. A linear relation was then found as predicted by the model of Dr. Kerans and Dr. Parthasarathy. However, two things were slightly disturbing about the resulting plot. The coefficient of static friction that was derived from the plot was an order of magnitude lower than expected and there was considerable scatter in the data. It was determined that there should be some direct measure of this roughness that we assumed to be directly related to the grit of the polishing material that was used on the rods. Optical methods proved to be qualitative at best so a stylus probe that dragged across the surface much like a needle on a record player was used. From this direct measure of roughness could be obtained for each fiber pushed out.

Originally it was assumed that the average peak to valley for this roughness should be used as the value for roughness. However, examination of the plots obtained from the stylus probe indicated that this simple measure would be somewhat misleading. It was suggested that some type of RMS value should be used. It was also proposed that some type of fourier analysis should be done on the roughness data to measure spatial period and amplitude. This was confirmed by others that this would be a reasonable thing to do and a fourier analysis was written to analyze roughness in this way. Most of the fibers were then measured and analyzed for roughness in this way. This new measure of roughness was then plotted vs. sliding load and this did indeed reduce scatter in the data and produce a reasonable coefficient of static friction. This work also fulfilled senior project requirements.

The last year has involved exposure to many different types of equipment and techniques including computer data acquisition analysis, laser interferometers, optical microscopy, SEM and microprobe. However, the most important thing learned during the Task was sound experimental science. Being able to observe experiments develop from start to finish and work with many excellent researchers is an invaluable asset universal to any technique training.

QPA for Pulsed Laser Oblation

Task Order No. 44
Student Support Program

Richard L. Hawes
Wright State University

21 April 1992

Guiding Engineer
Dr. Jim Malas
WL/MLIM

ACKNOWLEDGMENTS

Thanks to Victor Hunt who provided technical assistance with performing flux compensation experiments. Thanks to Jeff Heyob who provided assistance with writing software. Thanks especially to Oliver Patterson who tutored me in using the Macintosh and Lightspeed C.

TASK ASSIGNMENT

The Task worked on is QPA for MBE (Beta Controller Development). The description of the Task is described as follows: The objective is to develop an advanced process control system for the semiconductor growth process, Molecular Beam Epitaxy. This system is being developed on a Macintosh II in Lightspeed C. Software was written that reads in through a digital input-output board. Data from the newly installed Varian lon Gauge then displays and plots it on the computer screen. Software was also written that performs an experiment to measure the transient behavior of the source cell flux upon shutter opening. The purpose of these experiments is to obtain the data necessary to regulate the intensity of the flux and prevent it from decaying exponentially.

The flux compensation experiment was performed by heating the cell until it reached the desired temperature and then waiting for the temperature to reach steady state. The shutter is opened and the data is recorded. After the flux reached steady state, a step change in temperature is made and data is recorded. Using this data, a feed forward compensation technique can be tested in which the temperature setpoint is manipulated before and after shutter opening to maintain a steady flux.

A Macintosh IIfx ran the software written in Lightspeed C. This computer is interfaced to the Molecular Beam Epitaxy machine furnace controllers and sensors through digital, serial communication protocols.

The data was analyzed by reading it into Matlab. Matlab has a mathematical language that allows the data to be plotted easily. A sophisticated script file was written with a set of function files for analyzing the results of the experiments. To find the time constant of the exponential decay of the flux on shutter opening, the data is normalized so that it decays to 0. The log of each data point is then taken. The best straight line fit to the data is made by least squares. The negative reciprocal of the slope is the time constant. In order for this method to work, the period that the data was recorded has to be about three times the length of the time constant. This requires trial and error analysis of the data, using less or more data, until the size of the error in finding the time constant is acceptable. The Matlab function "fmins" that finds the minimum of a function of several variables (nonlinear optimization) is used to find the time constant.

The Varion lon gauge code works reliably. This software allows for greater precision in measuring flux.

QPA for Pulsed Laser Oblation

Task Order No. 44a
Student Support Program

Stephen Adams
Wright State University

21 April 1992

Guiding Engineer
Dr. Jim Malas
WL/MLIM

ACKNOWLEDGMENTS

I would like to thank the following people for their help in making my first quarter here very pleasant and rewarding: Dr. Walter M. Griffith, Chief, Manufacturing Research Branch, Dr. Steven LeClair, Visiting Scientist, Dr. Jim Malas, Materials Research Engineer, Mr. Oliver Patterson, Visiting Scientist, Capt. Elizabeth Stark, Mr. Jeffrey Heyob and Mr. David Tucker.

I also would like to thank the Student Support Program for allowing students the opportunity to work with the Wright Laboratory of Wright Patterson Air Force Base. I have found that the people in this group are very friendly and are willing to help you if you are having problems with your project. They also seem to go out of their way to make you a part of both the professional and the social groups that are formed at work. The exposure to technology and equipment the Air Force has to offer is an experience that I am glad to be a part of and is something that has changed my outlook on future job prospects.

TASK ASSIGNMENT

This Task was started on January 28, 1992 and completed on April 20, 1992. The Task was to provide programming support for Qualitative Process Automation of the Molecular Beam Epitaxy (MBE) process. The work was performed for the in-house manufacturing research group of the Materials Directorate at Wright-Patterson Air Force Base.

The MBE process is used to grow semiconductor thin-film on a substrate for electrical and optical applications. The process uses several materials such as: Ga, As, Al, Si and In. These materials are held in a Knudsen Cell and heated until they are in a gaseous state and released to bond to the substrate. The Knudsen Cell is composed of a crucible that holds the material, a shutter that released the flux into the chamber and a heater coil that is wrapped around the crucible. The flux is the name for the fluid form of the material in it's gaseous state.

The project goal was to control the rate of flux from the cell. The problem is that the flow of flux first starts out at a very high rate and decreases until it reaches a steady state value. The reason for this response is that the source material is hotter than the chamber because the closed shutter holds the heat of the cell in. Because the shutter holds the heat in, the surface temperature of the source material at the shutter is higher than the temperature of the source material in the back of the cell. This will cause an initial value of the flux release into the chamber, when the shutter is opened, to be greater than the amount of flux that will be released at the temperature of the source material at the back of the cell. This characteristic of the cell is shown in the graph below.

A feedfoward flux compensator is being develped to minimize the decay of the flux after the shutter is opened. This will be achieved by increasing the temperature of the material in the cell before and while the shutter is open. By measuring the flux response after the shutter is opened and after the temperature step change, a formula can be found to compensate for the flux. The amount of time it takes for the flux to respond to a temperature setpoint change is called the lead time for the compensator.

Maintaining a constant flux is very important because very tight control of the thin-film composition is desired. Because the flux transients for the various source materials have different time constants, minimization of the transient is the only solution.

When this project was started, an equation had already been developed for the feedfoward compensator. The equation is:

$$\Delta T_{\text{feedfoward}} = \frac{\Delta T \times \Delta F_D}{\Delta F_P} \left(1 + \left(\frac{t_P - t_D}{t_D}\right) \exp\left(\frac{-t}{t_D}\right)\right)$$

This equation is based on the understanding that the characteristics of the disturbance and step change are exponential responses. This equation works great when the response is an exponential curve, but when the curve is not an exponential, this equation is no longer valid.

The variables in the equation are: ΔT is the step change in the temperature, ΔF_D is the change in flux for the disturbance, ΔF_P is the change in flux for the step change, t_P is the time constant for the temperature step change curve, t_D is the time constant for the disturbance curve and t is time.

The problem arose when the disturbance and step change curves were not exponential curves, and for that reason the original equation could not be used to fit the curve. The idea that is currently being worked on is to use a third order equation to fit the disturbance and step change curves. The use of the third order equation will give the compensator a wider range of possible curves that it will be able to fit, therefore allowing it to compensate for the various source material that all have different characteristic curves.

Once the compensator is written and proven to work for a single cell it will than be test and modified to work on all the different cells. The final implementation of the compensator is to integrate it into the growth process so that the quality of the growths can be improved.

Some problems that are currently being faced are that the flux readings that have been collected have not had much repeatability. Each time the same test is run on different days the characteristics of the curves seem to change to the point where a general equation cannot be written to fit them all. One theory is that the chamber in which the readings are being taken is contaminated with Arsenic and this is causing high background pressure readings to appear on the Varion Ion Gauge. This might be the cause for the initial hump on the curve shown on the previous page. To attempt to prove or disprove this theory, measurements are going to be made on a different chamber that has not been contaminated and see how those readings compare to the current readings. The last problem, is that each source material has a different response curve and characteristics. Therefore it is very hard to write one equation to fit to all the different curves that each source material is known to exhibit even under idea situations.

The results so far have not been conclusive. Tests are currently being run to find out how different cells respond to shutter openings and temperature changes. After the data is collected and then analyzed, a model is to be built that will compensate for the shutter opening induced flux transient. Experiments are currently being run with the GA cell in hopes that the data collected will be stable and consistent.

The exponential curve compensator has been written, but has not been tested due to the abnormal characteristic of the curves. The third order compensator is in the process of being converted from a mathematical model into actual code. Once the conversion is completed, tests will be run to see how well it performs.

Rapid Design System (RDS)

Task Order No. 45
Student Support Program

David Lovely
Wright State University

Guiding Engineer
Dr. James Malas
WL/MLIM

ACKNOWLEDGMENTS

I would like to thank all the people whom I have worked with and who helped me achieve my objective for this Task:

Capt. Baxter Turner, Task Leader
Dr. Gerry Radack, Design Developer
Mr. Cave Domermuth, Manufacturing Developer
Mr. Randall Spratt, Manufacturing Developer

TASK ASSIGNMENT

This Task involved work on the Rapid Design System (RDS). The objective of this project is to develop a comprehensive software package that integrates the three major aspects of producing machine parts for Air Force use (design, manufacturing and inspection). Each of these three areas has its own set of parameters when examining a part. A designer sees a part as a set of features such as holes, pockets and slots. A manufacturer views the same part as a set of machining operations such as milling and drilling. The inspector gives regard to the part's dimensions and tolerances. The goal of the RDS is to develop a system that will give the designer, machinist and inspector their particular view of a part in a single part model.

The current version of RDS is based on two commercially available software packages. The first is Wisdom System's Concept Modeller. This is a CAD package that the RDS utilizes as its "engine." The Concept Modeller is written in the Lisp language. Lisp is well suited to artificial intelligence applications, which is a future goal of the RDS. The other package, used to generate machining data, is Met CAPP. This package, which is written in the C language, produces the process plans by which parts can actually be machined. It will eventually generate instructions that can be loaded directly into automated cutting machines. Both packages run on a Sun workstation network running Unix and the X-Window system.

The Task was originally to translate design-oriented features into manufacturing-oriented ones. The Task leader, Capt. Tim Westhoven, left and was replaced by Capt. Baxter Turner. Capt. Westhoven was responsible for setting-up and maintaining the network of Sun stations. After his departure, Dr. Radack was in charge of the design environment development. Thus the primary responsibility of the Task became system maintenance.

Also included was the development of various utility modules in both the design and manufacturing environments. This required developing software in both the Concept Modeller's Lisp environment and MetCAPP's C environment. The version of Lisp that was used included an interface module that allowed programs written in other languages to exchange data with Lisp programs. Several programs were written in C whose major function was to interface and be run by Lisp. This proved to be the most interesting aspect of the Task.

The results of this Task differed from the original expectations, but the outcome was pleasurable. Developing software modules for the manufacturing domain was originally expected, but acquiring a broad range of experience in not only software development but also in maintenance and administration was the result.

Experience working with personal computers (Macintosh) for documentation and reporting, Unix-based workstation (Sun) development and administration and programming experience in both C and Lisp in addition to the language interfacing was gained.

This Task was a great learning and application experience that will help in a software engineering career.

Analytical Process Modeling

Task Order No. 46
Student Support Program

Bryon J. Foster
University of Dayton

15 May 1992

Guiding Engineer
Mr. William O'Hara
WL/MLLN

ACKNOWLEDGMENTS

Thanks go to Vinod Jain of the University of Dayton, Mr. Robert Goetz and Mr. William T. O'Hara for all their assistance and guidance during the last year.

TASK ASSIGNMENT

The ALPID code is a program sponsored by the U.S. Air Force to simulate material processing. The simulations were performed on a VAX/VMS machine at Wright Patterson Air Force Base in Dayton, Ohio. The first, and one of the more important steps involved is to set the computer system properly to insure a reasonable response time and an equal distribution of the available resources. The quotas for each individual account are specified by the producer of the code, but a large amount of disk space and CPU time must be allocated for the simulations.

The ALPID code is broken into two sections, a heat transfer section and a deformation section that can be combined to simulate a non-isothermal deformation process. The validation of the heat transfer part of the code was done by simulating a real event and comparing it to a theoretical model. It was found that when a large billet to TiAL was placed into a furnace at a high temperature for heating, the resulting thermal stresses were enough to produce a crack in the billet. A theoretical model was generated for this specific case by using heat transfer equations for a cylindrical object. The equations were then solved to obtain a temperature distribution in the billet. An ALPID simulation was then performed to match the conditions of the model. The resulting temperature profiles were extremely close. These temperature profiles were then put into a program designed to find the stresses involved with temperature distributions. It was found that the stresses involved from the temperature distributions were great enough to produce a crack in the material.

To validate the non-isothermal deformation part of the code heat transfer data from Dr. Jain's previous work was used. Kunogi's method of determining friction factor in isothermal ring deformations was then extended to non-isothermal by using the ALPID code. Several simulations of non-isothermal ring compressions were performed using different friction factors. The inner diameter of the ring after the compression was then extracted from the simulations and calibration curves were created for various initial temperatures and material interfaces. Simulations were performed using Waspalloy for the die material and As-Cast IN 718 and Medium Grain IN 718 for the ring material. Additional simulations were also performed with H-13 tool steel as the die material and Fine Grain IN 718 as the ring material.

Actual ring compressions were then performed to match the simulated conditions and materials. The inner diameter of the compressed rings were then measured and plotted onto the calibration curves, which were generated from data from the computer simulations, yielding the friction factor between the ring material and the die interface for a specific starting temperature. The data collected from plotting the tests onto the calibration curves yielded friction factors that were within an expected range.

The friction factors obtained from the ring compression tests can be used in simulations that accurately simulate forging operations in more complex models. This would allow computer simulations to indicate whether certain forging operations are possible in a single step. It also would allow the designer to see temperature distributions in the materials during the process, which would be impossible to measure in actual operations.

Dynamic Oxygen Absorption Study

Task Order No. 47
Student Support Program

Mark Roselius
Wright State University

27 May 1992

Guiding Engineer
Dr. Harvey Paige
WL/MLBT

ACKNOWLEDGMENTS

I wish to extend my sincere appreciation to Dr. Harvey Paige and Mr. Ed Snyder of the Wright Laboratory Material Directorate for their guidance and assistance during the performance of this Task and with OXAB data analysis. I also would like to thank Dr. Jim Liang of UDRI for his assistance with infrared data analysis. Finally, I would like to thank Dr. Arthur Krawetz of Phoenix Chemical Laboratory for his assistance and for providing the OXAB reference manual.

TASK ASSIGNMENT

A two step program of oxygen absorption rate measurements (OXAB) was conducted on polyphenyl ethers. The first step, conducted during the latter half of 1991, consisted of a series of test runs on 7.5 - 8.5 gram samples of non-formulated (no additives) polyphenly ethers, 5P4E, at different temperature settings. This was done to determine what optimum temperature setting, with a 50 ml/min flowrate, would produce measurable absorption rates within a reasonable test length (2-6 days). The ultimate intention is to use the parameters and baseline developed to observe what changes various additive packages would make in the rate of oxygen absorption. This was done by running the experiments with a fluid containing antioxidant.

The second step, conducted during the first half of 1992, consisted of a series of tests using a variety of metals to determine what influence they have on the oxygen absorption rate of polyphenyl ethers. The results will hopefully offer insight into the potential corrosive affects on the metal surface in contact with the fluid and suggest whether anti-corrosion additives will be needed in the end use.

Finally, infrared data was collected in an effort to understand better the possible oxidation mechanisms involved in the OXAB studies.

Oxygen absorption rate measurements were conducted using the Phoenix Chemical OXAB instrument. Tests typically ran from 6 to 10 days and were conducted on 7.5 - 8.5 gram quantities of polyphenyl ether, 5P4E. The temperature settings were 260, 280, 300, 320 and 335°C with at least 2 runs per temperature. A flowrate of 50 ml/min was used. Data was recorded using a Rustrak Ranger Datalogger. Data analysis was performed with software provided with the datalogger and a custom software package provided by Phoenix Chemical, both run on an IBM XT PC.

For the metals analysis, the system described above was used with a temperature setting of 300°C with the addition of a metal coupon or washer of the appropriate metal to the sample fluid. These coupons were all the same dimensions. Consequently, surface areas were the same. The metals used are aluminum, copper, 410 stainless steel, titanium and M50 (a tool steel alloy).

Infrared data was initially analyzed with a Perkin-Elmer model 1750 FTIR. This proved to be limited in the amount of useful information that could be generated from the results. As a result, infrared analysis was shifted to a Hewlett Packard GC/FTIR and analysis confined to ambient trap products.

5P4E was found to be highly resistant to oxidation. Temperatures below 300°C produced an absorption rate so low as to appear to be zero within the time constraints of the test. In fact, one 280°C test ran for 3 weeks before termination. Background noise of the instrument interferes with the interpretation of the data over such long test durations where rates are particularly low. This was the case for the tests run below 300°C. Secondly, test durations of greater than 6 days provide no improvement over other methods of oxidation measurement. Consequently, any time advantage gained with this analysis method is lost, making comparisons of different runs are very time consuming at these temperatures.

A temperature setting of 320°C produced tests both with a reasonable duration of 6 days and with a significant oxidation rate that was not overly influenced by background noise. Tests conducted with the formulated polyphenyl ether (antioxidant additive) at this temperature revealed a marked reduction in oxidation.

A test conducted at 335°C reduced run duration to 4 days but lead to excessive vaporization of the sample.

The investigation of the influence of various metals on the oxidative process has opened up a new area of OXAB testing. While this investigation is not complete at this time, preliminary results of data collected through 140 hours revealed that aluminum has no discernable influence on the oxidation rate of 5P4E. M50, on the other hand, shows a substantial increase in the rate of oxidation during the first 24 hours before it falls off to a rate consistent with the baseline obtained with the virgin fluid. The copper test produced a substantial increase that appears to be sustained through 140 hours, however, an external system failure interfered with the data collection. As a result, copper will have to be rerun. Titanium and 410 stainless steel have not been examined as of this date.

The M50 result is not understood as of yet. A coating on the coupon, shiny brown in color, was observed and was removable with organic solvents. A possible explanation is that the products or reactants form a coating on the surface over a 24 hour period that acts as a barrier to any catalytic affect the metal might have. This time dependent reduction in surface activity would explain the drop in oxidation after the initial increase.

The aluminum coupon exhibited some visible signs of corrosion and what appeared to be copper salts were found in the bottom of sample tube. No surface coating was detected.

Fourier Transform Infrared analysis of sample tube, ambient trap and cold trap products was initially conducted using a conventional single beam instrument. Because of the thin films required coupled with the peculiarities of the 5P4E spectrum and the fact that the products were all mixtures greatly limited what useful spectral information could be seen. To get around this problem, a gas chromatograph with an FTIR detector was substituted. The GC performed the necessary separation of the various components of the mixture and the IR detector produced the needed spectra. In addition, the library function of the instrument was used as an aid in determining the possible structures of the products. This IR detector also could work with smaller quantities. Due to GC column limitations, only ambient trap products were used for this analysis. Also, GC/FTIR system failures lead to delays with the main result being that analysis was confined to products from metals tests.

This FTIR analysis revealed that primary products from the oxidation of 5P4E in the presence of metal are phenol, m-bis(phenoxy)benzene and phenoxybenzene. Unfortunately, the identity of the minor products could not be pinned down with any degree of confidence with IR. Further analysis should be conducted using GC/MC that will provide the necessary detail to identify the minor components.

NDSANDS Fortran Programmer

Task Order No. 48
Student Support Program

Hugh Cullin
University of Dayton

29 May 1992

Guiding Engineer
Dr. N. J. Pagano
WL/MLBM

ACKNOWLEDGMENTS

I would like to thank Dr. G. P. Tandon for his everyday guidance. Without his help, none of this project could have been undertaken. I would also like to thank Dr. N. J. Pagano and Dr. Tandon for initiating all the paperwork necessary for this Task. Dr. David Chu also must be thanked immensely for writing some subroutines that aided in the overall project. Lastly, I would like to thank the Student Support Program for giving me the opportunity to gain invaluable experience in problem solving which this Task has encompassed.

TASK ASSIGNMENT

The program NDSANDS, originally written on an IBM compatible machine, was taken in its IBM form and transferred to the Macintosh. The problem was not this simple, however. The software package Mactran was used for the Macintosh. The programming language was found to be slightly different on each machine. This caused some inherent problems that were not foreseen by Dr. Tandon at the outset. Dr. David Chu wrote some subroutines to help remedy this problem.

NDSANDS stands for N Directional Stiffness AND Strength. This program theoretically looks at materials called composites and determines how they will react to different stresses, shears and other forces. The strength of the different composites also can be determined from this program. A new composite also can be created by the user and it can be theoretically tested. If the user likes the results of the test, a laboratory test of the material can be performed next. If the material passes the lab test satisfactorily, perhaps that composite will be used on the next space shuttle mission, or on a new bomber. These are two possible, practical uses for composites.

The program itself encompasses many different subroutines contained in over twenty different files. The program was modified by critically examining each one of these subroutines individually. By doing the analysis in this way, problems were eliminated one at a time. A valuable tool in this process was the use of dummy subroutines set up to look at just a small portion of one subroutine. By looking at each part of each subroutine separately, problems could be analyzed much more quickly and subsequently eliminated. When one part of a subroutine worked properly, the next section could be analyzed. This process was repeated until the entire program, with all subroutines included, was working.

The next step was to make the program look better to the user. The large data files that appear time and again throughout the problem were put in even alignment. Much of the text that appears was also altered slightly to make the program more user friendly. The ability to exit the program from more places and go to previous screens was added to make the program more user friendly as well.

The main menu of the program includes several options. These options include the material library, the composite library, composite analysis, parametric study and the plots. The material library contains many different materials which may by used to create or change a composite. New materials may be added or changed. The composite library contains present composites. New composites may be created while old ones may be deleted or changed. These composites can have either a parametric or analysis study conducted on them. Each different test runs a composite through a different set of tests that can be varied by the user. The results of each test can then be viewed. Plots of each test also can be viewed by the user.

The project is now in its final stages. The program is running well enough that the all important number crunching part now works. This number crunching is the part that tells the user how the material will react to certain conditions, or if that particular composite will have a practical use. The program will be modified to make it look more neat and to be more user friendly. Several graphing subroutines still need to be modified. These subroutines will allow the user to view the results of a hypothetical test that was already numerically run by using the other options. The experience gained throughout the two plus years of working on this project has been invaluable. The skill of problem solving is the basic tool of every engineer, and this project has certainly helped to develop those skills. The software package that will come when this project is finished will aid in the very rapidly developing field of composites.

Modification of OP Films and Plasma

Task Order No. 49
Student Support Program

Gwendolyn J. Steele
University of Dayton

13 September 1991

Guiding Engineer
Capt. David Curliss
WL/MLBC

ACKNOWLEDGMENTS

I would like to thank WL/MLB personnel, Capt. Dave Curliss, Dr. Kate Webb, visiting scientist of the University of South Dakota, SOCHE contractors Mick Arnett and John Sawvel, as well as the following University of Dayton Research Institute on-site personnel; Marlene Hougz, Brian Rice, Jim Lute, Bill Price and Ron Cornwall for their timely and helpful instruction and guidance throughout this investigation.

TASK ASSIGNMENT

This investigation involved the analysis and characterization of a BASF NARMCO bismaleimide resin; 5250-4 using infrared and UV/VIS spectrometers. The area of interest for investigation being the time and degree of cross-linking within the resin during the cure and post-cure cycles since it is dependant upon the chemical structure of the components and lends to the unique properties of polybismaleimide resins and their corresponding advanced composites. Like other bismaleimide polymers, it was suspected that the cross-linking mechanism for the 5250-4 resin involved the opening of the double bonds of the maleimide ring. The loss of the maleimide double bond giving characteristic maleimide absorption bands at 685cm-1, 1150cm-1 and 1380cm-1. Specifically, a decrease in intensity of the 685cm-1 and the 1150cm-1 as a monitor of cross-linking (1, pg 18). In addition, intensification of imide related bands at 1785cm-1 and 1717cm-1, during the cure cycle, can be used to monitor degree of cure (2, pg 14).

The completed analysis involved locating any significant peak changes in the UV/VIS and infrared spectra, correlate them to structural changes in the resin and quantitate these changes over time and/or compare them to non-changing peaks as possible measures of mechanism and degree of cure. The most useful means of exploring these changes being in-situ cure monitoring using infrared spectroscopy.

Samples used for UV/VIS analysis were made using resin extracted from prepreg. For extraction, approximately 8" by 8" strips of prepreg were cut from a prepreg roll and placed over an aluminum pan. Acetone poured over these strips separated the carbon fibers from the resin, which collected in liquid form in the pan. The resin was further dissolved by the addition of 50-110 mls of acetone to the pan which was set overnight to allow the acetone to evaporate. The carbon fibers and/or unused resin were heated in a 100° F overnight then thrown away. Approximately two tablespoons of the resin is then dissolved in a minimum amount of acetone (approx. 10 mls) and placed dropwise (approx. 7 drops) onto 2" by 2" squares of previously coated kapton. The acetone is evaporated then the application is repeated a second time.

The prepared samples are placed in a 50^{o} F vacuum oven and degased under 30" Hg vacuum for a minimum of 20 minutes or until no gas bubbles are visible. Once degased, the oven temperature is raised to 350° F and samples are removed starting at 50 minutes, then every 5 minutes after that up to 90 minutes. One sample is cured overnight to use as a comparison to a fully cured sample.

Samples used for infrared analysis included both 5250-4 resin and it's powder backbone, 1-1'-(Methylenedi -4,1-phenylene) bismaleimide spread as thin layers between KBR plates using a razor blade. All samples were analyzed in-situ using a controlled heating cell, containing the KBR plates with either the 5250-4 resin or powder backbone. The temperature of the cell was raised manually 100F/min until a set temperature of 350°F, corresponding to the standard 5250-4 cure cycle (4, pg 2057), was reached. Once the set temperature was reached, spectra were taken automatically using a time delay computer program. The series of experiments was run for both the 5250-4 and the powder backbone taking spectra every 15 mins, once the set temperature was reached, for 80 mins for a total of 16 scans. The infrared spectra obtained were analyzed using difference spectra to monitor appearing or disappearing peaks by plotting changes in intensity vs. time (mins).

UV/VIS and infrared analysis was performed using a lambda 9 spectrophotometer and a Digilab "Bio-rad" FTS-7 spectrophotometer, respectively. UV/VIS spectra plotted absorbance vs. wavelength in the near-IR range of 185nm-2600nm. Infrared spectra plotted percent transmittance vs. wavenumbers (cm^{-1}) with a range of 500cm^{-1} - 4000cm^{-1}.

UV/VIS spectra were obtained for three repeated series of samples run at 350° F. Peak changes were somewhat reproducible, but were complicated by differing sample thickness and a varying baseline due to increases in general sample absorbance corresponding to the darking of the resin over time. Two peaks were observed to undergo noticeable changes throughout the 50 min-90 min cure cycle including peaks at 1834.2nm and 2060.1nm. The peaks 1834.2nm and 1364nm visible after 80 mins of curing were not found after curing overnight (see spectra 1 and 11). Identification of a non-changing peak in the UV/VIS spectra with which to do a peak-ratio study, was not obtainable due to problems with a drifting baseline and vibration overtones observed in the near-IR region analyzed.

Infrared spectra obtained for uncured 5250-4 resin showed characteristic peaks for known components, which correlated to characteristic peaks given by the uncured 1,1'-(Methylenedi-4,1-phenylene)bismaleimide powder backbone including; C=O at 1719.2 cm^{-1}, CH_2CH_2 at 2927.1-2972.8cm^{-1}, free N-H bending at 1520.7-1519.7cm^{-1} and aromatic C=C stretching at $3100cm^{-1}$ (see Spectra's III and IV). For the 80 mins in-situ 5250-4 resin study, an intensification in the imide-related bands of 1717.04 cm-1 and 1175 cm-1 was observed with R values of 0.912 and 0.902, respectively, (see Figures 1 and 2) as well as a characteristic decrease in absorbance intensity for the maleimide absorption band at 690cm-1 (see Figure 3) and an R value of 0.950. For the 80 mins in-situ resin backbone powder study (see figures 4 and 5), a similar intensification in the imide-related band of 1180 cm-1 and a decreasing absorbance intensity for the maleimide absorption band at 690cm-1 with R values of 0.857 and 0.950, respectively.

The similarity between spectra for uncured 5250-4 and it's powder backbone, 1,1'-(Methylenedi-4,1-phenylene) bismaleimide, allowed for conformation verification as to the basic components of the resin and allows for more accurate identification of comonomers, reactive diluents, processing additives, and catalysts. The appearance of the 1175.5cm-1 - 1180cm-1 band and the decrease in absorbance intensity for the maleimide absorption band at 690cm-1 in the 5250-4 resin using in-situ infrared spectroscopy are reported to be indicative of cross-linking (1, pg 19) during bismaleimide polymerization with the continuance of these trends (increase or decrease) showing that the resin had not been completely cured (see Figures 1, 2 and 3). The observance of similar changes in the powder backbone (see Figures 4 and 5) confirm the cross-linking mechanism to be the opening of the double bonds of the maleimide ring.

In the UV/VIS analysis of 5250-4, problems with a drifting baseline and vibrational overtones in the near infrared region analyzed allowed for only small observable changes in peaks and suggests a UV/VIS fiber optics probe might be necessary in analyzing similar resin matrix (polybismaleimides) including identifiable peaks (C=C 190nm, C=C-C=C 220nm, phenyl 270nm, C=N 190,nm, C=O 280nm) found in the more interpretable region of the 180-280nm. The use of an internal standard for a peak ratio analysis would seem to facilitate the use of such spectra in monitoring the mechanism and degree of cure.

The performance of in-situ composite cure monitoring using infrared spectroscopy for 5250-4 and 1,1'-(Methylenedi-4,1-phenylene)bismaleimide allowed for absorbance vs. time plots of specific peaks to indicate mechanism and degree of cure. Using infrared spectroscopy in the characteristic resin peaks and their changes during the cure cycle seems to be a more effective approach than UV/VIS spectroscopy and has already demonstrated industrial usefulness in combination with fiber optic probes in obtaining real-time (absorbance vs. temperature) cure monitoring data; using a few key bands and specifically in the mid IR-spectral range of 3250-1250cm-1(2, pg 11). Certainly, this portion of the analysis confirms the usefulness of such a probe for polybismaleimides with obtained difference spectra, and plots as useful references for future comparison to spectra obtained using a fiber optics probe.

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Plasma Modification of OP Films

Task Order No. 49a
Student Support Program

Michael S. Arnett
Wright State University

13 September 1991

Guiding Engineer
Capt. David Curliss
WL/MLB

ACKNOWLEDGMENTS

I would like to thank the following persons for their guidance and support in the execution of this study: Capt. David Curliss, William Ragland, Bill Price, John Sawvell, Gwen Steele and John Russell. Without these persons assistance I would not have been able to finish this task in a timely manner.

TASK ASSIGNMENT

This task has been divided into two different projects, these being: (1) measuring the specific gravity of several composite materials and, (2) examining the sensitivity of several advanced composite materials exposure to military jet fuel, JP-4.

EXPERIMENTAL PROCEDURES AND RESULTS

I. Specific Gravity Measurements

The purpose of this study was to measure the changes of the specific gravity for many advanced composite materials under varying temperatures and pressures. These changes in specific gravity are of particular interest to the Air Force in their pursuit to develop stronger and more flexible materials to be used in aircraft research and design.

The device used to measure the specific gravity changes, (known as a dilatometer), was purchased and installed by materials lab. Numerous calibration runs were executed in order to obtain proper instrumentation of the dilatometer. This device is currently being used to measure the specific gravity of common composite materials, namely APC2 and HTA.

The month of August was spent dismantling the dilatometer and replacing the sealing rings along with other general maintenance.

II. JP-4 Exposure on Advanced Composites

The sensitivity of two advanced composite materials (APC2 and HTA) exposure to military jet fuel, JP-4, was studied. This was a carry over study from last year's task. The previous objective was to investigate how commonly used plastics would be affected after continual exposure to jet-fuel. This study focussed on examining the materials for fuel absorption and any significant changes in their mechanical properties. The present study is being conducted in an attempt to reproduce the previous study's results. At present time, the materials have been processed into [+/-45]2s laminates and cut into standard geometry coupons. The coupons were exposed to JP-4 @ 180°F for a total of 1680 hours and gained the predicted amount of fuel, less than 1% weight gain. The coupons are presently waiting to be mechanically tested.

A second study was started in July to compare how different processing techniques could affect the absorption of JP-4. The same materials, APC2 and HTA, were used under three different processing methods.

The first method utilized was the standard process of slow cooling the material after its initial fabrication (the material's temperature is over 700°F and cools for 3 hours). The second method is known as "quenching". This method toughens the material and involves heating a material up to its fabrication temperature and then quickly cooling it down (about 5 minutes). The third method is known as "annealing". This method reduces inner stresses in the material and is similar to quenching but the temperature used is much lower.

Tensile test coupons (specimens) were cut from each material and half of the coupons were then exposed to jet fuel @ 180°F for 1600 hours. Routine checks were made into fuel absorption and surface degradation. At this time the coupons are awaiting mechanical testing.

CONCLUSIONS

Further work will be conducted on measuring the specific gravity changes for various advanced composite materials in order to produce enough results for a comparison study. The jet fuel soaked coupons are awaiting mechanical testing. Upon testing completion, the results will be compared to those obtained from the previous study.

Hot-pressing of Ceramic Matrix Composites

Task Order No. 50
Student Support Program

Brian Hornback
Wright State University

23 June 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Dr. Randall Hay for his guidance in completing my Task assignment. I would also like to thank Mr. Ed Hermes and any other scientists who assisted me in my responsibilities.

TASK ASSIGNMENT

The Task included the synthesis of ceramic sols and gels used for coating ceramic fibers. These coated fibers were then used in the production of ceramic composites that would then be tested for their properties. Sol and gel synthesis comprised was a time consuming part of the task. This work involved the use of ordinary laboratory equipment (glassware, ph. meters, thermometers, heating lamps, bunsen burners, etc.) and a variety of chemicals such as organic solvents, mercury catalysts, aluminum, gallium and others. A thorough knowledge of the risks associated with these materials was required, which mandated the use of safety handbooks and other references. Safety in the laboratory was stressed and practiced.

One the ceramic fibers were coated with a sol or a gel it was of interest to examine the fiber-coating interface with a microscope. Thus, the majority of time was spent in the metallography laboratory preparing thin cross sections of the coated fibers for examination by electron or light microscopy. This involved mounting the fibers in a hard matrix, then grinding and polishing the sample to a thickness of 20 to 30 microns.

The building and wiring of a high temperature furnace was also included in the task. Also the construction of a high temperature clam shell furnace for fiber coating was involved. This project was not completed at the termination of this task assignment.

Hot Pressing of Ceramic Matrix Composites

Task Order No. 50a
Student Support Program

William Davis
Wright State University

23 Jun 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLN

ACKNOWLEDGMENTS

I would like to thank Dr. Ronald Kerans for his guidance. I would like to thank the Student Support Program for giving me the opportunity to gain invaluable experience.

TASK ASSIGNMENT

The objective of this Task Assignment entails the production of YAG (Yttrium Aluminum Garnet) powder that is used for the development of high temperature composites used for engine parts. It also involves the development of new hot pressing techniques and several other processing procedures or methods that produce dense ceramic matrix composites with very small flaw sizes. The responsibilities of the student in relation to this objective included assisting with the fabrication of these dense ceramic matrix composites. Duties involve laying up tapes, impregnation with matrix and hot pressing in graphite die. Other duties involve assisting with metallography for characterization of the composites.

Even though the Task objective and responsibilities consist mainly of techniques and procedures stated above, the student has been primarily responsible for the development of YAG powder. The responsibilities with this development involve several organic, quantitative and qualitative chemistry steps that leads to the trial production of what is believed to be a course product of the YAG powder. The procedures used are very effective, however tedious, due to the physical and unpredictable nature of YAG.

The responsibilities also included the synthesis of solutions known as metal alkoxides that are the initial steps in the production. These metal alkoxides consist of Aluminum and Yttrium metals completely reacted or refluxed for many hours in separate flasks of Isopropol Alcohol and a catalyst of Mercuric Chloride. Once these reactions are successfully completed, with no traces of metal evident, a filtration procedure follows shortly thereafter. This procedure only involves the filtration of the completely reacted Yttrium alkoxide solution. The Aluminum alkoxide solution is excluded because it is less concentrated and does not produce a sediment in contrast to the solution of Yttrium. Shortly after the filtration procedure, the filtered Yttrium Alkoxide solution is canulated or transferred and mixed with the other alkoxide solution that contains Aluminum. This final solution is known as a mixed alkoxide solution that is then heated or refluxed for a half-hour in order to form a more stable product for hydrolysis (addition of water). After the solution has been hydrolyzed, another filtration procedure occurs that involves the washing of the solution with acetone and de-ionized water several times. The final solution is then calcined or heated in a programmable oven at a temperature range that leads to the formation of a solid. The solid crystals that are formed are subject to a series of tests such as x-ray crystallography to determine whether or not YAG has been produced.

During this Task much useful experience, knowledge and exposure to the research and development scientific world was gained. Even though the Task wasn't completely chemistry, the correlation that exists between chemistry and the materials' world was seen. Exposure to new laboratory techniques that include Pressure Filtration, Hot pressing and Metallography was also gained. This Task has been a valuable experience that has sharpened laboratory skills and enhanced knowledge of chemistry applied in the material world.

Characterization of Ceramic Matrix Composites

Task Order No. 51
Student Support Program

Heather Walls
Wright State University

13 September 1991

Guiding Engineer
Dr. Paul Jero
WL/MLLM

ACKNOWLEDGMENTS

My deepest appreciation and thanks goes to Dr. Jero for all of his insight and guidance. The knowledge he shared with me is unobtainable in school, and I am grateful for it.

TASK ASSIGNMENT

In order to design composites with specific needs, one must be familiar with the properties of the fiber/matrix interface. The interface can dictate whether or not the composite will be able to support an applied load. Especially in ceramic matrix composites, where the fiber and matrix are both brittle, the interface can prevent the composite from failing catastrophically such as a monolithic ceramic. In other words, with the right type of interface, the fracture toughness of the composite can be increased.

One important consideration in the design of composites is the effect of the environment on their behavior. The objective of the research performed was to show that moisture decreases the peak load and the frictional stress at the interface by degrading the interface. Push-out tests were performed on three sets of composites containing SCS-6 monofilaments and potassium-borosilicate glasses. The compositions of the matrices were chosen so that the coefficient of thermal expansion (CTE) varied from slightly less to slightly greater than that of the fiber.

The composites were initially kept in a desiccator; fibers were pushed until de-bonding and immediately re-pushed for a 0 hour control. Each specimen was then put into one of three environments: dry, moist, or wet. For the wet environment, specimens were submerged in distilled water; for the moist environment, the specimens were enclosed in an area that contained a wet sponge. The dry specimens were kept in the desiccator. These same fibers were re-pushed after 1, 4, 24, 48, 72, 96 and 168 hours.

For the most part, the longer the composite was exposed to its environment, the average frictional load decreased. In composite F (residual compression) the moist average frictional loads were higher than the dry at low exposure times; the loads seemed to saturate at 120 hours. In composite D (residual tension) the loads decreased from dry to moist to wet and all saturated around 96 hours. In composite E (zero residual stress) saturation occurred around 72 hours without much difference in the magnitude of the dry and moist loads.

In composites that were exposed to a moist environment, the stick-slip behavior seemed to decrease slightly in composite E and decrease after 6 hours and reappear after 48 hours in composite F. In composite D, the fibers exposed to a moist environment had frictional loads that decreased after 168 hours of exposure time.

Subsequently, fibers that had been exposed to moist and wet environments for 1-6 weeks were pushed to observe the effects of long term exposure on measured peak load. The results were a decrease in peak load as a function of exposure time as expected.

Further work will be needed in order to better understand the effects of the moist and wet environments on stick-slip behavior since these results did not agree with previous work.

Synthesis of Powder and Coatings Solution Precursers

Task Order No. 52
Student Support Program

Cynthia Kurek
Wright State University

31 December 1992

Guiding Engineer
Dr. Ron Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Ron Kerans and Randy Hay for their guidance and direction in the project. My gratitude also goes to Mike Scott and Eric Fletcher for their assistance in metallography and to Cameron Begg for his help on the SEM.

TASK ASSIGNMENT

The Task was performed in conjunction with Dr. Randy Hay. He is investigating the kinetics of the YAP (Yttria-Alumina-Perovskite) - Alumina eutectic transformation to polycrystalline YAG (Yttria-Alumina-Garnet). This reaction is very interesting as it is accompanied by an 11% volume increase. The volume increase leads to the possibility of a fiber material that would experience large compressive stresses, which is desirable in composite design.

Small pieces of YAP-Alumina eutectic were heat treated for selected times and temperatures, looked at the samples in the SEM (scanning electron microscope) and prepared them for the TEM (transmission electron microscope). Cubes of the eutectic were heated in a furnace between 1100° and 1400°C for 6 to 6000 minutes. The samples were then cut in half, mounted and polished. The ones with large amounts of the YAG reaction product were looked at in the JEOL 840X scanning electron microscope. Photographs were taken, showing where and how much of the material had transformed.

All samples, whether they were looked at in the SEM or not, were made into thin foils for TEM inspection. This involved gluing the polished surface to a glass slide, cutting off the excess mount and polishing the backside of the sample to a thickness of 20 microns. The sampled were then covered with copper grids, cut into 3 mm disks and ion milled. The foils were then studied in the TEM and photographs were taken.

All the photographs were looked at, both from the SEM and TEM, and reaction thickness was measured as a function of time and temperature. This data is currently being analyzed by Dr. Hay to calculate activation energy, grain boundary diffusion coefficient and other constants related to the reaction. Other future work involves heat treating several larger pieces and looking at them in the STM (Scanning Tunneling Microscope) to check for possible relationships between surface relief and amount of reaction.

A graph is attached showing current results of average reaction thickness for the various times and temperatures. Several heat treatments are not shown, as the TEM foils are still in production. Results from this study are to be presented at the annual Ceramic Society meeting to be held in Minneapolis in mid April.

Synthesis of Powder and Coating Solution Precursors

Task Order No. 52a
Student Support Program

Cynthia Emerick
Wright State University

31 December 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Dr. Paul Jero for his guidance during this year. He has helped me understand the significance of the research I assisted with this year. It is truly amazing how the scientific ideas arise into progress in the research laboratories. I also would like to thank Dr. Ronald Kerans for allowing me to be involved with such a group. The experience working in the environment of Wright Patterson Air Force Base and interacting with the people has enhanced my future in the engineering field.

Other people that I would like to thank in other departments are Mike Scott, Bob Lewis, and Eric Fletcher. They have all helped me adapt to all the metallography equipment. I also thank all other facility members of WPAFB. I have learned so much from everyone.

TASK ASSIGNMENT

The properties of the fiber/matrix interface in ceramic matrix composites (CMC's) are a current interest in the research of the Ceramic Division at WPAFB. These properties are important factors in determining the mechanical behavior of CMC's. A number of techniques have been advanced to measure interfacial properties by a push-out or push-in fiber testing method. Fiber push-out and push-in tests have been used to examine the interfacial behavior in a variety of ceramic composites. Numerous push-out tests have been performed and the data analysis calculated so that the interfacial behavior of CMC's can be evaluated. This process of making and preparing the samples to be tested, testing the samples, and then evaluating the properties and behaviors of the composites are all part of the responsibility held in the Ceramic research department. These behaviors will be very important for determining the methods of use of ceramic matrix composites.

There are several theoretical techniques to measure the interfacial parameters of fiber/matrix composites. Selected techniques are being evaluated together to find the significance of each and to estimate their accuracy and repeatability. These properties of the fiber/matrix interface are recognized to play a key role in controlling the behavior of ceramic matrix composites. Interfacial debonding requires a relatively weak interface between the matrix and the fiber; therefore, different coatings on the fibers are tested to find the coating that gives the best properties. Numerous characteristics are being measured so that conclusions of the composite can be made.

Knowledge of most of the equipment is necessary in the different labs. In the metallography lab, the use of the polishing equipment, microscopes, photography, and chemicals is a basis for preparing specimens for testing. Another piece of equipment that is introduced is the push-out tester. The fiber push-in/out apparatus includes a loading probe mounted on a load cell and an X-Y stage on a slider that allows precise positioning of a sample. The use of a microscope and a video camera allows the sample to be viewed on a video monitor. The specimens' fibers range from 15 μm to 25 μm in size; therefore the accuracy of the positioning is important. This entire apparatus is mounted on a universal test machine that provides a constant displacement rate and load. These tests are performed with two types of loading probes, a tungsten carbide cylinder or a truncated diamond cone. After the tests were performed a series of data analysis was done on different computers, different programs and different ideas. Therefore, a background in computers is useful in the engineering field. The tests for this report have all been performed and the majority of the analysis have been calculated. The analysis begins with subtracting the compliance of the testing system from the raw data so that the data holds only the behavior of the fiber debond. Results of this type of research are only as good as the analysis of the data. Much more still needs to be learned on this subject to derive conclusions; but the continuation of guided experiments and interpretation can only yield answers in the discrepancies of research. The processes of research techniques are an important factor that is taught a student working at WPAFB.

Synthesis and Characterization of Polymers

Task Order No. 53
Student Support Program

David Flora
Wright State University

23 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to recognize the following people for their assistance with my research at WL/MLPJ: Dr. Phillip Cruz (thesis advisor), Dr. Tom Cooper, Dr. L.V. Natarajan, Dr. Zibgnew Tokarski, and follow students: Teresa Wilson, Tim Bunning, Kelly Minster and Ernie Fuentes.

TASK ASSIGNMENT

This Task involves the synthesis and characterization of polyamino acids (peptides) for possible use as matrices for optically active compounds.

In previous work our group focused on aligning NLO molecules by droping them into liquid crystal matricies. Recent work has focused on the direct attachment of NLO molecules to amino acid side chains of peptides.

To produce the peptides, Solid Phase Peptide Synthesis is used.

The chemistry chosen to synthesize the peptides is based on the work of E. Atherton and R.C. Sheppard at Cambridge University, England.

It is based on an orthogonal protection scheme (to prevent side reactions among the various amino acid residues) using 9-Fluorenylmethoxycarbonyl as the base sensitive alpha amino blocking agent and tert-butyl based side chain blocking groups. Some amino acids require more elaborate side chain protection, for more information see SOLID PHASE PEPTIDE SYNTHESIS a practical approach, by E. Atherton and R.C. Sheppard, IRL Press, 1989. The tert-butly based blocking groups are acid sensitive, and in combination with the Fmoc group, enables the preferential removal of Fmoc groups during synthesis. The result is a reduction of side reactions (deletion of an amino acid from the sequence) that occurs during synthesis. The peptide bond formation is facilitated through the activation of the carbonyl carbon with pentafluorophenol and the catalyst 1-hydroxybenzotriazole.

The polymeric support, on which the peptide molecule is built, provides the easy separation of product (peptide), and reactants not used by simple washing with solvent.

The methods for amino acid, peptide and protein characterization have been well established for many years, and are: (1) compositional amino acid analysis, (2) reversed-phase high pressure liquid chromatography (RP-HPLC), (3) ion-exchange chromatography, (4) gel filtration and (5) amino acid sequence determination. However, these techniques must be adapted to the instrumentation of individual labs.

The Biotech lab is focusing on RP-HPLC, Compositional and Sequential Amino Acid Analysis for the characterization of peptides produced. Each is performed on a high pressure liquid chromatography (hplc) and requires the development of individual methods for each technique. In addition to the separation and identification of component peaks by hplc, the compositional amino acid analysis requires a pre-column derivatization of the amino acids by the PTC-derivative. The derivatized amino acids for sequential analysis are produced by the ProSeqencer protein sequencing system (Milligen/Biosearch) and are known as PTH-amino acids.

Work to date has focused on the development of hplc separation methods for the PTC and PTH-amino acids, and the production of PTC-amino acids so that peptides produced can be correctly analyzed.

The quality of a separation method was judged by visual inspection of the chromatograms generated, and was based on finding a stable baseline and baseline separation of component peaks.

The ground work has been completed for the separation conditions for PTC and PTH-amino acids, and production of PTC-amino acid derivatives.

The separation of all standard PTC-amino acids is not perfect and contains reagent peaks leftover from the pre-column derivatization.

The separation of all standard PTH-amino acids occurs in approximately 15 minutes, but the baseline is not stable. It shows a steady increase in absorption as the percentage of acetonitrile increases during the run.

It is critical that all reagent peaks and unsteady baselines be removed in order to be able to quantitate the peak areas and elution times. These are used to generate ratios of one amino acid to another (compositional analysis), and amino acid sequence (based on elution time).

Synthesis and Characterization of Polymers

Task Order No. 53a
Student Support Program

Karen Hussong
Wright State University

23 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Nat Natarajan and Dr. Tom Cooper who have directed and assisted me in developing the goals of my Task. I also would like to thank Laura McGivern, Tim Grinstead and Sungmee Yoon who have been helpful in various aspects of my research. A special thanks to Sungmee who demonstrated how to use most of the instruments in the laboratory.

TASK ASSIGNMENT

The objectives of the Task were to synthesize and characterize biopolymers that contained nonlinearly active molecules. This involved coupling reactions with polypeptides and nonlinear optical (NLO) compounds. This was followed by characterization procedures, that involve various measurements of the compound's properties to compare with starting materials. Using this information, conclusions about the possible NLO properties and applications of these materials can be formed.

Using coupling reactions, compounds containing non linear optical properties such as, Disperse Orange 13, 1-pyrenyl methanol, and 4-nitrobenyl alcohol are attached to a polypeptide backbone. The polypeptides used are poly-L-Glutamic acid. These are attached using synthesis methods referred to as DCCI coupling and transesterification. The lab equipment and glassware for the reactions include the following. A reaction vessel, condenser, a filtering device, a stir/hot plate and a centrifuge were all needed in the process. Purification involved repeated product precipitations.

Analysis of the purified product included a melting point apparatus, FTIR (Fourier Transform Infrared Spectrometer), TLC (Thin Layer Chromatography), Circular Dichromism spectrometer and UV-Vis spectrometer. The TLC, UV-Vis spectra and the FTIR spectra of the products were compared to the starting materials. This comparison often could prove substitution occurred and the purity of the product. Finally, solutions and thin films of the substituted polypeptides were made to be tested for grating and second and third harmonic generations efficiency. It has been found that for the thin films 100 percent modification of the polypeptides gives the greatest alignment. Therefore, once the percent modification is determined, another substitution reaction can be performed in order to increase the ability to "pull," which in turn increases the alignment. Furthermore, increased alignment improves nonlinear optical properties.

Using the UV-Vis spectra, the percent modification of the polypeptides was calculated. The conformation of the backbone of the substituted polypeptides was determined using the CD spectrometer. An Alpha Helix structure was determined for the poly-L-Glutamate substituted with 1-pyrenyl methanol. From the analysis of the products, it was found that the coupling reactions are a successful method of coupling polypeptides with nonlinear active materials to produce a polymer that retains the nonlinear optical characteristics of the attached groups.

Synthesis and Characterization of Polymers

Task Order No. 54
Student Support Program

Kelly Menster
Wright State University

23 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to express my appreciation for Dr. Zbigniew Tokarski. His leadership and guidance have helped prepare me for a career as a professional. I am also thankful to have worked with Tim Bunning these past two years. His determination and standards of excellence in research have influenced my research practices as well. Also, thanks to Dr. Tom Cooper for his enthusiasm and organization in the activities of the Biotech lab. Dr. Crane truly deserves acknowledgment as well for his hard work in maintaining the operations of the research laboratories and for his concern for the well-being of those who work there.

TASK ASSIGNMENT

Liquid crystalline materials were synthesized and characterized by various techniques. These materials were siloxane-based polymers and oligomers containing mesomorphic side groups. The packing behavior and thermal characteristics were studied in order to define trends in behavior with the types of side chains that were attached. The results of these studies may be used in determining the behavior of a nonlinear molecule in such a system. The goal of these studies is the development of materials with nonlinear behavior for defense applications.

Siloxane polymers and oligomers were purchased as used without further modification other than a distillation. Biphenyl-4-allyloxybenzoate (B4AB) and cholesteryl-4-allyloxybenzoate (C4AB) were synthesized by reaction of allyloxybenzoic acid with hydroxybiphenyl or cholesterol using the DCCI/DMAP coupling reaction. B4AB and C4AB were attached to the backbone of polymethylhydrosiloxane (MW=1800) or lower molecular weight methylhydrosiloxane materials by hydrosilation using dicyclopentadienylplatinum(II)chloride as the catalyst. The relative amounts of the side chains B4AB and C4AB were varied in these reactions to produce different types of liquid crystals. Nematic, cholesteric and smectic liquid crystals were obtained depending upon the relative amounts of the two side chains.

Transition temperatures were determined using a Perkin Elmer DCS-2C differential scanning calorimeter. An Ortholux II POL-BK microscope equipped with a Mettler FP82HT hot stage was used to identify characteristic liquid crystalline textures. A Perkin Elmer 1725X FTIR was used to monitor the hydrosilation reactions by observing the disappearance of the Si-H band at 2150cm^{-1}. A Bruker 360MHz NMR was used to verify that the mesogenic side chains had been attached in the appropriate ratios. A Varian Vista 6000 gas chromatograph was used to characterize these liquid crystalline products as stationary phases in capillary gas chromatography.

Siloxane products containing all biphenyl-based side chains were nematic liquid crystals. Those with 15% - 60% cholesterol-based side chains (the remainder being biphenyl-based side chains) exhibited cholesteric mesophases. Products with more than 60% cholesterol-based side chains exhibited smectic mesophases. These trends remained the same for each siloxane system (polymeric or molecular weight materials). Products with more cholesterol obtained more ordered liquid crystalline states. At the same time, the bulky cholesterol group hindered the side chains from becoming closely packed together.

These siloxane materials have both common analytical and defense related applications. The polymeric liquid crystals are useful as stationary phases in capillary gas chromatography for the separation of polycyclic aromatic hydrocarbons (common environmental hazards). Furthermore, the characterization of these systems of liquid crystalline siloxanes has led to the synthesis of similar materials with side chains possessing nonlinear optical activity.

Synthesis and Characterization of Polymers

Task Order No. 55

Laura McGivern
University of Dayton

23 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to thank Dr. Zbigniew Tokarski for his time and effort in guiding me towards the goals of my Task. He was very helpful in explaining the meaning and the purpose of my Task as well as instructing me on how to properly use much of the equipment in the labs. Tim Bunning, Kelly Menster, Sungmee Yoon and Keith Obermeier were also especially helpful in assisting me in my research.

TASK ASSIGNMENT

The objective was to synthesis and characterize nonlinear optical (NLO) materials consisting of chromophores and mesogens attached to cyclic siloxane based polymers. The synthesis involves physically making the materials from other chemical compounds in the lab. the characterization process involves studying the materials with various instruments for different properties in order to draw some conclusions about the physical and optical behavior of the newly synthesized materials.

The mesogens, biphenyl and cholesterol, and the nonlinear optical chromophore, disperse red one dye, are considered side chains being attached to the polymer "backbone," which is the cyclic-penta-siloxane molecule. Initially, the chromophores and mesogens were synthesized. Later they were attached to the cyclic-penta-siloxane molecules in different ratios for a total of twelve siloxane synthesis reactions.

The equipment used for the synthesis includes glassware for the distillation set-up, and glassware for filtering to recover the final product. The equipment involved in the characterization procedure is much more extensive. It includes both a Fourier Transform Infrared Spectrometer (FTIR), and a Thin Layer Chomatography (TLC) set-up for monitoring the extent of reaction, and the extent of purity of the material synthesized.

A differential scanning calorimeter (DSC) is used to do some thermal analysis on the materials and to determine where specific transactions, such as a glass transition point, occur for each material. X-ray diffraction experiments are then done on each material to determine the physical structure and design of the material. Finally, thin films of the material are made so that second harmonic generation (SHG) tests can be run on them, and then the optical, in addition to the thermal and physical properties of the materials can be studied.

It was discovered during the synthesis that the higher the ratio of Disperse Red One to Cholesterol and Biphenyl, the lower the final product per cent yield would be.

Synthesis with 100% side-chain attachment, however, can be easily obtained. During the DSC analysis, it was determined that a second order phase transition (or a clearing point) in the material containing 50% cholesterol occurred near 500 Kelvin indicating multiple liquid crystalline phases. The glass transition temperatures of the materials were all nearly the same around 320 Kelvin.

X-ray studies showed that the primary reflection increases with Disperse Red 1 concentration, and that Disperse Red 1 is a larger than Biphenyl and requires larger separation distance. Changing the mesogen ratio did not significantly affect the macroscopic structure (x-ray analysis) or the phase behavior (DSC) of the materials. The thin film studies have shown that colored, glassy films of good optical quality can be produced with the siloxane materials. Studies continue in order to determine other physical and optical properties of the materials.

Synthesis and Characterization of Polymers

Task Order No. 56
Student Support Program

Ernesto Fuentes
University of Dayton

10 July 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank all the personnel at the Wright Labs for providing a stimulating environment and amicable workplace. Particularly, I thank Dr. Tom Cooper and Dr. Ruth Pachter for their patience and guidance during my tenure with SOCHE.

TASK ASSIGNMENT

The project was to synthesize and characterize peptide backbone molecules that will act as skeletons for nonlinear optical chromophores. Peptide synthesis will be done using solid-phase methodologies. Additionally, molecular modeling of these peptides in conjunction with or prior to synthesis will be completed.

Previously, a leucine/lysine heptamer peptide was synthesized using FMOC chemistry. This peptide was characterized via HPLC and capillary electrophoresis techniques. Currently, this peptide is being derivatized with the non-linear optical dye, spiropyran. This dye is unique because it displays distinct properties when exposed to light. Thus, potentially, this derivatized peptide could act as light controlled non-linear optical material. In order to predict how the structure of the peptide may react to dye attachment it was desirable to investigate the problem theoretically. During the last year, I have developed three-dimensional models for derivatized and non-derivatized heptamer peptides.

The process of modeling peptides and peptide derivatives for structural properties has been used in the past with much success. Our goal was to model the aforementioned peptides to determine their suitability as non-linear chromophore hosts. To this end, several computational schemes were incorporated to develop a novel approach in solving the tertiary structure of a peptide using only secondary structure data. The tertiary structure represents the three-dimensional topology of the peptide whereas secondary structure defines the backbone of the structure.

A few strategies have been developed to enable the incorporation of distance constraints (usually derived form NOEs), along with covalent bond and bond angle information to Cartesian coordinate space to obtain a three-dimensional topology. Recently, the PROTEAN methodology was introduced (Altman et. al.) for protein solution structure determination. This method uses the principles of Bayesian probability, namely in double-interated Kalman filter procedure (DIFK), to sequentially refine estimates of the mean position and the variance of each atom, thus providing a set of atomic positions consistent with the applied constraints, as well as an explicit quantification of the uncertainty in atomic position for each segment of the protein. The technique was shown to perform well for deriving a structure form NMR data of the cyclic peptide Cyclosporin A, the lac repressor headpiece, both the secondary structure and tertiary of the trp repressor, and the Kunitz inhibitor domain of the amyloid precursor protein involved in Alzheimer's disease. We have used secondary structure along with the DIFK and CHARMm molecular dynamics to develop and analyze a detailed three-dimensional model of a leucine/lysine heptamer peptide and derivatives with the dye spyropyran.

To begin the modeling process, it was necessary to learn and become proficient with state-of the-art computer systems and computer software packages. Initially, the UNIX computer operating system was learned. This allowed easy access to the various programs (PROTEAN - part II) on the Cray supercomputer necessary for modeling the task. Additionally, the software package Quanta was learned. This package is a well established molecular modeling program that interfaces molecular mechanics and dynamics calculations with high resolution molecular graphics.

The leucine/lysine heptamer [Leu-Lys-Leu-Lys-Lue-Lys-Leu] peptide primary sequence was built using programs contained within PROTEAN - Part II. Using the Double Iterated Kalman Filter (DIFK) methodology a mean three-dimensional structure consistent with experimental data was calculated, as well as the variance and covariance of all Cartesian coordinates. The input to this program consisted of distance and the angle constraints in the from of mean values along with associated vaiances. In addition to the normal bond lengths (variance = 0.1) and bond angles (variance = 0.1), we provided dihedral angle constraints corresponding to the ranges allowed values for Φ and psi backbone angles for an anti-parallel ß-sheet (Φ = -139 mean, variance = 25; psi = +135 mean, variance = 25). Prior to dynamics analysis, a N-carboxyethyl spiropyran dye molecule was covalently attached via a peptide bond to each lysine using the facilities within Quanta/CHARMm. Non-polar hydrogen atoms were implicitly included by adjusting the parameters of the heavy atom bound to them and polar hydrogens were added. In acetyl and amino groups, respectively in order minimize de-stabilizing interactions they may cause.

Further refinement of the three-dimensional structures, and the theoretical study of the dynamics of the macromolecular system, was carried out by using the molecular mechanics program CHARMm. This involved an initial energy minimization using the CHARMm force field energy function and subsequent integration of the classical Newtonian equation of motion to determine the position of atoms at later time points. The CHARMm force field energy function contained all default terms, excluding hydrogen bonding. In addition, a dielectric constant of unity was used for energy minimizations and dynamics simulations. Routinely, 10,000 steps of the Steepest Descent (SD) energy minimization followed by Conjugate Gradient (CG) minimization until convergence (less than 10,000 steps) was employed prior to molecular dynamics simulations. The peptides were initially heated form 0 K to 300 K over a 5 psec span. Equilibration and simulation periods were preformed at 300 K for 10 psec and 50 psec, respectively. Over the 50 psec dynamics simulation, 1000 structures were written out for future analysis.

Overall, the change in tertiary structure of the peptide due to dye attachment was not significant. Each version - closed, open and underivatized - maintaining phi and psi angles close to that of an ideal anti-parallel ß-sheet. Side chain torsions exhibited distinct transitions unique to each derivatized structure.

Currently, an anti-parallel ß-sheet consisting of two underivatized and one derivatized molecule is underway. This study will enable one to predict how these molecules might pack when spread on a surface. These studies along with continued experimental work should provide enough data to judge the suitability of this derivatized peptide as a non-linear optical material.

Synthesis and Characterization of Polymers

Task Order No. 57
Student Support Program

Teresa Wilson
Wright State University

23 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Zbigniew Tokarski for his help and guidance during my contract. I would also like to thank Dr. Robert Crane, Mr. Tim Bunning and Dr. Tom Cooper for their support and advice.

TASK ASSIGNMENT

The object of my task was to assist in the programming and networking of computer hardware with the analytical instruments of the Material Laboratory's Biotechnology Lab, which will aide in the research and development of nonlinear optical material.

One of the main jobs during this portion of the task was to create a LabVIEW program to manipulate data collected from a rotating stage and laser reflection detectors. LabVIEW is an icon based language which can be used to control the I/O ports of a Macintosh computer. My job was to incorporate LabVIEW subprograms created by Keith Andrews into a larger outer program which allows the user to control the movement of the instruments. The program intializes the instruments and allows the user to input the movement factors, such as velocity, stepsize, etc. The program then moves the instrument inputs, and input ratios.

Another main job was the upkeep and maintenance of the laboratory's computer system. There are three Macintosh computers and four IBM compatible computers used by the lab personnel. Also, there are several Vax and Unix accounts on Mainframe computers which need occasional upkeep. These systems were backed-up and updated with new software.

I am very thankful for the opportunity to work in a research environment where I can put the skills I have learned to good use. I have learned about so many different aspects of computing that I have not encountered in my studies. I feel this learning experience will be very helpful together with the rest of my education and my future career.

Synthesis and Characterization of Polymers

Task Order No. 57a
Student Support Program

Aric Rothman
Wright State University

23 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Ruth Pachter for her patient guidance while I became familiar with the equipment and techniques used in pursuit of my Task. I also would like to thank Mr. Marc Martin for further assistant with the computer equipment and words of encouragement.

TASK ASSIGNMENT

The object of this Task Assignment was to assist a senior chemist in the characterization of siloxane polymer materials incorporating spiropyran molecules for use as matrices for optically active materials. Results of this research could be employed in the creation of practical non-linear optical materials for use in applications such as optical computing.

Characterization of polymers was accomplished using a variety of software packages running on several different work stations and mainframes. Programs such as Chem-X, Polygraph and MOPAC were used. Computers used included DEC VAX systems, a Sun Sparc work station, and a Silicon Graphics work station. The typical procedure for characterization of a material was interactive assembly of a computer model at a graphic terminal, execution of specialized software to make adjustments to the model to make it a closer representation of the material in nature, and printing of the final data for evaluation by the supervisor. In addition to using several different commercial computer programs, in-house software was designed to assist in analysis of data produced and other programs were written to enable interfacing between existing programs. Source code for the ZINDO characterization program was available to Dr. Pachter but was not running due to several bugs in the code. ZINDO was debugged recently and data produced by ZINDO is being evaluated o see if its output is correct and useful. Various other programs have been written to assist other researchers in their work.

A working knowledge of the programs used for material characterization was acquired. Knowledge was also gained about the UNIX operating system and the writing of programs for use in a research environment. Knowledge of the C and FORTRAN programming languages and the UNIX script language have improved. Introduction to computer analysis techniques using linear algebra and the MATLAB computer program was accomplished.

Synthesis and Characterization of Polymers

Task Order No. 58
Student Support Program

Scott Brittain
Wright State University

30 June 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to mention Dr. Tom Cooper in his guidance in the peptide chemistry lab, along with Dave Flora, now at Eli Lilly Crop., for his "hands-on" guidance in the science of peptide chemistry.

TASK ASSIGNMENT

This Task was undertaken to enhance the production of laser hardened materials for the electromagnetic materials survivability branch and included the synthesis of polypeptides used for laser hardening. The production of synthetic peptides of varying sequences is necessary so that the subsequent attachment of energy absorbing molecules, or chromophores, is as desired and successfully attached. This Task involved becoming acquainted with solid phase peptide synthesis and all that it entails, keeping in mind the desired finished product to be used in laser hardening. the process of peptide synthesis involves working with automated/computer driven equipment and analytical machinery that must be learned to produce pure peptides. The Air Force is interested in utilizing this typically biomedical application for a materials application in laser hardening.

The process of solid phase peptide synthesis involves many steps that eventually lead to a pure peptide. Peptides are synthesized utilizing an insoluble support, or resin, that the first amino acid is attached to, and the subsequent peptide chain is attached to this resin from the C terminus to the N terminus. The chemical dynamics are set up so that when the chain is completed, the entire peptide can be detached from he insoluble support and retrieved. This process is analogous to placing beads on a string; if the beads are not attached in the right sequence or one or more of the beads is omitted, the entire structure may be useless. This leads to the process of purification involved in the research. Many techniques were undertaken to ensure the purity of the peptides synthesized. Low pressure chomatography and gel filtration chromatography. These processes were used for initial removal of reagents and "deletion" peptides (peptides with one or more of the amino acids in the sequence deleted). Next, a high pressure liquid chromatography device was used to identify the correct peptide and separate it. To confirm the correct sequence of a peptide, other devices were used to analyze the sequence. Capillary electrophoresis was used to separate the peptide either into it's constituent amino acids, or in tact as a peptide. This process separates by a mass to charge ratio. Other biochemical processes were utilized in the confirmation of peptide sequences, which are important in that the structure of the peptide chain is directly related to the sequence of amino acids making up the chain.

The synthesis of a potentially alpha-helical peptide was undertaken and successfully synthesized on the automated synthesizer, and purified under low pressure chromatography. Although some success has been found, many problems were encountered and consequently present much work for the future. The process of Fmoc chemistry in solid phase peptide synthesis was utilized, competent and useful. The capillary separation of constituents enables one induce the purity of the sample, of which the peptides created were evaluated. YAEAAAEAAAAEAAAA is the one letter designation for the peptide undertaken (Y=tyrosine, A=alanine, E=glutamic acid) and has been a potentially alpha helical and useful peptide for future laser hardening applications.

Intermetallic Eutectics

Task Order No. 59
Student Support Program

Brett Painter
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23 June 1992

Guiding Engineer
Dr. Dan Miracle
WL/MLLM

ACKNOWLEDGMENTS

I'd like to thank the following for their help during my term at Wright-Patterson Air Force Base: Dan Miracle for his guidance on the projects I participated in; Scott Wampler for making every day a new experience; Karen Teal for her help with the composites and for never allowing work to become dull; and Tony Houston for his unending knowledge of composites.

TASK ASSIGNMENT

Single-fiber composites of Ti-6-4 matrix/SCS-6 fiber (silicon-carbide) are currently used for evaluating fiber-matrix interfaces. A thorough understanding of fiber-matrix interfaces will promote development of advanced fiber-reinforced materials, a critical field for the aerospace industry.

The existing process for making these Ti-6-4 composites requires two steps: an initial Vacuum Hot Press (VHP) followed by Hot Isostatic Press (HIP). Because of equipment limitations in the Materials Lab, the latter step (HIP) requires considerable time.

It is therefore desired to develop a single-step process based on the VHP to create consistent, reliable single-fiber specimens.

A Ti-6-4/SCS-6 fiber composite is made by laying down a fiber or fibers onto a panel of Ti-6-4 (in this case dimensions of 1 inch x 3 inches x 50 mil thickness), then laying another panel on top. This "sandwich" is then placed in the VHP to be consolidated (pressed to form one single, solid specimen). The VHP process has three primary factors to control consolidation: temperature, time of heating and load during heating. The VHP parameters for the 2-step process are 1700° F, 2400 lbs., 30 minutes.

These parameters must be balanced against each other in the 1-step process to obtain reliable specimens. Two conditions are required for achieving reliable specimens: complete consolidation of the matrix and minimal "reaction zone." Silicon-carbide fiber has a thin protective coating that reacts with the Ti-6-4 (the "reaction zone") during VHP. Higher temperatures and longer heating times result in more of the coating being destroyed. It is critical for the coating to remain intact for the sample to be usable. Therefore, it is necessary to keep the VHP temperature as low as possible and the heating time as short as possible.

Opposing this low time/temperature objective is the requirement for complete consolidation. This means that the Ti-6-4 must "flow" sufficiently enough around the fiber to form a pore-free interface between the fiber and the matrix. Time, temperature and pressure must all be increased to allow sufficient material flow. The VHP conditions must therefore be balanced between the two goals. Care also must be taken not to crush the fiber by increasing the load too heavily.

The tested samples were created in the usual "sandwich" form, with one panel having a groove pushed into it by a chisel. The groove was used to secure the fiber in place without the need for MEK (a glue that potentially interferes with the consolidation of the sample). The groove also took much of the load off the fiber by recessing the fiber below the panel surface. This allowed higher VHP pressures to be used.

Finished samples were cut, mounted and polished using normal metallographic procedures. All samples were cut perpendicular to the fiber length, to reveal a cross-section of the fiber in the matrix. The samples were then examined for consolidation and reaction of the protective coating. Optical and electron microscopes were used to photograph the sections.

Initial testing results indicate that a reliable 1-step process is feasible. The likely VHP conditions for this precess will be:

Temp: 1700° F - 1750° F
Load: 4000 + lbs.
Time: 60 + minutes

Results from tests done at 1700° F/4000 lbs./30 minutes looked especially promising. Consolidation was nearly complete in all sections examined, and the reaction zone was minimal. Unfortunately, several samples were stained, indicating that porosity was present. Porosity is unacceptable and indicates consolidation was not compete. It is expected that longer time (1 to 2 hours VHP) will be sufficient to form force complete consolidation, and therefore obtain usable specimens.

The most striking thing I realized from working is the large number of people and the length of time required for any project to be completed. No project is being executed by any one person; many people need to be coordinated to get the job done. This requires one to be able to cooperate with others and to be able to express ideas readily. In relation to actual metallurgy, I learned the basics from my work (although limited) on the intermetallics. I learned grinding and polishing of samples, which all metallurgists must learn, and the useful tests to be done on any material undergoing development. This kind of on-the-job experience will be a tremendous help when classes begin.

Fatigue of Ceramic Composites

Task Order No. 60
Student Support Program

Ty Olmstead
Wright State University

15 September 1992

Guiding Engineer
Capt. John Pernot
WL/MLLN

ACKNOWLEDGMENTS

I would like to give special thanks to Capt. John Pernot and Larry Zawada and to all the engineers who assisted me in my Task assignment.

TASK ASSIGNMENT

This Task was to help Larry Zawada and Capt. Pernot in their research of Ceramic matrix Composites (CMCs). These Composites are being considered for aerospace applications for the Air Force, such as the Advanced Tactical Fighter (AFT) and National Aerospace Plane (NASP). The CMCs are far superior to their metal counterparts in their capabilities to withstand elevated temperature and with respect to their lower density. Before the CMCs can be built of material for an engine, the reliability of these materials must first be tested to determine if they are candidates to replace their metal counterpart. One of the many groups testing these CMCs is the Materials Behavior Branch in the Materials Laboratory at WPAFB. This group tests the CMCs in an in-house program under the lead of Larry Zawada. The team's main interest is in the mechanical behavior, the thermal behavior, and in estimating the life expectancy of the CMCs being tested.

The equipment used at the in-house group includes computer controlled Servo-Hydraulic fatigue machines and a Schenk thermal-loading machine. This equipment allows the engineers to test the CMCs with great accuracy.

To evaluate a material the engineers first run a tension test at room temperature to determine the maximum strength of the ceramic or glass composite with which they are working. After the test is completed, the tensile data is analyzed to find the most interesting peculiarities (ie. the stress where extensive micro-cracking leads to failure of the specimen). Then the specimen is mechanically fatigued at and near this stress level. If information at room temperature is already known, the ceramic matrix composite also may be tested at elevated temperatures. After testing the data collected by computer sampling, it is transferred to the VAX computer system and converted into the proper units. The data is then sent to Macintosh computers and graphed. Engineers examine these graphs to see if any prevalent patterns of trends are present.

Air Force research in CMCs is aimed at doubling performance of present day jet turbine engines.

High Temperature Superconductivity

Task Order No. 61
Student Support Program

Benjamin P. Smith
Wright State University

30 June 1992

Guiding Engineer
Mr. Tim Peterson
WL/MLPO

ACKNOWLEDGMENTS

I would like to acknowledge Mr. Jerry Landis and Mr. Dave Walsh for their assistance with the chemicals and clean room procedures.

TASK ASSIGNMENT

High temperature superconductivity as a relatively new field has opened many avenues for experimentation. Many experiments at WL deal with the characterization of the high temperature superconductor samples, particularly thin films. The first report submitted by the author dealt with a four-point measurement system designed to determine the critical temperature Tc, and the critical current, lc. When these levels are reached the sample loses its superconducting capability and is said to "go normal."

This subsequent report deals with the concept and process of photlithography, which concerns film patterning. When a film is said to be pattered, it has undergone a process in which some of its material has been stripped away to test a particular part of the sample. Three methods of patterning have been encountered prior to working with photlithography. These previous methods are the following: manually, using a diamond scribe to cut grooves in the film; laser etching, using a laser to burn away part of the film; e-beam lithography, using an electron beam to etch away at the film. The scribe method works very well in a case where precision is not a primary issue. If, for example, a narrow strip of 20µm was to be patterned on a film, a scribe would not be the tool of choice for two possible reasons. First, the human hand is not that skilled, and second, the tip of the scribe is of the order 200µm, it would be impractical to use it to cut a smaller width. The method of laser etching is fine were precision is an issue. The laser can be set to follow a precise X-Y coordinate system; however, if the need would ever arise to make a precise curved strip on the film, the laser would not be able to oblige. Another drawback of the laser etch is the heating of the edges. As a laser burns through the film, the film on the periphery of the laser gets heated. If the strip to be patterned is too thin, the heated film on one side can come in contact with the heated film on the other side. The result is bad film being measured. E-beam lithography presents the problem of being expensive, inconvenient, time consuming and inadequate. it is inconvenient because it would not be an in-house project, would be time consuming because it is designed for use on much smaller systems, more on the order of angstroms instead of microns. The time needed to pattern a film would require more time and resources than the outside lab would be used to, hence the increased expense. The alternative currently available is photolithography.

Before a project is begun, it is preferred to know what form the outcome should have. Various film structures are needed for the types of measurements being performed at the Materials Lab. For the resistivity type measurement discussed in the first report, a film patterned into strips is preferred, to measure high critical current densities with the least amount of applied current. For ac susceptibility measurements other film structures are preferred.

The process of photolithography begins on drafting table and ends in a chemical etch. Individual patterns take shape on a dual layered sheet of plastic called ruby-lith, getting its name from red coating. Patterns are cut into the ruby-lith at a much larger scale than actually used. The large patterns are then reduced in size, typically by a factor of fifty, by a photographic process. The final pattern emerges on a five inch photographic plate. This plate is then taken to the lab's clean room where copies of it are made on iron oxide plates, and the original is safe from harm.

The film is placed on a spinner, used to spin the sample at varying rates of rotation. While on the spinner, a drop of photoresist coats the surface of the film in a fairly uniform manner. The nature of the photoresist is basically the same as that of a photographic emulsion. The iron oxide mask is placed over the film where the pattern is to be, and the entire film-mask combination is placed on a holder where it is exposed to ultraviolet radiation. The exposed photoresist is then developed off and the remaining photoresist protest the underlying film. The film is then cured in a nitrogen oven. When removed, the film undergoes the etching process in a bath of phosphoric acid. The film that remains is in the shape of the designed pattern and the remaining photoresist is rinsed away in the solvent.

Once the film is patterned, it is necessary to put some type of metal contacts on it so that it can be used in the four-point measurement. This process is called metallization. The susceptibility measurement does not require the contracts since the current that is applied to the film is magnetically induced.

Previously, metal contacts had been applied with silver conductive paint, or through a process called sputtering that result is that the film is coated with a thin layer of silver so that electric leads can be attached to the film.

To begin, the process of metallization with respect to patterning films is very similar to the patterning process. The chemically etched film is again cured after its solvent bath to remove all trace of solvent. It is then placed on the spinner where it receives a new layer of photoresist. A mask with the image of the designed contact pads is placed over the film, exposed and developed. This film is then taken from the clean room to the evaporator or sputtering machine where the actual process of metallization takes place. After the layer of metal has been grown on the film, the film is taken to the clean room where it is undergoes another solvent bath. This solvent is used to rinse away the unexposed photoresist. The result is, ideally, small pads of metal at predetermined locations on the film.

Attempts are being made by several different branches of WL on the request of the Materials Lab to perform different metallization techniques on the patterned films. These attempts include and have included evaporation of Au (gold) target, an Au-Pd (gold-palladium) alloy target, a Au-Ti (gold-titanium) alloy target, and a Au-Ti-W (gold-titanium-tungsten) alloy target. The gold, being the noble metal does not bond to the polished surface such as the substrate, it therefore needs to be combined with another metal to serve as a junction between itself and the substrate. The other metals listed have their drawbacks. For example, titanium bonds well to both the gold and the substrate but it is much more resistive than the gold. If 1500Å of gold has a resistivity of 1 to 3 mΩ-cm, adding 200Ω of titanium boosts that resistivity to approximately 5mΩ-cm. The palladium on the other hand tends to be a very highly stressed material so that when deposited over the film the stresses could possibly be enough to cause the film to peel up from the substrate around the edges. Tungsten is simply added to the titanium to strengthen the bond.

Synthesis of Fused Ring Aromatic Compounds with Donor and Acceptor Groups for Evaluation as Non-Linear Optical Materials

Task Order No. 62
Student Support Program

Paul Roderer
University of Dayton

30 June 1992

Guiding Engineer
Mr. Bruce Reinhardt
WL/MLBP

ACKNOWLEDGMENTS

I would like to extend my sincere thanks and gratitude to Gibby Dombrowski and Marilyn Unroe for guiding me with their wisdom. I would especially like to thank Bruce Reinhardt for his patience and guidance with a wealth of wisdom and knowledge that seems to be never-ending. Thank you.

TASK ASSIGNMENT

This Task was an explorative extension of last year's assignment. The major goal of this Task, like last year's, was the development of Non-Linear Optical (NLO) compounds. NLO research is on the cutting edge of physical and chemical research with ultimate goals of improving communication and computer technology though the use of compounds that refract electomagnetic radiation in a non-linear fashion. This Task focuses on the development of organic NLO compounds with emphasis on synthesis of possible NLO compounds.

Extensively conjugated organic compounds have shown a high degree of promise in NLO activity. This Task focused on development of novel, extensively conjugated compound which were sent to Dr. Paris Prasad at his research facility in Buffalo, N.Y. for NLO characterization. Task duties for this year included a substantial amount of library research, completion of over 70 chemical reactions, work-ups and purifications of the products form those reactions, characterizations for the isolated products and basic lab maintenance.

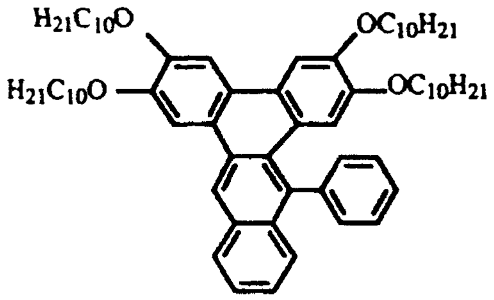
All conducted research followed a specific four step route of protocol. This research route began with a designated library assignment initiated by Mr. Reinhardt. The library assignments yielded reported reaction schemes used to develop similar compounds. Once a potential synthesis route had been determined, lab experimentation began. The third step was characterization of isolated compounds. The final step was the compound preparation for NLO characterization.

The major portion of this task was dedicated to the development of the novel class of fused ring compounds indicated in figure one. The synthesis route for compound A was primarily developed during the last Task. However, the final synthesis steps and major advancements in streamlining this synthesis route were accomplished during my Task. These advancements increased product yield cited in any literature. Interest in developing compounds B & C stemmed from the observation that powerful electron withdrawing groups attached to the conjugated ring system increased NLO activity. Synthesis of all three of these compounds followed a five step synthesis procedure. The reaction scheme for this procedure is outlined in figure two.

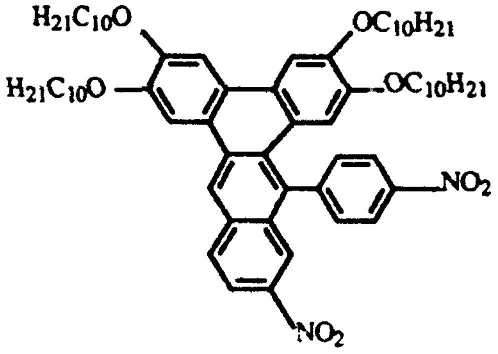
After development and isolation of these compounds, the isolated products were characterized. Characterization techniques used included IR, ^{1}H NMR, ^{13}C NMR, Mass Spectroscopy, Elemental Analysis, UV Spectroscopy, Thermal Analysis, Melting Point determination, Thin Layer Chromatography and solubility characterization. While this Task included actual use of instrumentation to accomplish the above characterizations for only part of these procedures, complied data from the non "in-house" procedures needed to be analyzed and familiarity with all the above procedures was essential to the Task.

Following the characterization process, these compounds were placed in a polymer film and set to Buffalo, N.Y. for NLO characterization. Current NLO data results for compounds indicated in figure one are reported in the results section of this paper.

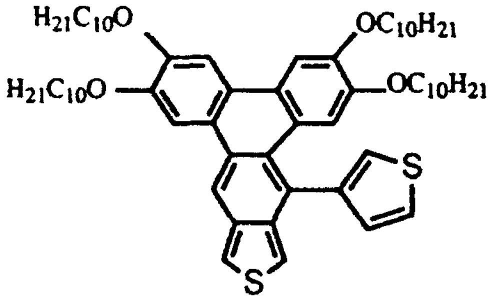
Figure One: Major task objectives. Development of novel fused-ring systems.



Compound A
$C_{68}H_{98}O_4$



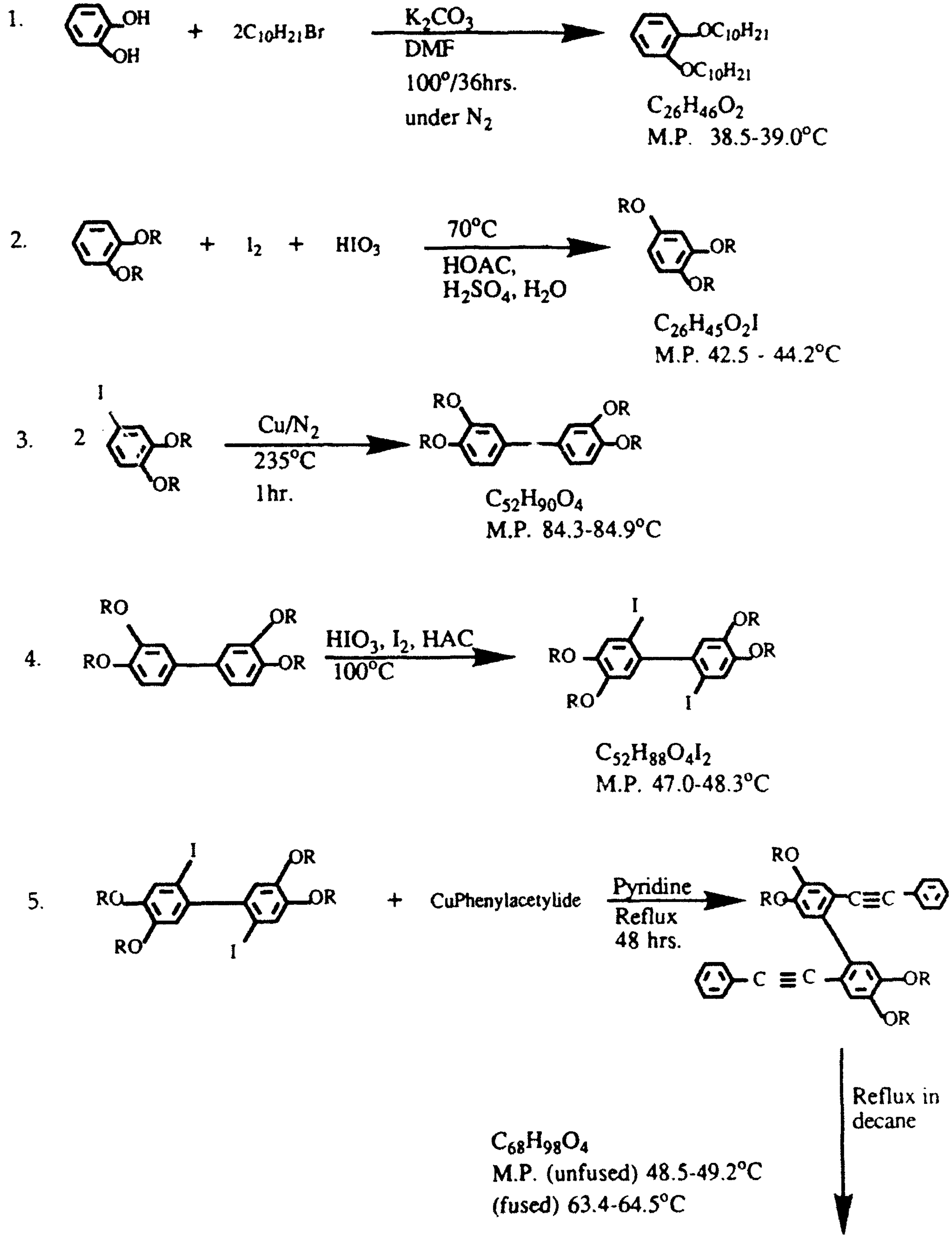
Compound B
$C_{68}H_{96}O_8N_2$



Compound C
$C_{64}H_{94}O_4S_2$

While synthesis of the above three compounds comprised the majority of this task, other important projects were also attempted. A charge transfer complex of compound A in fig. 1 and tetracyanoethylene was developed and tested for NLO activity. The results from this testing are reported in the results section of this paper. Currently, heptamer and pentamer ring structures are being developed for use in NLO instrumentation set-up and testing. These structures are displayed in figure 3. While the synthesis routes for these compounds had previously been developed, the synthesis procedure was streamlined and yield was increased by a factor of three.

Figure Two: Reaction Scheme for Synthesis of Compound A



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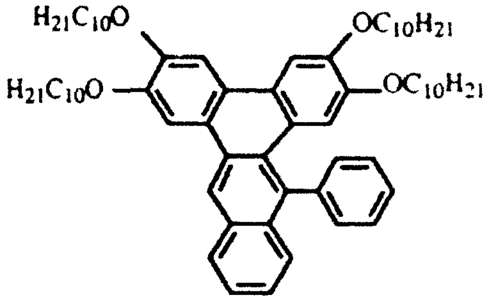
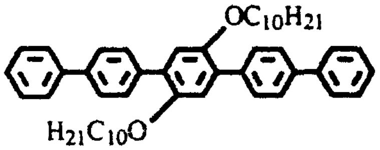
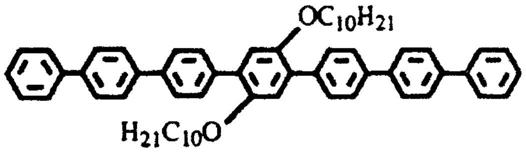


Compound A
$C_{68}H_{98}O_4$

Figure Three: Heptamer and Pentamer model compounds.



Pentamer
$C_{50}H_{62}O_2$



Heptamer
$C_{62}H_{70}O_2$

RESULTS/DISCUSSION

The developed synthesis routes for the discussed compounds contained several steps in their respective sequence. Overall reaction yields are indicated in table one.

Table One: Overall reaction yields for primary research products of task #62

Compound	Overall Yield
Compound A (figure one)	9.0%
Compound B (figure one)	2.6%
Compound C (figure one)	2.8%
Heptamer (figure three)	32.0%
Pentamer (figure three)	inconclusive at this time

While this yield data may appear low, the synthesis route covers many steps for each compound and each individual step's yield typically fell in the 50-70% range.

NLO data for four of the above listed compound was obtained. This data is listed in table two below. Since compounds B & C in figure one were not isolated in sufficiently pure samples, no NLO data on these compounds were obtained.

Table Two:

Compound	χ^3	γ	γ real	γ imaginary
Compound A	-	1.3×10^{-34}	-	-
Charge Transfer of Compound A	6.8×10^{-42}			
Heptamer			-3.3×10^{-33}	7.2×10^{-33}
Pentamer		3.5×10^{-34}		

* All measurements are in esu

None of the above values are high enough in NLO activity to serve any major practical application. However, developments in this area are ongoing. For example, compound A and its derivative's NLO γ value indicate that while extensive conjugation increases NLO activity, activity decreases as the number of fused rings increase. This activity probably decreases because the polarizability of these molecules decrease. Compound A represents a more conjugated system than the pentamer but as indicated in table one the pentamer produces a higher γ value. The pentamer is more polarizable than compound A. Furthermore, since organic NLO synthesis is currently in its infantile stages of development, many factors remain to be discovered before useful organic NLO compounds will be developed.

This task concentrated on the synthesis portion of organic NLO compound development. Several new compounds were developed and sent to Buffalo N.Y. for NLO activity evaluation. From the synthesis standpoint, this task was successful with the development of several new compounds, streamlining of old procedures, and over 70 reactions completed, purified, and the resulting products characterized during the allotted 1040 hour period. The lab experience has been personally rewarding and I hope that I can contribute in a positive and productive manner during my task.

Polymer Characterization

Task Order No. 63
Student Support Program

David Carpenter
Wright State University

30 June 1992

Guiding Engineer
Dr. Fred E. Arnold
WL/MLBP

ACKNOWLEDGMENTS

I would like to thank everyone at the Materials Laboratory Branch for their friendliness and cooperation. Also, I would especially like to thank Dr. Fred Arnold and Dr. Thuy Dang whose guidance made this possible.

TASK ASSIGNMENT

I worked at the polymer research branch at Wright-Patterson Air Force Base as a lab assistant. My duties were to maintain the lab working area, observe reactions and procedures, clean glassware, characterize monomers and polymers and perform various other tasks.

One of the major parts of this Task was to keep all the glassware ready for use because research tends to dirty a lot of dishes. Cleaning glassware requires a great deal of time and patience. Extremely clean glassware is of vital importance when running reactions, because the slightest amount of unwanted material can cause the reaction to run improperly. That is why there are many steps and precautions used to do the job properly. First, the dirty glassware is washed with water or acetone to rid the majority of unwanted compounds. Then the glass is placed in an ultrasonic bath for a couple of hours. This is a tub-like creation filled with distilled water and an ammonium solution to disinfect. It heats and vibrates the liquid to loosen any particles on the glass. After this point the glass is nearly free from any residue, but to be sure, it is washed repeatedly with tap water and dishwashing soap, if necessary. Finally, acetone (finger nail polish remover) is sprayed all over the glass to give it the finishing wash and prevent water spots. They are then hung on a rack to dry. The result is sparkling clear glassware.

A major objective was to learn material pertaining to my field of study and gain understanding of the professional world. Also, I wanted to apply some of my knowledge obtained from school. At this job I did use alot of chemistry, especially material on organic chemistry. Monomers are the building blocks of polymers, and as part of my experience, I observed the synthesizing of the two. It was then my duty characterize the reaction products for thermal stability (melting points), solvent resistance (viscosities), and chemical composition. At our lab, there were two ways to detect chemical composition. One was nuclear magnetic resonance (NMR) and the other was by infrared. Both are spectrometric methods. They work on the basis of frequency shifts. Certain structures appear at specific frequencies and are graphed as a peak. From books and diagrams you can determine the compound's makeup. It took many attempts to learn hoe to use the IR machine, but it was soon mastered. The hard part was mixing the sample at the right concentration with potassium bromide. This was necessary to get a better printout. As for the NMR instrument, I had not become accustomed to using it.

Melting points are taken by placing some sample in a tiny tube and slowly heating it. The temperature at which the sample begins to melt and the temperature at which the sample is totally melted is rad. The smaller the range of these two temperatures, the more pure the sample is. Viscosities are the most time consuming process. With this process, the adhesiveness and thickness of a polymer can be determined. First the solvent time is measured. The solvent time is the time it takes for the liquid to drop from one line on the viscometer to the other. At least five trails are taken for every step. Next, the polymer in the solvent time is measured. The reason for this is the polymers are sometimes so tough they must be dissolved into the solution before they will flow. An example of such a solvent would be methane sulfonic acid (MSA). After all this, the trail times are compared, calculations are done to determine the instrinsic viscosity (IV). This determines whether or not you have "good stuff." The higher the IV is the better the polymer.

It wasn't until about six weeks on the job I achieved another of my goals, to synthesize a polymer on my own. With 2.5 grams of 2, 5 - diamino - 1, 4 - benzene dithiol and 1.5 grams of terephthalic acid at 10% concentration, with 14.5 grams of poly-phosphoric acid and 8.5 grams of phosphorous oxide, stirred under nitrogen and heated for three days, I finally produced 2.5 grams of a poly-benthiazole polymer. This process took approximately 5 days. It wasn't until the 5th day that the moment of truth happened. An instrinsic viscosity was taken on my polymer. It came out to have an IV of 11. That wasn't high enough for recognition, but that's research.

This experience gave me a taste of the type of environment I may soon experience upon graduation. Even though I did not make a super polymer, I thoroughly enjoyed the experience and friendly people I worked with. Thuy D. Dang said that his lab has never looked better of more organized. That was of great satisfaction to me.

Polymer

Task Order No. 64
Student Support Program

Melissa Gonet
University of Dayton

30 June 1992

Guiding Engineer
Dr. Loon Seng Tan
WL/MLBP

ACKNOWLEDGMENTS

Thanks to Dr. L. S. Tan and Ms. S. Simko for their help and guidance in my Task Assignment.

TASK ASSIGNMENT

While working this past year in the Polymer branch at Wright-Patterson Air Force base I have been responsible for keeping the lab in working condition. This requires doing various analytical work, assisting in ordering and keeping an inventory for chemicals, working on the computer to aid in chemical analysis, doing dishes and getting supplies for the laboratory usage.

The analytical work done includes taking samples that have been developed in the lab and analyzing them to determine if they meet certain specifications. The samples are usually polymers and sometimes monomers. The analysis methods include (but are not limited to) use of a Nuclear Magnetic resonance Spectrum (NMR), an Infrared radiation machine (IR), viscosities and melting points. The NMR uses magnetic fields to determine the placement of hydrogen atoms in a structure. Doing this is helpful because the spectrum for a particular sample distinguishes functional groups by their placement on the spectrum and, therefore, verifies the sample's structure as determined in its synthesis. The IR is (i.e., alcohols) but it utilizes the ability of the bonds to stretch to do this. Viscosities and melting points are other information concerning a new sample's potential for future development. It is this potential that the lab is trying to achieve.

Computer work is also done to help in the analysis of samples. Graphing viscosities, drawing out chemical structures, and putting on paper the results of entire synthesis projects, work together to create a compact image of the "whole picture." This is beneficial because it takes one beyond specific jobs and allows a view into the whole synthesis process.

Keeping track of most paperwork, including logging in chemicals for analysis by other workers and keeping an updated list of all Material Safety Data Sheets for chemicals, is another responsibility for this position. This job, as well as inventory and safety, aids the efficiency of the lab. The responsibilities are various and necessitate awareness in running an efficient laboratory. All jobs insure less wasted time on minute details and more beneficial time for running and improving reactions.

Another major responsibility has been government ordering that occurs from November to early may. This involves much patience. Prospective orders are received from all the members in L. S. Tan's group. They are monitored, funds in the budget for future orders are calculated, the orders are then allocated to the next person in the chain of processing. This job is demanding but also one of my favorite assignments.

These responsibilities have helped me learn about the intricacies or working in an analytical occupation. Though not a large amount of actual synthesis is done in this position, the analytical procedures aiding the synthesis are abundant. I have learned about the trails of developing not only a pure sample but also one that has potential as a future monomer or polymer. I realize how important the "little details" are for completion and hope during the next year I will feel just as needed and personally rewarded.

Electronics and Computer Support in Electromagnetic Material Research

Task Order No. 65
Student Support Program

Larry Brunson
Wright State University

30 June 1992

Guiding Engineer
Mr. Ronald Perrin
WL/MLPO

ACKNOWLEDGMENTS

I would like to give a hearty thanks to the two electrical engineers, Mr. Ron Perrin and Mr. Tom Kensky for their friendliness, patience and willingness to share their expertise with me.

TASK ASSIGNMENT

The Task objective was to provide systems support for research in electronic, electromagnetic and optical materials. Specifically this was to be accomplished through designing and maintaining electronics, computer hardware and software. Following is a summary of how these objectives were met.

I built a number of electronic circuit boxes for various applications. For example, stir circuits for crystal growth tanks in an optical lab were needed. These circuits involved digital and analog components with a couple of water flow solenoids and associated plumbing. I was responsible for the machining, layout, assembly and setup of the circuits.

Mr. Perrin gave me a circuit project building a 8 amp multi-output dual tracking power supply for use in the optical lab. Given a large circuit box, associated parts and raw materials, I was responsible for the layout, machining and assembly of the power supply.

I also received a project to design a temperature controlled cooling fan circuit to cool a laser safety shutter for the optical lab. This project involved the use of Douglas CAD/CAM to lay out a printed circuit board and to get a template to use for etching the board of a prototype circuit. This project was very interesting because of the experience of making a printed circuit board.

The computer hardware support I gave during this Task involved servicing some of the numerous Z-100 computers used in the labs. I was responsible for setting up the hard disks after they were repaired by prepping, parting and formatted them. I also modified the RAM and video memory by adding or replacing computer chips. On one or two occasions I installed faster timing clocks in the computers.

By far most of my Task was in computer software support. This was divided into the use of existing programs and the writing of new software. I spent a lot time using KaleidaGraph to make graphs for Dr. Ohmer whose research in the field of optical semi-conductors. He not only needed various plots, but he also needed the curves manipulated by expanding or moving them to a particular spot on a set of axes. This proved to be a very enjoyable application of my mathematics training.

I used MacDraw to modify a flowchart for the branch chief. Tom also gave me an assignment to neaten three freehand drawings done by one of the scientists. This proved to be a valuable experience since it used most of the features of MacDraw, including the freehand drawing tool.

Mr. Perrin gave me a project to revamp and revive the maintenance department's data base program. This involved learning dBASE II and dbASE III and modifying an existing program to suit the needs and desires of those using it. This turned out to be an ongoing project since the maintenance department upgraded to a more powerful computer and their needs and desires evolved during my Task.

Probably the most significant program I wrote arose from a need expressed by Dr. Ohmer. Much of my graphing I did for him came from the data sets obtained by digitizing curves found in various technical journals. He expressed a desire to be able to subtract one curve from another. This could be accomplished in one of two ways. If the equations for the curve segments could be found or if the digitized data could have the same "x" values, they could be readily subtracted one from the other. The first alternative seemed like a tedious and complicated exercise in curve fitting. After investigating the second alternative, which arose due to the random nature of digitizing, I wrote and polished a fairly universal data interpolation program.

The preceding is only a very brief summary of my Task. I learned skills in electronics, computer hardware and software. In electronics, I developed technician skills learning how to solder and de-solder and how to program PROM chips. In computer hardware, I learned how to work with delicate dual in-line package chips. I developed a feel for computer mechanics. In computer software, I enhanced my skills in algorithm development and program modularization.

Mechanical Fatigue of Titanium Aluminum Composites

Task Order No. 67
Student Support Program

Alisha Hix
Sinclair Community College

21 July 1992

Guiding Engineer
Dr. Jay Jira
WL/MLLN

ACKNOWLEDGMENTS

I would like to give special thanks to everyone working in MLLN. They've all been helpful whenever they could, and made me feel like an important part of the lab. I also would like to thank Mike and Eric in the Metalography Lab for putting up with my endless questions. I especially want to thank Dr. Jay Jira. The knowledge I gained about engineering from him can't be acquired in school.

TASK ASSIGNMENT

The Task Assignment required me to assist Dr. Jay Jira and his colleagues studying Metal Matrix composites (MMCs) in the data analysis aspects of their research. My duties included both quantitative and qualitative analyses. A large part of my time was spent running raw test data through data analysis programs on a Micro VAX computer system. It was also my responsibility to analyze the data from the photos taken during fatigue crack growth tests. In addition to these, Dr. Jira requested information for small scale studies and I assisted in collecting the necessary materials required.

The analysis of the raw test data was a multi-step process that started with analysis of each photo taken during the test. (Photos were taken of each test specimen at regular intervals as a record of the crack growth rate). A technician would take a roll of film for each test and measure the total visible crack length in each photo and record it with a program on a Textronix 4052 computer. Then that data file containing these crack lengths would be transferred to the Micro VAX. The first step was to merge the optical crack length file with the run time data file. (This file contained crack lengths measured by a different method). The resulting file contained the most meaningful data from both files and allowed for easy comparison of the two crack length measurement methods. After merging the first two files, another program was run on the merged data file to generate a fourth file containing new calculated values. This file allowed for easy comparison of crack growth rates from several specimens. (Sometimes the above programs had to be rerun several times on the same specimen because of the changes in the analysis programs).

After reducing and plotting the numerical test data, Dr. Jira requested 8x10's of the picture taken just before the specimen broke, or the last picture taken during the test if the specimen didn't break. Sometimes we also needed the first picture in the test for reference. I was responsible for having the prints made, making sure we got a print of good quality to see the necessary features, and organizing and labeling the prints so we could find each one easily, if necessary.

During the last year Dr. Jira also had me work on several small projects. The first was a crack initiation angle study. For this study we took specimens that had been tension tested and measured the angles on each where cracks had initiated around a bolthole. After generating a sizable database we evaluated the data using Statview II in the Macintosh IIfx. Another project that I conducted for both SCS-6/Ti-24Al-11Nb and SCS-6/ß21S materials was to measure the crack length on each specimen from the photo taken near the end of the test. For ß21S this was measured from photos and from the fracture surface of the specimen (if it failed and was tested at high temperature). These measurements were compared to those measured during the test and those measured during the digitizing of the test photos. I organized the different measurements on a spreadsheet for Dr. Jira to analyze. The shortest project I worked on measured how much of a given unit of ß21S material was the metal alloy and how much were the fibers that give the material its strength. The latest project assigned to me, which is not yet completed, had two specimens that were already tested and sectioned them so that different cross sections of the crack were visible. The purpose of this was to measure the length of the fiber-matrix separation resulting from testing.

The company to whom we send our test results has requested certain data from each test. My responsibility is to see that the photos they requested were ready to send on time.

Finally, I see that log sheets, test film, and our computer directories are organized. I also helped Dr. Jira with periodic presentations at out-of-state symposiums or informal local meetings. I have been asked to generate diagrams and special plots for various papers and meetings.

I have gained a great deal of practical experience during the past year. The data analysis I did gave me more experience on a VAX and introduced me to the world of Macintosh computers. My work in the Metalography Lab exposed me to metalography techniques and the capabilities of photography. I also learned a great deal about photography from the people at Tech Photo, who have done much of the printing work I needed. Most importantly, Dr. Jira has taught me to be objective with scientific data, which will be valuable to me wherever I go after graduation. Overall, I have enjoyed the past year and look forward to the next year.

Structure Failure Analysis Support

Task Order No. 68
Student Support Program

Kuo-Chu Colin Huang
University of Cincinnati

15 July 1992

Guiding Engineer
Mr. Ron Williams
WL/MLSA

ACKNOWLEDGMENTS

I would like to take this opportunity to thank the following people for helping me throughout this quarter:

Technicians	Engineers
Andy Logue	Ron Williams
Tom Dusz	Larry Perkins
John Ziegenhagen	Michael Oliver
Dan Laufersweiler	Neal Ontko
Gary Stevenson	

TASK ASSIGNMENT

The investigation of four stainless steel barrels was one of the failure analyses that I helped with. Four stainless steel barrels had been designed to store and transport Hydrazine for the Department of Transportation. Due to the poor manufacturing, these 55-gallon drums did not pass the pressure test that had been performed on them. Therefore, WL/MLSA was asked to investigate the problem.

First, all drums were visually examined, followed by chemical paint removal and more extensive non-destructive examination using dye penetrate and x-ray techniques. Each drum had a large and small plug. The large plugs were difficult to remove from the flange on two barrels. Meanwhile, the small plug from one lot would not tighten due to a stripped thread resulting from weld distortion and undersize threads.

The top of two selected barrels was removed by an abrasive cut-off wheel below the circumferential welds. On the inner surface of the flange welds, severe corrosion was observed. The dye penetrate inspection detected cracking within the welds. The large plugs were also removed for x-ray analysis.

A small section of the material was removed for chemical analysis and evaluation of the weld. The result from chemistry evaluation showed that the drum material was 304 stainless steel, however, did not meet the minimum specification requirements. Likewise, the plugs did not meet the specification requirements. In each case, the chromium and nickel were found lower than the minimum requirement. This increases the difficulties in welding and reduces corrosion resistance. Another cause of reduction in corrosion resistance was during welding the base metal (304 stainless steel) was sensitized. The sensitization occurs when the time at welding temperature is long enough to allow chromium carbides to precipitate. This was clearly noticeable at the welds under the optical microscope.

Few conclusions were drawn after all the examinations. The flange and plugs were out-of-tolerance and the base material chemistry was found below the specification minimum. The practice of welding was poor and the excessive heat sensitized the welds and the base material that reduced mechanical property and corrosion resistance. Finally, the drums that were manufactured using these processes and procedures should not be used to store hydrazine.

Structural and Electronic Failure Analysis Support

Task Order No. 68a
Student Support Program

Joseph Hunter
Wright State University

15 July 1992

Guiding Engineer
Mr. Ron Williams
WL/MLSA

ACKNOWLEDGMENTS

The following projects were worked on under the direction of Larry Perkins, welding engineer, Brad Pinnell, mechanical engineer and Tom Dusz, professional technician.

TASK ASSIGNMENT

The failure analysis division at Wright Patterson AFB was one of the first and largest failure analysis departments in the country. All branches of the armed forces as well as outside companies contact the lab when a metallographic or composite failure occurs. It is the concern of this department to determine the cause of failure of a part or system and to recommend a preventative action to alleviate the problem(s).

Provided below are a few of the projects in which I was directly involved; described are procedures and recommendations involved in the project.

My first project was to determine the cause of uneven wear of seal. The seal was being worn by a high pressure, hydraulic power steering system consisting of copper brazed SAE 1018 steel plates. The uneven wear resulted in leaks during operation.

The purpose was to evaluate the SAE 1018 steel plates for internal surface inclusions believed t cause the wear and identify the type of inclusions.

As required for each destructive analysis, photographs were taken of the assembly for documentation purposes. A wet cut-off saw was used to cross section the brazed assembly so the part could be prepared for microscopic analysis using standard metallographic procedures. The following description of the procedures is incorporated each time a part is to be analyzed or photomicrographs (photographs of a part while under magnification) are needed.

After a part is cut to a specific size, it is put into an opoxy mold (bakelite of lucite usually) using high pressure and temperature; this allows for easy handling for future steps.

To obtain a smooth, scratch-free surface, rough grinding, using first 60-grit and then 120-grit silicon carbide paper, is employed. Distilled water is used during the process to prevent material heat-up. Fine grinding requires the sequential grits of 240, 320, 400 and 600 paper.

Polishing the 600-grit scratches requires the use of six and then one micron diamond compound solution. The specimen is placed on, and rotated counter to the direction of the polishing wheel containing the solution and a liquid extender. Fine polishing uses distilled water and a alumina slurry (0.3 and 0.05 micron size particles) on the rotating wheels. The specimen is then cleaned ultrasonically for about two minutes.

Once the part was polished, a chemical etchant was applied to the surface to bring out the microstructure of the steel. An uneven etch around the edges of the cross-section was found that led to the belief that the steel had been carburized[1].

Surface carburization was verified through microhardness readings that revealed a dramatic increase in hardness toward the surface. The hardness varied form 63 near the surface to 22 in the bulk (Rockwell, scale C). The thickness of this carburized layer was approximately 1 to 1.5 mm.

Further analysis, using a scanning electron microscpoe (SEM), was performed on inclusions found near the surface in the carburization layer. both manganese-sulfide and carbide inclusions were identified in this area. The sample was evaluated by chemical analysis using X-Ray Fluorescence (XRF) technique to obtain a percent of the elements by weight.

It was concluded that the uneven wear of the seal was due to the varied hardness of the inclusions in the carburized layer compared to the base metal. A steel manufactured to reduce the size and amount of inclusions, such as SAE 8620 or SAE 4330 was recommended as a more appropriate metal for the application.

Brad Pinnell improved upon a method to estimate the percentage of voids in cured composite material (plys of woven metallic fibers cured with a resin), different to that of acid digestion and grid techniques.

This process required the metallographic polishing procedures previously mentioned, with the alteration of using cold instead of hot mounting. Once the specimens were polished scratch-free, a black permanent ink was applied and allowed to dry on the surface of the specimen to be analyzed. The excess ink was wiped off while the rest stayed in the voids, which ultimately darkened them.

A sample was then placed under a microscope at 250x, the image of which was transferred to a large television screen. This image was transferred to an image analysis system that was set up to allow the operator to adjust the light

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[1] Carburization increases the surface hardness of steel by shooting hot methane gas on its surface, increasing the carbon content in the form of carbides.

intensity given off by the analysis system. The light intensity was changed until the amount of intensity given off by the system (seen in yellow in the system screen) covered the darkest areas of the image (voids). The amount of light at this point, determined by the operator, was calculated by the system as a percentage of the total image on the screen, was relatively a small area of the specimen being analyzed. This recorded percentage relates to the percentage of voids for he particular area. The specimen was then moved to another specified area, the path of which covered an "X" pattern on the square sample. This pattern maximized any variations in void content through the thickness of the specimen. Each time a different area of the surface was analyzed, the void percentage of the part; the numbers corresponded very closely to the grid technique.

This project seems to offer a more accurate and less time consuming technique of determining the amount of unwanted voids in composites. This is important for researchers to find ways of improving the fabrication of the composite materials, which are constantly being explored. The amount of voids present is key in deciding whether to alter the fabrication technique.

Described were two projects that typically represent my work. Of course, there have been many others that required similar investigative techniques such as a cable failure from Coast Guard helicopter; a jammed 20-mm gun form an A7; pitted aluminium jet fuel tubing. Currently, I am examining corrosive defects of heat exchanges form a C130 transport; plated-through hole failures on electronic circuit boards; and weld failures on inconel tubing.

Having metallography experience was very helpful in becoming familiar with the work required for failure analysis. Photomicrographic and documentation experience was also helpful.

I also learned to use other equipment such as an image analysis system and a microhardness tester. I became indirectly familiar with the scanning electron microscope (SEM) as it provided detailed images the optical microscope could not.

Synthesis and Characterization of Polymers

Task Order No. 69
Student Support Program

Timothy Grinstead
Wright State University

11 July 1992

Guiding Engineer
Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to recognize the following persons who helped guide me though my Task: Dr. Zbigniew Tokarski, Dr. Tom Cooper, Dr. L.V. Natarajan, Sungmee Yoon Kim, Paul Pike and Morley Stone.

TASK ASSIGNMENT

This Task Assignment involved the synthesis and characterization of numerous different polymers. These polymers were synthesized to examine how the addition of Nonlinear Optical groups affected the structure and properties of these polymers. Each additional polymer synthesized led to a better understanding of how an ideal Nonlinear Optical polymer can be produced.

The synthesis of the polymers was attempted through several different methods to determine which method produced the best results. Transesterification reactions, dicyclohexylcarboimide (DCCI) coupling reactions, and triphosgene reactions were all employed to determine which approach allowed for maximum substitution of nonlinear optical pendant groups. Once these polymers had been synthesized and purified, the final product was analyzed through instrumentation and the use of various software applications.

The instrumentation used for analyzing the polymers synthesized involved employing state-of-the-art equipment. Measuring the structure of the polymer involved use of a circular dichroism spectropolarimeter, a lambda-9 UV-VIS spectrometer, a fourier transfer infra-red spectrometer and nuclear magnetic resonance spectroscopy.

The data collected was analyzed by using numerous different software applications and interpretation of the spectra produced. Entering the data from the instruments into spreadsheets and graphical applications allowed for data manipulation to examine the results.

After analysis of the data complied to date, it was found that the bulkier NLO groups (dyes) were more difficult to substitute on the polymer backbone. Use of these dyes also made purification difficult, but allowed the use UV-VIS spectroscopy for determination of percent modification. It was also learned that elemental analysis results were some what misleading when attempting to determine percent modification, so NMR will be used in the future. The use of triphosgene will be instituted in further research to allow for extensive modification of the dye to take place.

Processing and Properties of Molecular Composites

Task Order No. 70
Student Support Program

Morley Stone
Wright State University

30 June 1992

Guiding Engineer
Dr. Robert Spry
WL/MLBP

ACKNOWLEDGMENTS

I would like to thank Dr. Chyi-Shan Wang of the University of Dayton Research Institute (U.D.R.I.) for allowing me to work on his project and for the trust and confidence he displayed in my abilities. I also would like to thank Dr. Robert Spry (WL/MLBP) for his help and support as group leader of the processing and properties group within the Polymer Branch, Wright-Patterson A.F.B. Dayton, Ohio.

TASK ASSIGNMENT

This Task focused on the processing and testing of various polymeric films and fibers. The main area of research was the investigation of polymer conductive properties. The "workhorse" in this research was the polymer BBL. BBL was a prime candidate due to its numerous overlapping pi orbitals (Figure 1). Although there are two film processing methods, aggregation and casting, the current task was centered around cast film preparation because its quality was superior to that of an aggregated film. Both BBL film and fiber samples had their conductivity measured on a Keithly electronic test system. Variables, such as time and heat treatment temperatures, and their effect on the polymer's conductivity were examined.

This thrust to find the best intrinsically conducting polymer is a major task in the laboratory. The Air Force is interested in this area of research due to the revolution this could bring to aircraft electronics. This new role of polymers, aside from structural applications, emphasizes the Air Force's commitment to finding alternative materials to improve existing and future aircraft systems through materials research and development.

Cast film samples of BBL were prepared using a 1-2% solution of BBL in methane sulfonic acid (MSA), various molds and a sublimator (Figure 2). Sublimation time for the MSA to be removed from solution varied according to the desired film size and thickness. BBL fiber samples had been extruded earlier for mechanical property determination. Heat treatment was performed in a DSC chamber for 2 minutes. Heat treatment temperatures covered the 100°C - 600°C range in multiples of 50°C increments. Both film and fiber samples were mounted in IC sockets using silicon rubber inserts (Figure 3). This setup was then mounted on top of another IC socket that had current and voltage leads going to the Keithly test system. Software on the Keithly test system allowed the user to vary measuring time, effective length and current. The system then measured voltage and calculated resistance and resistivity from the inputs. Finally, the reciprocal of resistivity was calculated to determine conductivity. As stated earlier, much of the testing was done as a function of heat treatment temperature and time after heat treatment. Also, the difference between fiber and film conductivity was examined. Calculations of importance were:

Effective Length = A(area) / l(length)

$R = V / I$

Resistivity (ρ) = (R)*(Effective Length)

Conductivity (σ) = 1 / ρ

Upon completion of the preliminary testing, various interesting phenomena were observed. When conductivity was examined as a function of heat-treatment temperature, a nearly parabolic curve was produced with a maximum of around 300°C - 350°C for the BBL fiber. Testing of the BBL film produced a similar curve with a maximum conductivity occurring at 400°C. There was a definite increase and symmetrical decrease in conductivity over the heat treatment range of 100°C to 600°C. Often, three orders of magnitude difference in conductivity existed within the range of minima and maxima heat treatment temperatures.

This result was hypothesized to be the result of either a structural change or due to the production of free radicals within the polymer itself. If structural in nature, the change could then be either a chemical or a physical change to the polymer. To get an early indication of which of these two situations existed, various fiber samples were subjected to MSA solubility tests after heat treatment. However, further analysis is required. Conversely, if the conductive change was due to the production of free radicals upon heat treatment, then an increase should be transitory and reversible in nature. This hypothesis is based on the assumption that conductivity is directly related to electron mobility multiplied by the number of charge carriers.

Another area of emphasis dealt with how long this increased conductivity would last after the initial heat treatment at 400°C, since this produced the maximum conductivity. Conductivity changed little as a function of time within 8 hours after the heat treatment. However, 24 hours after treatment, the BBL film exhibited a two order of magnitude decay in conductivity. After three days, it had dropped another order of magnitude and within seven days another order of magnitude. This represented a total change of approximately four orders of magnitude from the conductivity measurement immediately after heat treatment. Although the film did exhibit rapid loss in its conductive properties within the first week, the sample was remeasured at 27 and 38 day intervals. These measurements showed less change and suggested a plateau was being reached with respect to conductivity loss.

A similar study was done on BBL fibers heat treated at 350°C. The fiber was measured every day for approximately 22 days. Interestingly, the fiber did not show an appreciable loss in conductivity over time. In fact, the readings remained quite constant over the entire measuring period. Day to day fluctuations were observed, but these differences were within an order of magnitude and plotted out linearly as a function of time. Electronically, the BBL fiber did exhibit ohmic behavior when the voltage was plotted as a function of current. However, the maximum amount of current that could be applied to the BBL fiber decreased as time increased. This showed signs of a long term trend; our data consisted of 21 points over a 527 hour post-heat treatment time.

Finally, it should be stressed that this is only the first bit of data from a preliminary study. There is strong evidence that the test geometry within the software does not match the experimental geometry. Additionally, there is concern that the electronic configuration of the Keithly test system may not be entirely correct. This is why no specific values were stated within this report. Despite this, one is still able to identify viable trends within the data. It is these trends that will garner more attention in the next series of experiments.

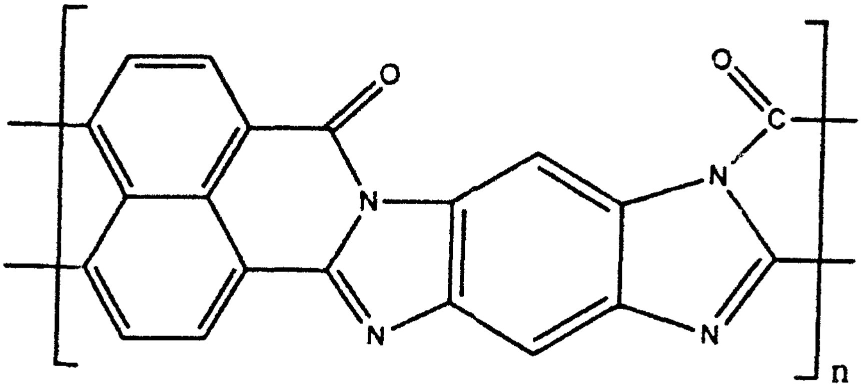


Figure 1: Structure of BBL

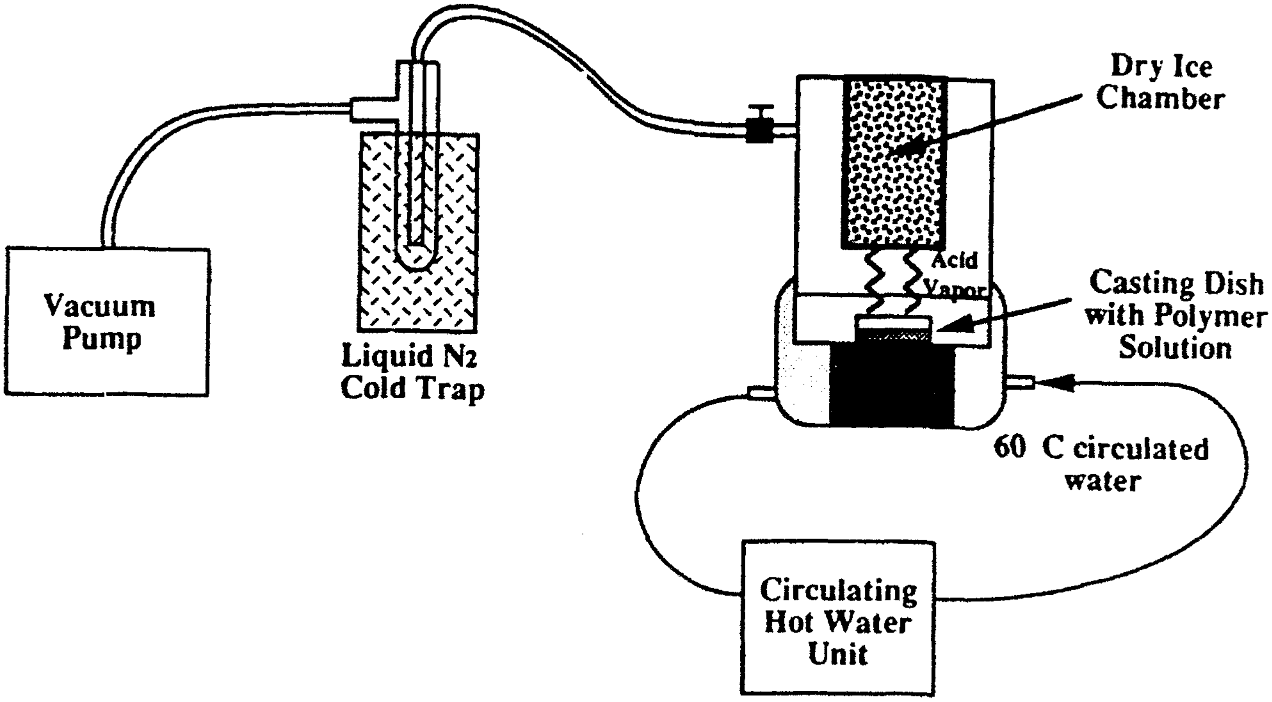


Figure 2 : Sublimation Chamber Set-Up
For Cast Film Processing

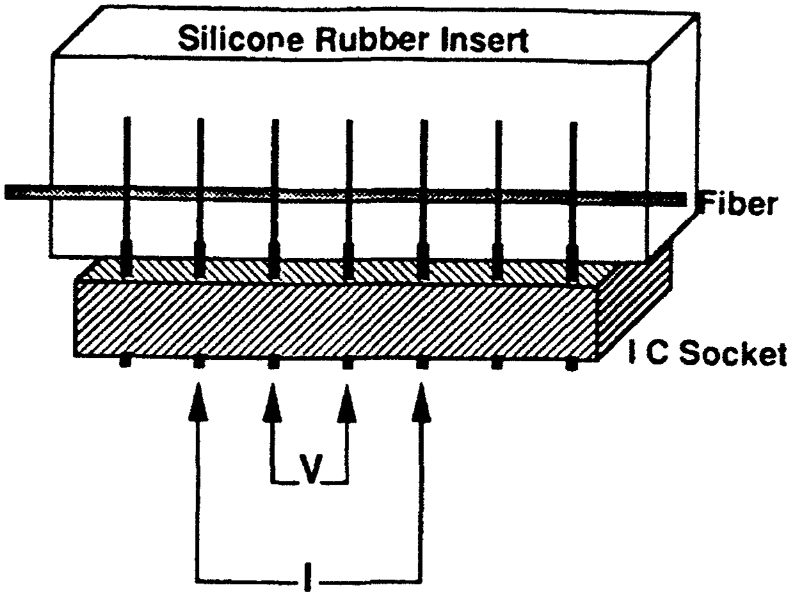


Figure 3. Schematic diagram of conductivity measurement device.

Processing and Properties of Molecular Composites

Task Order No. 70a
Student Support Program

Henry Derstine
University of Dayton

30 June 1992

Guiding Engineer
Dr. Robert Spry
WL/MLBP

ACKNOWLEDGMENTS

Thanks to Dr. Spry for his overall guidance, to Dr. Bai for guidance in optical and conductance testing, to Dr. Wang for processing instruction, Dr. Narayan, Mr. William Click and the rest of the polymer division.

TASK ASSIGNMENT

This Task required concentration in three main areas: casting of thin film polymers, study of optical characteristics of polymers and study conductivity of polymers. Each of these areas required me to learn how to use certain apparatus as well as build or refine equipment.

While casting the thin film polymers the use of a "doctor blade" was instrumental. The "doctor blade" was the device that spread the polymer over a glass plate creating the film, that later would be removed from the glass. Much of the time spent in the casting was concentrated on the thickness of the films. A thick film being of little use in the intended optical measurements because it would be too thick to measure the absorbance and transmittance. However, a thin film is much more difficult to cast, the polymer does not appear to have a uniform thickness as the film becomes thinner and holes may become present. This non-uniformity spread was believed to be due to the viscosity of the polymer and to try to alleviate this problem while maintaining a thin film, several different steps were taken.

Air was evacuated and the polymer solution was heated just prior to "doctor blading." This was intended to eliminate bubbles in the film as well as enabling it to flow more readily. The process helped, but the difficulties were not eliminated. The idea of heating the polymer was carried to another level.

The solution was heated while undergoing the "doctor blading." This was done by placing a small heater under the blade platform heating the glass substrate as well as the blade itself, improving the "doctor blading" process.

The testing of the conductivity also required some modification in the test equipment. The tests were done on a computer aided Keithley test instrument. A current of specified amperage was forced though the sample and the voltage across the sample as well as the actual current through the sample was measured and recorded by the computer. From this information, along with some physical measurements, the resistivity of the sample may be determined, which if inverted, will give the conductivity.

Modifications were needed in the sample holder, when the samples became thinner and thinner to meet the needs of the optical tests. These thinner samples on order of .02-.03mm would fracture or break in the old arrangement. Next was the process of building a new sample holder that not only could handle the fragile samples, but also met the requirements to give valid data back. These requirements were a good contact, a module made of material that would prevent current leakage, as well as a module in which the sample could easily be refused.

The first material we tried to construct the module of was a glass block. We soon realized that the conductivity of the glass itself was about the same as the samples to be tested that would create a problem with that data that we would be taking. Then we thought a quartz substrate would work for the conductivity of the quartz is on the order of 10-15. This did not happen because the quartz we would have used was not available in the lab. There was a rod of Teflon that is a better insulator than the quartz, so we then constructed the first module. Throughout its construction, the module underwent minor changes to its structure until it could produce the results that were needed. That module was not the final one to be built to alleviate the situation, which was done easily.

For the optical tests there was very little actual building or modifications involved with the test unit, a Hitachi U4001. The main problems dealt with the way a sample was to be held for the incident light source. This problem changed from sample to sample because each sample had its own characteristics. Some could be easily mounted on the given stand while others had to be cast on a substrate that was taped over the source opening. The other problem in dealing with the Hitachi was the preparation before each test scan. A base line scan was run to eliminate the noise in the system. For the films cast on a substrate, the reference beam had to be covered by the same substrate to nullify the effect of the beam on the beam. The graph was then plotted as intensity or absorbance as a function of wavelength. The information given by the graph was regarding the amount of light that could pass through the film at certain wavelengths as well as giving some insight on the structure of the polymer or perhaps if impurities had gotten into the synthesis procedure.

The time spent dealing in these three areas not only educated me about the specific areas, but also showed me how actual lab research is done. The data gathered in the experiments were important but the techniques used to obtain this data will be more vital for my future in the field of science.

Research on Ternary and Higher-Order Silicide Intermetallic Alloys

Task Order No. 71
Student Support Program

J. Renee Roesner
University of Cincinnati

31 December 1992

Guiding Engineer
Dr. Dennis Dimiduk
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank P.R. and Dennis Dimiduk for allowing me the opportunity to work with them. It has been a great experience, and the knowledge I have gained will be very useful during my approaching school quarter.

TASK ASSIGNMENT

During the task assignment at the materials laboratory at WPAFB four projects were involved in which different techniques of analysis were explored. The information obtained from each project was directed toward a common goal. This goal was to understand and develop new alloys for use in high temperature service in turbine engines. Achieving this goal would increase the life of some parts in an engine, thereby decreasing the frequency of repair, and it would increase the efficiency by allowing the engine to run at higher temperatures and speeds thus allowing the plane to fly faster.

In one project, the effects of oxygen melted in solid solution to TiAl were examined. Knowledge was needed regarding how large and numerous the oxides were within the surface of the specimen. The reason for looking at oxides is oxygen has a significant effect on mechanical properties, and the presence of oxides indicates that the material is saturated with oxygen in the solution. Oxides, like oxygen in the specimen, can make the material stronger. However, as the amount of oxides increase, the fracture toughness of the sample will decrease because the oxides have no fracture resistance.

In the second project, crystals were characterized by trying to grow a single large crystal. The point of this project was to understand slip by orientating large single crystal planes so slip can be recorded and mapped. This will be done by subjecting the specimens to both tension and compression testing. The mapping obtained can help us understand how the orientation of stress can ease or make difficult the slip motion; but the first major challenge is to grow crystals.

In the third project, the formation of phases in heat treated alloys with varying compositions of Ti, Nb, Si, Al, and Mo were studied for understanding. The specimens were heat-treated, prepared and examined in an SEM (Scanning Electron Microscope). From the examination of these specimens, we will create part of a phase diagram. In addition, selected specimens were subjected to a hot air furnace in which the oxygen was absorbed into the surface of the metal.

In the last project, the diffusion of Ta form (Mo,Ta) metal into the surrounding metal of $MoSi_2$ and the diffusion of Si into the (Mo,Ta) metal was observed. This was carried out by hot pressing a piece of (Mo,Ta) metal with surrounding $MoSi_2$ powder. The hot pressed item was then cut into pieces and heat treated by two conditions. Some were heat-treated at 1200°C for periods of twenty-four, one-hundred and three-hundred hours and the others were heat-treated at 1400°C for the same periods of time. Metallography was performed and then the samples were inspected on an SEM. To gain knowledge of what kinds of composition lies in the sample, an EPMA (Electron Probe Microanalysis) test across and in middle of the samples was conducted. Microhardness measurements were taken at the accepted distance of 0.5 mm. A graph was constructed of the distance form one side of the sample to the other side versus the time.

The four projects mainly required the preparation of specimens and the follow-up analysis. Many samples were ready to begin surface preparation but some needed to be cut down and mounted. A Struers Diamond Saw was needed to slowly cut the samples so no burning would mar the surface. After acquiring an acceptable size, the specimens were mounted using the compound Konductomet in a mounting press. The Konductomet was popular because it contained carbon that could conduct without the assistance of a copper wire or a silver paint coating; thereby facilitating examination electron microscopy.

The samples were prepared by two different methods. One method was to prepare their surface by hand on rotating grinding wheels of deceasing grit size. The samples were rotated ninety degrees after each grit size. In the rough polish stage, the specimens were rubbed in a figure eight form on a index card smeared with diamond paste that was kept lubricated by Hyperez solution. The specimens were polished with 15 and washed and polished with 6 and washed. For the final polish, the specimens were constrained in a weighted holder and placed in a machine called a Vibromet and Mastermet polishing agent. They were left in the machine for approximately two days and then washed and steam cleaned.

The other method was to use a machine called a Maximet. The specimens were firmly secured in holes in a metal disc holder by screws. The disc was then attached to an extended arm, the head, on the machine that would wither sweep across the platen, a rotating plate or remain stationary. The head would spin, whether it was sweeping or in a fixed position, in the opposite direction of the rotating motion of the platen. The platen was covered with the same decreasing sizes of a large grit paper. When the machine was finished grinding the specimens on each grit size, the metal disc was removed and washed. The next step was to rough polish the samples with the 15 and the 6 perforated Teximet cloth platens. The 15 platen replaced the plain platen, diamond suspension solution was sprayed onto the surface of the holder was again removed and rinsed. The 15 platen was scrubbed and rinsed. This procedure was repeated for he 6 platen. The next step was the Mastermet final polish. The plain platen was used with a Chemomet material adhered to the surface. The Chemomet surface was soaked with water and then the machine was started. The Mastermet was administered intermittently during the process. The specimens were immediately rinsed after the machine was done and released form the metal disc holder.

Some specimens dealing with the second project, characterizing crystals, had to be etched to make the grains visible. The surface of the specimens was rubbed with a swab of Knoll's solution (100ml H_2 O, 2-6.ml HN03, 103ml HF) and quickly doused with sodium bicarbonate to neutralize the acid.

The specimens were analyzed by a JOEL JXA - 840 Scanning Microanalyzer and a Hitachi S-900 Scanning Electron microscope. Many photographs were taken from different positions on the surface of the sample.

Since the start of my co-op at WPAFB, I have been able to improve the quality of my sample preparation techniques as well as learn new methods of preparation.

Mechanical Properties of Metal Matrix Composites

Task Order No. 72
Student Support Program

Michael Moran
University of Dayton

15 September 1992

Guiding Engineer
Mr. Stephen Russ
WL/MLLN

ACKNOWLEDGMENTS

During the past year I had the pleasure of working with some exceptional individuals. I would like to thank Steve Russ, Dana Hanson and Lt. Bill Revelos.

TASK ASSIGNMENT

For the last year experiments were conducted on SCS-6/Ti-24-Al-11NB and SCS-6/Beta 21-S metal matrix composites. Data and plot results were analyzed. Beta 21-S is a titanium-aluminide composite. Silicon carbon fibers are interwoven in the composite. Beta 21-S is a state-of-the art material being tested for use in National Aerospace Plane (NASP).

Experiments were conducted in thermal and thermo-mechanical fatigue in laboratory air at a wide range of temperatures and loading conditions. Data was collected and analyzed to determine stiffness and damage accumulation as a function of number of cycles. Graphs were also plotted with stress versus strain, stress versus cycles, strain versus cycles, loading modules, and strain (%) versus cycles and temperature curves. These plots were used for engineering evaluation and presentation.

Tests were run on cycles until they failed. Then the fractured surface was taken to the metallography lab. There the fractured area would be cut and sectioned, the specimen mounted in epoment, and the finished sample polished and etched. Then the sample would be analyzed on the optical and scanning electron microscopes.

In the metallography lab, mounting presses, grinders, low-speed saws, polishers and vibromets were used. Experiments were run on servo-hydraulic and creep test machines. IBM compatible and Macintosh Apples were used to collect and analyze data. Many computer programs were used in the collection and analysis, such as: Mate, Dana's, Excel, Word Perfect, Kaleidograph and Apple file Exchange.

When I started my Task in September, 1992, I was computer illiterate and very unfamiliar with beta 21-S and the testing procedure. With guidance form Steve Russ and others, I have become very familiar with Beta 21-S and the testing process. I am now comfortable in my ability to use any computer.

I have learned about thermal fatigue, iso-thermal fatigue, creep and thermo-mechanical fatigue (TMF) tests. I have gained a better understanding of advanced materials composites. I have also learned how to analyze graphs and understand failure curves.

Overall, I have learned a lot during my Task at WPAFB Materials Lab and have enjoyed working with a very professional engineering team.

Mechanical Properties of Metal Matrix Composites

Task Order No. 73
Student Support Program

Evan Dolley, Jr.
University of Dayton

14 October 1992

Guiding Engineer
Capt. William Revelos
WL/MLLN

ACKNOWLEDGMENTS

The author would like to thank Captain Revelos for his assistance and great leadership. A special thanks also to all personnel working in MLLN and the University of Dayton Research Institute (UDRI). All personnel was very helpful and gave a good example to follow. Dedication and professionalism prevail each day. Everyone has helped in making this learning experience very valuable.

TASK ASSIGNMENT

The requirements of the task assignment were to assist Captain Revelos in performing thermal fatigue testing of fiber reinforced metal matrix composites (MMCs) as well as data analysis and metallographic evaluations. The reason for doing this work was to help the National Aero-Space plane institute for the Mechanics & Life Prediction of High Temperature Composites (NIC) compile a data base for the investigation into different material systems in consideration for the plane.

In the beginning of the task the material SCS-6/Ti24Al-11Nb was being investigated only to be later ruled out thus bringing in the material SCS-6/Ă21S. The remainder of the task was devoted to the investigation of the Ã21S material system. The fibers in the metal matrix are oriented in three different layups that are [0],[0/90] and the [0/90þ45].

The material is thermally cycled in either a lab air or an inert atmosphere. A resistive heating element furnace was used to heat the specimen to the desired maximum temperature. Once the specimen was at the maximum temperature a pneumatic cylinder would react and the specimen would be removed from the furnace. The specimen then cools until it reaches a desired minimum temperature. The cylinder then extends and the cycle is started again. The tests were performed using different cycle counts and different maximum and minimum temperatures.

The first set of tests that was ran were controlled by using a Micricon controller. These controllers were replaced by a computer and Barber Coleman controllers. The software that was written by UDRI had to be fine tuned for each of the three furnaces. The new software was very accurate in keeping each time per cycle approximately the same. Accuracy was important since each test for similar conditions should be run nearly the same time for comparison reasons. The time/cycle and heating- cooling rates should be similar for each test ran under similar conditions.

Once the specimens are thermally cycles they are tensile tested. The tensile test reveals the mechanical properties of the material after being thermally cycled.

These mechanical properties include the residual strength, yield strength, strain at failure and modulus. The tensile test shows the integrity of the material after being thermally cycled in either a lab air or inert atmosphere. The data form the tensile test is then put into tabular and graphical form. The graphical form of the data includes the stress/ strain curve. The modulus of the material is determined from the slope of the stress/ strain curve. Other plots generated form the tensile test data include a comparison of residual strength and modulus with other layups. One specific plot may show the residual strength of all three layers. The data is also plotted against other material systems that are already in use to show the feasibility of the new material.

The next step beyond the data reduction is doing the metallographic evaluation. The specimens are first cut and polishes to reveal any cracking or oxidation as a result of thermally cycling the material. Two different orientations of the fibers are mounted in Epomet mounting material and then polished. This procedure of polishing MMC's is very difficult. Great care must be taken to ensure that an optimum polish is obtained. After polishing, the specimens are chemically etched and photographs are taken with a Riechert-Jung optical microscope. If needed the Scanning Electron Microscope (SEM) is also used to show a greater depth of field. This is why the polishing procedure is so important. The optical microscope cannot focus if there is a height differential between the fibers and the metal matrix. Therefore the fibers must be polished even with the matrix. This may be difficult since the fibers are much harder than the metal matrix and tend to polish away at a slower rate than the mental matrix. The SEM is also used to do the fractography work. Fractography shows the fracture surface of the material possibly showing the mechanism of failure. The reduced data and metallographic results are then looked at together and an evaluation is made as to how the material behaves at high temperatures when thermally cycled.

The thermal fatigue data is only a small part of the large data base generated for the material. Other areas include thermal mechanical fatigue, creep data, high cycle fatigue and low cycle fatigue. All the data from the lab is sent to NIC and at the end of the testing a decision will be made as to the use of the material SCS-6/X21S with the three different layups. This task has been a great learning experience since the work was done with very professional scientists and engineers that have much to offer in expertise.

Aeromechanics Experimental Research

Task Order No. 74
Student Support Program

Eric Lundberg
Ohio State University

31 December 1991

Guiding Engineer
Dr. Jim Olsen
WL/CA-F

ACKNOWLEDGMENTS

Thanks are in order to all at the Mach 6 High Reynolds Test Facility and the men in the Aero-Performance group at Wright Patterson Air Force Base for their help and support. A special thanks goes to Norm Skaggs and Capt. Lello Galassi for their time in the wind tunnel and their expertise in the area of areo-performance.

TASK ASSIGNMENT

The Aero-performance group at Wright Patterson Air Force Base (WPAFB) takes a look at different designs and concepts that apply to hypersonic aircraft and tries to make good predictive methods for prediction of the heat transfer, skin friction, drag and heating of localized areas on the aircraft body. Support was given in the setting-up of the wind tunnel and in the setting-up the initial conditions for the model. Support was also given in the reduction of data and in the checking of the reduction code for bugs and errors. Since limited knowledge of aero-performance was at hand, support only in the mechanical sections of the testing was permissible. Knowledge of the codes used was available due to Engineering Graphics 141 & 143 at the Ohio State University, so the checking of the code was also permissible. There were also lectures on the basic concepts of aerodynamics and aero-performance, of which these were a great help in the grasping of the ideas and concepts being used for the analysis of data from the wind tunnel. Several handouts and packets were given out so as to have a tangible resource from which to draw support for classes. One report was given on a special plane being developed for the Special Operations Force for long range missions. This was very informative and also gave an insight into the conceptual design of aircraft. These lectures are a useful resource for the grasping of fundamental concepts of aerodynamics being taken at the Ohio State University.

Some of the time was spent helping Dr. E. G. Brown-Edwards put together a report he had made on a flare model at different angles of attack in the Mach 12 hypersonic wind tunnel. Putting together pictures with titles and plotting the windward and leeward pressures from the shock wave were some of the duties performed.

The research of hypersonic aircraft was done in the Mach 6 High Reynolds Test Facility at WPAFB. The design being studied was a compression ramp used in the refining of predictive methods for use with the National Aerospace Plane (NASP). Different pressures were run at the same speed to accommodate for the varying pressures at different altitudes. Pressure ranges for heat tests were 100, 300, 700, 1400 and 2000 psia (pounds per square inch absolute). Skin friction and heat transfer were the main topics being studied, although pressure and temperature gradient data were also being taken and used in the predictive methods. Some of the equipment used was the Mach 6 wind tunnel, which actually ran in the Mach 5.7 - 5.8 range. Also used was a compression ramp, which was designed using a 132 inch radius circle, and was approximately 10 inches across. The model had various holes on the surface to accommodate the flush temperature and pressure sensors. Temperature was measured using thermocouples. Pressure was measured by comparing the pressure on the surface of the model with an absolute vacuum on the other side of a transducer. A pitot tube was used to sense the pressure difference from the model to the transducer. A Preston tube was used primarily in the measuring of skin friction by the measuring of the shear stress on the surface of the ramp. Laser Doppler Velocimeter (LDV) was used in conjunction with seed (fine oil particles blown into the freestream airflow) to study the boundary layer and how the freestream airflow reacted to the compression ramp. Schlieren photography was used to take pictures of the shock wave by the interference of light waves through the shock wave and the boundary layer.

A high-speed numerical data cruncher was used to take the data at an incredible speed and put it into a program which in turn set up a format for a computer data file that could be reduced later. Data was reduced by an IRIS minicomputer using FORTRAN codes written by project chief Capt. Lello Galassi specifically for this project. The data files were called up from the PRIME (the central net where data files are stored) and plugged into the program where the data was reduced into a form that could be plotted on a graph and easily interpreted. The data was reduced to temperature, pressure, Mach number, data point number, Reynolds number and Stanton number. The plots were of Reynolds number vs. Stanton number, which told about the temperature and pressure differences as related to other data points. These graphs were used to see if the predictions from CFD (computational fluid dynamics) were close to actual experimental data. This work experience has proven to be very beneficial in that several disciplines of aerodynamics were experienced. There was also the chance to see just how the ideas and theories that are used to design and explain flight are used. The data from the tests proved that the CFD analysis and experimental data lined up very well when compared to each other. The data will be used for the design of the NASP and for any other programs requiring Hypersonic data on simple shapes.

Electrical Properties of High Temperature Superconductors

Task Order No. 75
Student Support Program

Douglas Buchanan
Wright State University

17 November 1992

Guiding Engineer
Mr. Tim Peterson
WL/MLPO

ACKNOWLEDGMENTS

I would first like to thank those who have helped me with my first year. I would like to thank SOCHE for the contract and Wright State University Co-operative Education for suggesting that I apply. I could never offer enough thanks to the people I've had the opportunity to work with in this first year nor thank them enough for their patience in helping me. I would like to specifically thank Mr. Tim Peterson and Mr. Pat Hemenger. I also owe a special thanks to the many different scientists who have been able to answer my many questions, like Mr. Iman Maartense, Mr. Tom Kensky and Mr. Gerry Landis. I think it's most important to thank the one who trained me on almost every task that I am now able to perform (transport measurement, X-ray diffraction, and clean room operations), Ben Smith, another SOCHE student.

TASK ASSIGNMENT

It would be best if each operation was reported in full length discussion, describing each task and the reasons for doing that task. The most important part, is the transport measurements, where the critical values of the materials are obtained. Second, is the X-ray diffraction of the materials and their usefulness and third scanning electron microscope.

The task assignment objective was to measure the properties of high temperature superconducting thin films (HTSF's). The description is simple but the actual collection of data requires many different analyses. Concentration has been in taking the data during transport measurements, or in simpler terms, a material was cooled to a temperature at which an applied voltage no longer gave way to electrical resistance. This is the property of superconductivity and it usually occurs around 70K to 100K (-203 to -173 degrees Celsius). At these low temperatures, certain currents of electricity could be applied to a point where the sample could no longer superconduct and thus would transform back into an ordinary conductor. The current one can apply to make a superconductor return to its normal conducting state is called the critical current of the sample. Another aspect of the transport measurement, is to apply a certain current across a large temperature range. This type of measurement is called a resistance vs. temperature curve or simply a RT curve. With every sample this is one of the first transport measurements. Data from an RT curve can be used to tell what temperature the sample can no longer superconduct, called the critical temperature or Tc. The significance of the Tc is that information above the temperature is no longer considered, since it is no longer a superconductor. Instead the critical current of a wide range of temperatures below the Tc is measured in small increments. A relationship between the critical currents and temperature is then evaluated.

A high critical temperature or low one can partly be accounted for by the orientation of the crystals and by the surface structure of the film. At this point the actual physical properties, rather than inherent ones are looked at. X-ray diffraction gives indication what kind of crystal orientation exists upon the surface of the film. This can in turn lead to why the sample superconducts superbly or poorly. The X-ray machine holds the sample while X-rays are shot into it from many different angles. At certain angles the X-ray may be picked up by a measuring device. This leads to a graph that is flat for the most part, but then at certain points, it will have peaks. These peaks tell what kind of spacing there are between the crystals, the orientation the crystals have, and most importantly what type of material is being used. X-ray diffraction also has the advantage of being very inexpensive for all the information that it can give. It does not take very long to do an X-ray measurement, usually one can get a good measurement in about three quarters of an hour. (See Fig. 1)

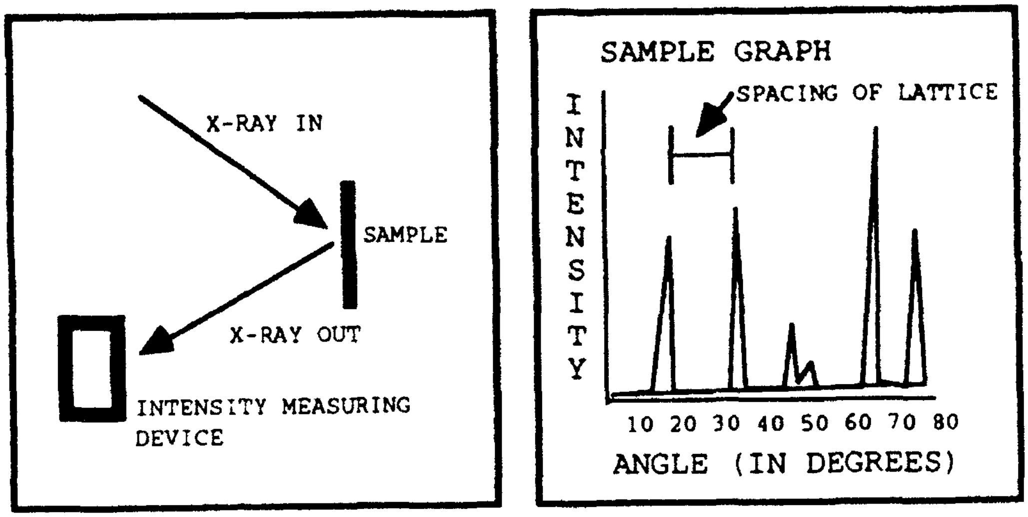


FIGURE 1
LEFT: HOW X-RAYS ARE COLLECTED
RIGHT: SAMPLE GRAPH OF DATA

Another useful physical property is to find out how the surface of the films' look. This also helps describe why a film is a good superconductor or not. The scanning electron microscope showing great magnification of the film, usually 1,000 to 10,000 times its normal size is adequate. The films are usually magnified and photographed for later analysis. It has been found that most samples with a partially roughened surface superconduct much better than those that are very smooth. The microscope itself is fairly complicated to operate and requires training before operation. Another type of microscope has been used to obtain extremely large resolution. It is the atomic force microscope (AFM) and is used to see down to about 50 angstroms (50 x 10^{-9} m) but to have this done the sample must be sent away.

The last task occurs before the sample is ever grown by laser ablation. Cleaning of the substrate, to which the sample will later bond, is of the utmost importance. It is the substrates' orientation that gives the superconducting material its orientation during growth. The smallest particle of dust can render a superconductor ineffective over a considerable area of film. For this reason, just before a films' growth, its substrate is chemically cleaned to rid all dirt. This is done by soaking the sample in three different chemicals. First, the substrate is soaked in heated trichloroethylene and then in cool trichloroethylene while applying ultrasound. After the first ultrasound, the substrate is rinsed. Next, the substrate is soaked with acetone in an ultrasound and then rinsed. Finally, the substrate is soaked in methanol with the use of the ultrasound. After the substrates have been cleaned and are free of dirt, they are put into an ozone oven for one hour. The final item before returning the substrates to their holders is simply blowing them and their holders with a small stream of nitrogen. All this insures clean substrates for growth of a superconductor.

These are the areas in which I have worked in my first year with SOCHE and the people here at Wright Labs. For me, it has been the chance of a lifetime and I hope I am able to continue learning and researching at the labs. There are other areas I will most likely become involved with should the opportunity arise. For example, there has been consideration in purchasing an atomic force microscope (AFM) which I would like to learn to operate. We are also starting to apply heated oxygen to the superconductors in an experiment to try to improve upon the films after growth, but this is in it's early stages. As one can see, there is a lot of opportunity here at Wright Labs.

Synthesis and Characterization of Polymers

Task Order No. 76
Student Support Program

Sungmee Kim
Wright State University

28 October 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to recognize Dr. Tom Cooper, Dr. L.V. Natarajan, Dr. Zbigniew Torarski, Dr. Tim Bidding, Tim Grinstead and Karen Hussong Myers for their assistance with my research of the ML/MLPJ.

TASK ASSIGNMENT

This task involves the synthesis and characterization of siloxane based polymer materials to incorporate spiropyran molecules for use as matrices for optically active materials. Also, mesogenic units (a component of a molecule that induces a mesomorphic or liquid crystalline phase) and leader groups that have photochromic and NLO (Non Linear Optical) properties were synthesized and characterized.

Synthesis: Various carbon length leader groups, allyloxybenzoic acid (3C leader group), penteneoxybenzoic acid (5C leader group), and vinylbenzoic acid (2C leader group) were synthesized by following a procedure modified by Tim Bunning from reference literature 167 except for the vinylbenzoic acid (Aldrich). Dicyclohexylcarbodiimide (DCCI) coupling reaction was used as a catalyst, dimethylaminopyridine (DMAP), to attach various compounds such as hydroxybipheny, cholesterol, hydroxy ethyl BIPS, 6-hydroxy BIPS, 4-phenylazophenol and pyrene methanol. Different solvent systems were used to purify the products in trying to find the best systems (cost and toxicity).

A few silanization reactions were conducted using a 90:10 ratio of the cholesterol-4-allyloxy benzioc acid: allyl BIPS in a D5 ring system and 1:1 ratio of azo diphenyl-4-vinyl benzoate to a D5 ring system. The silanization reactions were done under positive argon pressure with platinum catalyst in toluene at 80-90°C. The reactions were monitored by comparing the ratio of Si-H peak to C=O peak with the FTIR (Fourier Transfer Infra Red Spectroscopy). These various reaction products were purified by recyrstallizations, TLC (This Layer Chromatography), and flash column chromatograhpy that were analyzed by either FTIR, Lambda 9 Spectrophotometer, NMR (Nuclear Magnetic Resonance), elemental analysis, mass spectroscopy and/or DSC (Differential Scanning Calorimeter).

Results: The silanization reaction with vinyl group did not go to completion after 137 hours. Temperature above 100°C may drive the reaction to completion. Several different ratios of solution mixtures were used in the purification process of cholesterol-4-vinyl benzoate (C4VB) and biphenyl-4-vinyl benzoate (B4VB). B4VB can be purified by using acetone, and C4VB can be purified by a ratio of 1:1 ethyl acetate: ethanol. The reaction of hydroxy ethyl BIPS and allyloxy benzioc acid to obtain the "T" product was successful, but the yield after purifying by flash column chromatography was very low. The reaction of 6-hydroxy BIPS and allyloxybenzioc acid to obtain the "I" product was thought to be unsuccessful, but later this was found not to be true. More synthesis and examinations of the "I" product will be done.

Advanced Composites

Task Order No. 77
Student Support Program

Deborah Gill
University of Dayton

31 December 1992

Guiding Engineer
Mr. John Russell
WL/MLBC

ACKNOWLEDGMENTS

Many thanks to Mr. John Russell who patiently taught me all I know about materials Engineering and Volumetric Dilatometry and to Dr. Saliba who guided me in my research.

TASK ASSIGNMENT

The mechanical and physical properties of thermoplastics are dependent upon their processing thermal history and environmental conditioning. Previous work has utilized volumetric dilatometry to study the PVT characteristics of PEEK resin. Furthermore, thermal history and solvent exposure were shown to have an effect on the mechanical properties of APC-2. This study used volumetric dilatometry to examine the effects of thermal history and solvent exposure on the PVT characteristic of the APC-2 composite.

The effects of exposure to JP-4 jet fuel and to water on APC-2 were examined. Coupons with three different thermal histories; slow cooled, quenched and quenched then annealed, and two different layups; $[0]_{12T}$ and $[+/-45]_{2S}$, were soaked in JP-4 and water. The percent weight gain due to absorption of the solvent was found to be dictated by the degree of crystalization in the [0] coupons. While the percent weight gain due to absorption of the [+/-45] coupons was dictated by residual stress. The least crystallized quenched samples experienced the greatest weight gain due to absorption of JP-4 of water. The pressure, volume, temperature relationship of the slow cooled and quenched, dry and JP-4 exposed coupons were studied through the use of volumetric dilatomety. The melting temperature, Tm and the recrystalization temperature, Tc were determined for most of the samples. Overall, the JP-4 served to lower the Tc and Tm while the thermal history had little effect.

Advanced Composites

Task Order No. 77a
Student Support Program

Robert C. Weber
Wright State University

31 December 1992

Guiding Engineer
Kristen Kliner
WL/MLBC

ACKNOWLEDGMENTS

I would like to extend my thanks to the leader of this task, Kristen Kliner, as well as to Wanda Vogler and Pamela Douglas and SOCHE for the guidance and support they provided.

TASK ASSIGNMENT

The advanced composite task consisted of a software engineering design dealing with the carbon-carbon composite pyrolysis process. The general design collected data acquired from the process, and manipulated the data for display purposes. The program also served as the main storage and retrieval system for the data. In the future, the program also will ideally serve as a closed loop control source from which the carbon-carbon composite process can be controlled in a manner that aids in the efficiency of the process. The main goal then, of the computerization of the process is the idea of speeding up the process while at the same time preventing deficiencies (i.e. cracks and delaminations) in the composite formation.

The design of the program to collect data began with gaining a familiarization with the data acquisition equipment and the data processing unit. The data acquisition equipment included an 8900 Locan AT Acoustic Emissions Analyzer, and an ACRO-400 Data Acquisition and Control Unit. The main processor used was an INTEL '286 based personal computer (PC).

The Locan AT Acoustic Emissions Analyzer was used to collect information relating to sounds generated by the cracking of the carbon-carbon composite as it was heated. At some point it is hoped that this information will lead to signature signals that are easily identified. This information will then be used in the carbon-carbon composite process in a manner that will prevent such signals from occurring. The main type of noises generated in the process come from either cracking, or delamination. This information is sent to the PC for analysis and storage.

The ACRO-400 Data Acquisition and Control Unit was used to collect temperature information from thermocouples attached to the composite, and to the furnace. This data was very important as well, because it was the driving factor in the carbon-carbon process. It was also taken into consideration that the raw voltage data may be needed at sometime in the future.

The PC was used to collect, display, and selectively save all the incoming data. This implementation was not completed, but was partially implemented using a previously written prototype program. The prototype was written in the Microsoft QuickBasic Version 4.5 computer language. The final program also will be coded in QuickBasic. This programming project is essentially the heart of the task to be completed.

This technical report is based on work undertaken on a part-time basis for less than one month. The task was not "completed," but the end of the year meant a technical report needed to be written. The partial work put into the task so far was essentially a learning period. From the work so far several things were learned. First, a better knowledge of the QuickBasic language was gained through hands-on programming and intensive study of all available QuickBasic texts. Second, the basic principles of acoustic emissions data sampling were broadly surveyed, sample experiments were undertaken, and basic information pertaining to acoustic emissions was read. Finally, the prototype program for the task was briefly studied and in doing so the need for good software design practices were emphasized.

Systems Support Materials Behavior and Evalution

Task Order No. 78
Student Support Program

Cathleen Gustafson
Wright State University

31 December 1992

Guiding Engineer
Mr. Robert Urzi
WL/MLSE

ACKNOWLEDGMENTS

This task was completed with the help and guidance of the immediate supervisor, Jim McKiernan and MSgt. Bryan Cramer. Mr. Robert Urzi, the Tech Area Manager was very supportive. The engineers in the section, especially, Jim Mazza, Mark Forte, and Brett Bolan were very helpful and willing to answer any question.

TASK ASSIGNMENT

In Composite Supportability, the work is centered on testing composites and adhesives. Testing is done mainly on different types of adhesive systems including; film adhesive, two-part paste adhesives and resin systems. The tests are done by making test specimens, including but not limited to, lap-shear panels, blister panels, composite panels for four-point flex-shear and compression tests, flat-wise tension specimens, wedge specimens and plate shear specimens.

All the panels have to be cured either in a press, in an autoclave, in a vacuum bag or zip-vac system. Different specimens are prepared different ways for the actual testing. The panels have to be cut and machined to the correct size. Exact measurements are taken to be used in the final calculations. The actual testing is usually done on a tensile machine.

The Air Force is continually looking for better, faster, and less expensive ways of doing things. One reason for these tests is to determine the best product. Companies send in samples of their products in hope that the Air Force will determine that their product is the one to use. Some of these products are tested by the group. They also try to develop better ways of doing things like repairing battle damage. This group will also double check other people's findings if the result could have an impact on the Air Force.

The results of the tests can have direct and immediate impact on how things are done. If a new method is developed to make patches for repairing battle damage, the new method will be used. One of the immediate projects going on is to repair a crack in a part on an airplane that has never been repaired before. If the group can come up with a way to do this it will save the government millions of dollars. This is not an isolated problem but is on all the planes of this type.

Also, the better preforming adhesives will be used. One of the ongoing projects is doing an extensive in house study on the shelf-life of film adhesives. By finding which adhesives keep their properties longer, the Air Force will save money by not wasting adhesive that is not used very fast. The results are usually published so others can benefit from the research.

An engineer first has to submit a project plan before anything can be done. Once a plan has been approved and assigned a number, the project engineer goes over it with the technician. The technician is then responsible for seeing that the project is completed.

The first thing done is to make sure that all the supplies required are available and are still within their shelf life. The next step varies depending on the type of testing that is going to be accomplished. Metal to metal panels (i.e. lap-shear) have to be cut to bonding size, the surfaces prepped by either the Phosphoric Acid Anodized (PAA) or Pasa-Gel 105 method and sprayed with primer before they can be bonded. Composite panels are made by a procedure called a wet lay-up. The adhesive for the wet lay-up is usually an epoxy resin system that is spread evenly on the composite cloth. The cloth is then cut to the correct panel size. Both types of panels, metal and composite are cured according to the test plan. Different curing methods yield different results. Next, specimens have to be cut from the panels. The metal specimens are cut using a band saw and machined smooth on a milling machine. The composite panels are cut with a cutting wheel. It is important to get some specimens uniform in size and shape because shape directly affects the test results.

The type of test that is to be run is decided by what specifications are needed. Most all the tests are run on a tensile machine. The tensile machine can be configured for various types of tests. A plot of the test results can be obtained by connecting the tensile machine to a plotter.

The results of the tests are complied by the technician and given to the engineer. The engineer then analyzed the results and determines if additional testing needs to be accomplished. If everything is complete, the engineer writes the final report showing the results of the tests.

Co-oping was the best decision made. This task provided many opportunities to apply knowledge gained in school. The practical experience obtained in this task will aid in the understanding of several topics in future classes. Also, understanding the types of machines implemented and their capabilities will be a valuable asset as an engineer. The task also helps to figure out if this is the career field of choice.

Systems Support - Materials Behavior and Evaluation

Task Order No. 79
Student Support Program

David Collins
Ohio State University

31 December 1992

Guiding Engineer
Mr. Robert Urzi
WL/MLSE

ACKNOWLEDGMENTS

I would like to thank all the personnel in the Systems Support Group for making my stay both enjoyable and educational. Special thanks to those I worked directly with: Mr. Robert Urzi, Mary Cooper, MSgt Brian Cramer, Jim Mazza, Mark Forte, and Brett Bolan, of MLSE: James McKiernan, Bill Lawless, Gary Andrews, Bob Hicks, and Dale Pearson of the University of Dayton Research Institute; and fellow SOCHE students Brian Knapke and Cathy Gustafson.

TASK ASSIGNMENT

In the second quarter of my work with the Materials Directorate Systems Support Group, I continued my work as a lab technician. As such, I performed such tasks as specimen preparation, mechanical testing, data reduction and reports. However, my level of input into the decision-making parts of the process considerably increased, due both to my increased knowledge of the research methodology and the long-term absence of the primary technician, Jim McKiernan. Where before I had operated primarily under the guidance of Mr. McKiernan, this summer I was basically on my own in performing tasks I had learned earlier.

Over the course of the quarter, work was primarily focused on two projects. The first of these was the continuation of a project from the Air Force Office of Special Investigation (AFOSI). This project involved the testing of actual aircraft parts removed from planes currently in service. The goal of this testing was to gather data on the properties of the adhesive used in these parts. This data would help determine what, if any, contract violations the manufacturing company had made.

The second project involved obtaining data on a number of room-temperature storable, low-temperature curing epoxy and adhesive systems. In contrast, most field-level repairs to composite aircraft components use adhesives or graphite/epoxy materials ("prepregs") that cure at temperatures of 250°F or above, and must be stored at 0°F; this, in combination with a short shelf life, often necessitates discarding large amounts of unused material. The object of this research was to determine how well these newer, more usable and storable adhesives and prepregs meet Air Force requirements.

The AFOSI investigation required the use of techniques learned the previous winter for this project. Specimens were provided by the OSI in several formats for several types of tests: lap-shear, honycomb-peel, and flatwise-tension, each at room temperature, 350°F, and after 3000 hrs at 350°F. Shear specimens and honeycomb-peel specimens required very little preparation, and could be tested almost as-is. Those specimens slated for flatwise-tension testing had to be first bonded to aluminum testing blocks before they could be tested. All specimens were tested using the Instron testing machines. Ultimate strength, failure modes, and other pertinent data were calculated, tabulated, and submitted to the project engineer and the OSI for analysis.

The room-temperature storable materials project involved a large number of different materials: three different film adhesives and three prepreg systems, two of impregnated graphite cloth and one of impregnated unidirectional graphite. With the adhesives, three types of tests were used: flatwise tension, floating roller peel and lap shear. Each of these consists of a piece of adhesive sandwiched between two pieces of aluminum. First, the aluminum panels had to be cleaned and surface abraded. Next, they were anodized in a dilute phosporic acid bath, which was prepared from concentrated phosporic acid. The aluminum then had to be primed with a spray-on primer and dried before bonding. All specimens were bonded and cured under pressure in a small press, with the exception of a few lap shear panels cured under vacuum in a vacuum-bag. All panels had to be cut, measured and tested under conditions varying from -65°F to 250°F. Further samples of adhesive were set aside for testing after extended aging, to confirm their room-temperature storability. The prepreg panels were layed up, and cured in an autoclave on the U.D. campus. However, once cured they were turned over to me for cutting, measuring, and testing. All mechanical testing was done in W.P.A.F.B facilities, with either the Instron or Tinius-Olsen testing machines.

During my summer work term, the AFOSI project, which had begun during winter term, was finally completed. Data collected and calculated was submitted to the project engineer and the OSI for further review. The results and conclusions for this program remain restricted information.

As for the room-temperature storables project, this is an investigation which will continue as an ongoing project for several years. At the time of my departure, insufficient data had been collected to make any conclusions - the size and scope of the project was truly immense.

My experience at the lab proved, again to be personally educational and practical. I continued to learn the basics of the use of composite and adhesive materials. More technically demanding tasks, such as phosphoric acid anodizing, adhesive primer application, and post-cure specimen treatment, were added to my list of tasks. As I became more immersed in the lab environment and dealt with an increasing number of engineers, technicians and managers I grew aware of the importance of establishing communication and dialog between all parties and documentation of both work plans and work performed. In general, this second co-op experience allowed me to experience more deeply a working materials laboratory, in all aspects from hands-on laboratory work to management. I feel I have gained a lot of practical experience and knowledge that will help me in the future. I regret that I will be unable to return as a co-op student in the Materials Lab, but I know that my experience has been both valuable and memorable.

Tribological Studies on Research Fluids

Task No Order. 80
Student Support Program

Scott A. Long
Wright State University

31 December 1992

Guiding Engineer
Dr. Wayne Ward
WL/MLBT

ACKNOWLEDGMENTS

I would like to thank Shashi K. Sharma and Ronald P. Mortimer for their part in helping me throughout the task. They were very understanding when I needed help, and they allowed me to progress at a pace set by myself.

TASK ASSIGNMENT

While working under this task, one primary job had been performed. However, within this job were many smaller jobs. Beginning in February, a hydraulic pump test stand had to be modified. The stand was constructed to handle a F-16 hydraulic pump, but the new orders were to test B-1 hydraulic pumps. The B-1 pump is approximately four times larger than the F-16 pump, so this meant some major rebuilding of the test stand.

The process of modification involved designing new flow patterns of the hydraulic fluid and integrating a new cooling system. The cooling system consisted of base water, at normal temperature, being pumped through three heat exchangers and into a drain. The hydraulic fluid also goes through these heat exchangers, but at a slower rate, therefore the water cools the fluid. The new set up of the test stand was almost completed by the end of July, but two of the heat exchangers were new, and they had to be cleaned thoroughly. Normally, the tube side of the heat exchangers circulates the water, but our situation has the hydraulic oil circulating through the tube side. After scrubbing the 420 tubes in the exchangers, metal chips were observed to be in the tubes. This was unusable, and seemed to have come about from the process of roller expanding the tubes at the factory. At the suggestion of the manufacturer, non-hydrocarbon fluid was used to exhaust the chips. However, this process failed, and brass wire brushes were then rotated by a drill in each end of the tubes. Finally, after this procedure, and a final rinsing, the chips were removed.

At the beginning of October, the entire hydraulic circuit was completed, and the first test fluid, MIL-H-5606 (MLO 92-80) was put into the stand. The stand was bled of air, and attempts to bring the pump up to test speed (5,250 rpm) failed. The pump depressurization circuit was then utilized, and after a speed of 5,000 rpm was reached, the circuit was de-energized. There were a variety of problems occurring during each start up, and as a result, small modifications were performed. Near the end of October, the circuit that runs the primary motor failed, but after a week, the problem was located and fixed.

Finally, at the time of this paper, the test stand ran up to the proper speed, but the flow of 54 GPM had not been reached. Modifications were still taking place, and hopefully within a few weeks, the B-1 hydraulic test will officially begin.

I have learned a great deal about hydraulic pumps and hydraulic fluids during my task. Also, I obtained a lot of hands-on work while constructing the hydraulic circuit and preparing the heat exchangers. Within the near future, I hope to run the test stand and analyze the results, but for now, being able to construct the stand is a great accomplishment. I always try to do my best at every thing I do, and I feel that because of this, I can succeed in anything I do. I am grateful to be given this opportunity to enhance my knowledge, and I hope that I will be able to further my knowledge in the time to come.

Smart Skins and Structures

Task Order No. 81
Student Support Program

Steven H. Boyd Jr.
University of Dayton

31 December 1992

Guiding Engineer
Capt. Michael W. Holl
WL/MLBC

ACKNOWLEDGMENTS

I would like to thank Capt. Michael W. Holl for the opportunity to work at Wright-Patterson AFB. His guidance and suggestions made the task assignment both informative and enjoyable. I also would like to thank Jim Lute for the training I received at the beginning of my task assignment. There are many other people I would like to thank but the list would be too long.

TASK ASSIGNMENT

The objective of the first task assignment was to study the effects of fiber optics embedded into advanced composite laminates. The mechanical properties were studied to determine if these inclusions had any adverse effects on the composite. This information will help to determine the possibilities of fiber optic embedment into composites for smart skin and structure purposes.

The second assignment was to examine the effects of cyclical loading on a fiber optic Bragg grating sensor. The objective was to examine the stress vs. strain characteristics of the sensor at varying fatigue cycles and compare it to a commercially available foil strain gage. This will help to determine the behavior of the Bragg strain sensor. The size and sensitivity of the fiber optic Bragg grating sensors are optimal for health monitoring systems of aircraft and characterization of advanced materials.

The smart skin study used 125 μm and 240 μm outer diameter fiber optics that were embedded in the carbon composite and the static performance of the material was evaluated. The fiber optics were placed in the midplane of the specimen both parallel and perpendicular to the loading direction. The orientation of the specimens varied. The static tests performed included the following: tension, compression, impact and first-ply failure.

The composites were cured in an enclave at 350° F for two hours. The specimens were cut by a diamond blade wet saw and the static tests were conducted using appropriate testing equipment. A Scanning Electron Microscope (SEM) was used to examine and document the damaged specimens.

The data collected was used to determine if there was a reduction in the static strength or modulus of Elasticity when fiber optics were embedded. The SEM and other documentation indicated whether or not the fiber optic influenced crack propagation or initiation in the composite.

The Bragg grating is formed by exposing a short section of the fiber to coherent UV light beams. This produces permanent variations of the index of refraction. This grating forms a band pass filter that reflects the wavelength that is in resonance with the gratings. The wavelength that is reflected constructively interferes with the light of the same wavelength.

The Bragg grating was attached to a carbon composite. The specimen was secured to a mechanical testing fixture for cycling and applying load. The specimen was cycled for 110, 1110, 10,000 and 100,000 times at 5 Hz between 32 lbs and 325 lbs. The specimen was cycled the appropriate number of times and then the load was applied in 50 lb increments until 500 lbs was obtained. The strain, load and the light spectrum data were recorded at each increment.

The results showed that the embedded fiber optics have little or no effect on the static strength or modulus of elasticity. The results show that the interfacial bond between the fiber optic glass core and coating were poor as well as the bond between the coating and the composite resin. This did not, however, increase the chances of crack propagation or initiation in the composite near the fiber optic.

The investigations showed that the Bragg sensor is sensitive to longitudinal strain. The experimental results showed that the sensor does not exhibit the hysteresis that was expected but the sensor does become more erratic as the number of cycles increase.

There were dual peaks in many of the light spectrum data. The difference in the strain between the Bragg and foil gage is likely due to the difficulty in determining the exact wavelength that satisfied the Bragg condition. The erratic behavior could also be contributed to the adhesive that was used to attach the Bragg gage to the composite. The adhesive may have experienced some relaxation during the cycling.

There are some recommendations for the continued testing of the Bragg sensor. The sensor should be removed from the specimen and retested. This should determine if the adhesive experienced any relaxation and/or the Bragg gage encountered any hysteresis. Another experiment should have the Bragg gage being tested under wet conditions.

The cycle load should be increased so that the fatigue on the Bragg sensor is larger. This should increase the possibility of hysteresis. The light source could be changed so that the light intensity is a constant throughout all wavelengths.

Rapid Design Systems Features Development

Task Order No. 82
Student Support Program

Barry Caslin
Wright State University

31 December 1992

Guiding Engineer
Mr. Jim Malas
WL/MLIL

ACKNOWLEDGMENTS

I would first like to thank Mr. Jim Malas and all the members of the Rapid Design System working group for the faithful assistance and abundance patience which every member of the RDS' team extended. I would be remiss if I failed to mention the insightful and critical direction that was shown me by Dr. Radack, who was the researcher with whom I completed the majority of my Task and was the person whom I worked closely with daily.

TASK ASSIGNMENT

The primary task was in the area of testing, debugging and modification of the graphical user interface of the Rapid Design System (RDS). The Rapid Design System (RDS) is a high-level design system that incorporates expert knowledge about probable production and inspection. A feature-based approach is used to describe part geometry, manufacturing information and inspection information. In traditional CAD systems, design information is represented in terms of geometric and topological primitives (points, lines, surfaces, vertices, edges, faces, etc.) and text. Feature-based design is an attempt to establish a higher level of representation, i.e., primitives (features) that have meaning and are interpretable by both the design system and the designer. It is also the intent of the designers of the RDS system to make this feature based system very intuitive and simple for the designer to use, which today means using a Graphical User Interface (GUI). The GUI was updated and modified so it would meet the needs of the designers and future consumers.

The RDS is a system that runs on a Sun work station and uses Unix. The RDS exists within an Emacs environment and is coded in Common LISP under a package called Wisdom that has many features that allows the programer to interact with the part geometry, as well as allowing the user to manipulate the GUI in a fairly efficient manner. Also at times it was necessary to program in C.

In completing this project I had to learn Emacs, Wisdom, Common LISP, Unix system support and some X-windows programming. To accomplish the intended goals of this project, it was necessary to first `poll' the developers and the group's first intended user (the 4950 Test Wing) and discover what the user liked and disliked about each of the interfaces. It was then necessary to reconfigure not only the interfaces to most all the features, but also to rework the main display to match the setup of screens designed by other design groups within the RDS project itself. A GUI was added to handle the output of any part-model to a post-script laser printer. Many bugs were discovered in all parts of the system, all were reported and verified. These were repaired as well. In evaluating the Bug reporting system, it was necessary to rewrite the entire data base system from the ground up to fix the errors.

Maintaining our local area network of five sun work stations became part of my responsibilities. This included restructuring the entire UNIX file system and upgrading the operating system to SUN O/S version 1.4.3.

Feature-Based Manufacturing Research

Task Order No. 83
Student Support Program

Hilmi AL-Kamhawi
Wright State University

31 December 1992

Guiding Engineer
Dr. James Malas
WL/MLIM

ACKNOWLEDGMENTS

I would like to thank Dr. LeClair, Dr. Pao, and Dr. Chen for their help and support.

TASK ASSIGNMENT

The Task started on January of 1992, and it involved working on the development of the Rapid Design System (RDS). The RDS system is an attempt to develop a Next Generation design system that enables the coupling of design intent directly to product geometry. The central purpose of RDS is the integration of product and process design across materials and processes. The RDS is a feature based system in which the designs are made from basic blocks that are the design features. The uniqueness of RDS comes from the fact that it uses the Episodal Associative Memory (EAM) to store past designs. The designer can retrieve past designs by providing partial descriptions of the desired design. This means that a designer could use experience to improve her/his designs.

It was necessary to go through the Concept Modeler (the programming environment used by RDS) manuals before starting work on RDS. After reading the manuals, writing short programs in the Concept Modeler environment was possible. The next four weeks involved maintaining and testing the manufacturing environment. Working on ensuring that the manufacturing team from the University of Cincinnati communicated with the Feature Based Design Environment team, involved integrating code written by different groups of people with different concerns. While integrating the different pieces of code extensive testing had to be performed to make sure that the system worked properly. This part was completed the end of June.

In July work started on the research aspect of the project. This stage involved working with the memory part of the project. This stage started by studying some of Dr. Pao's theories concerning memory. Under the direction of Dr. Pao, Dr. LeClair and Dr. Chen the group started looking at the possibility of using a uniform data representation that can be used in RDS and in other areas. This involved using the 'Sand Box' approach (Dr. Pao), in which the memory is randomly populated, and certain units in the memory light up if they satisfy certain conditions. About ten different versions that implement this idea have been written. Each version uses a different search strategy for looking into the memory. At this time the group is focusing on using a genetic algorithm, simulated annealing and the 'Sand Box' to solve some of the manufacturing problems.

At the materials lab we are trying to create a conceptual memory (Dr. Pao, Dr. LeClair). This memory is fed a number of instances and is supposed to generalize and generate concepts and categories. For a start this memory is to be used to perform feature sequencing in RDS (an np-complete problem.) At this point the group is using Sun Sparc-2 stations. In the future (Dec., 16), the group will be using Sun Sparc-10 stations. The code is being written in C, and then will communicate with LISP using the foreign function interface provided by the Concept Modeler.

Different methods for looking into the 'Sand Box' have been experimented with. The group is also looking at different methods that might be used in solving the problem of feature sequencing. Our concerns lie in the area of computational time and the quality of solutions obtained.

It is appropriate to divide the work that was involved in this Task into two parts; a part where work involved performing software integration and engineering more than a programming or research, and a part where research was the main focus and thrust. Both parts were (are) exciting and provided a good learning experience. Part 1, involved software integration, testing and some project management. This part didn't involve writing large pieces of code, instead it involved modifying someone else's code or explaining the specifications that their code had to satisfy. Valuable experience was gained on how to deal with people who have different concerns, and on how to mediate between different groups. Part 2, involves the research aspect. Here the group tried different methods to solve an np-complete problem, some methods provided encouraging results others didn't. At this point the group is developing a hybrid method that combines two areas of artificial intelligence: Neural nets (coarse coding), and the area of genetic algorithms. It looks like the problem will be solved, and a memory that is able to conceptualize will be produced. The results expected will be exciting and could be implemented in other areas.

Design Methods for Manufacturing Processes

Task Order No. 84
Student Support Program

Daniel Johnsman
University of Dayton

31 December 1992

Guiding Engineer
Dr. James C. Malas
WL/MLIM

ACKNOWLEDGMENTS

I would like to give special thanks to Dr. James C. Malas, Venkot Samarathan, Carl Lombard, Steve Adams, Srivates Gopinathe, Baxter Turner, Bob Goetz, Walter Griffith and Oliver Patterson.

TASK ASSIGNMENT

The internship job with the Wright Patterson Materials Research Directorate involved statistical analysis, conducting curve fitting, and solving systems of equations. Throughout most of the contract, work was in some way related or would lead to a result in one of these jobs. This meant working with people from different areas of expertise from doctorates to other students that helped finish the Task, also using many different types of equipment to aid the work.

The Manufacturing Technology branch at WPAFB studies new or improves existing materials and manufacturing processes and then transfers the technology to applications for the Air Force and local industry. Although, work did not directly relate with some of the projects in this group, they worked on such interesting research projects as the Molecular Beam Epitaxy (MBE), a new thin-film semiconductor growth technique that actually deposits the semiconductor material on a substrate. The MBE "grows" a semiconductor used for computer chips by depositing vaporized materials on the molecular level and by controlling precisely what material and how much is being deposited.

The RDS (Rapid Design System), which in a simplistic way, may be compared to a CAD drawing program. A drawing is made using another program, then the entities in the drawing can be used to create machine code for a Numerical Control machine that will machine a detail from a piece of material. The RDS simplifies and speeds up the process for the designer by automatically generating the machine code as the designer "draws" his part in an optimized feature based drawing program that uses the features (rectangles, cylinders, holes, etc.) instead of entities to design the detail.

The first few weeks of the work period were spent getting acquainted with the materials laboratory, the people and my part in all this. I took a tour of the facilities with Dr. James Malas, my work coordinator, and experienced the many things the Materials Research Lab does for the Air Force, including the transfer of technology to private industry. The materials lab primarily does testing and formulating of new materials and processes that are applicable to manufacturing.

The job was to help Dr. Malas who is a materials research engineer investigating the area of processing science. Dr. Malas used material behavior models to incorporate materials characteristics into process design and control methods. The material behavior models that Dr. Malas worked on allows a designer with limited knowledge of a material to design a process. So, when designing the process the material responds favorably or produces favorable results when the designer uses the model to transform a work-piece into a useful finished product by employing manufacturing processes. The materials that were worked with during the term consisted of several pure metallic and metallic alloys.

At the start of the term I spent several weeks reading articles and journals that relate to what Dr. Malas is doing and became familiar with his work. Dr. Malas then assigned some systems of equations (matrices) to solve that relate to the process control of a carbon steel. The equations to process the steel and some others to process steel alloys relate Temperature, Strain, Strain Rate, Grain Size and Unrecrystallized Grain Size in 2x3 and 3x3 Matrices. The equations had to be solved for several different grain sizes that resulted in many calculations. To speed up the calculations several short software programs were written on a Macintosh. The program was called Mathematica and the programs employed a Do Loop to take care of the many iterations involved in calculating all the data.

Work began on quantitative measurements in metallography. This involved taking measurements of grainsize, the structure, in a material to measure all the polygon shapes in a magnified photograph of a highly polished sample. Also, different magnifications of the grain structures were looked at to see the unrecrystallized structure and the recrystallized structures. Unrecrystallized grains are those that are not broken down (before tension or compression tests). Recrystallized grains are those that are broken into small crystals as the result of the stresses incurred in the material. Volume fraction is the quantity of grain boundary intersections or the number of grains intercepted by a line drawn across the photograph divided by the true length of the line. Grid points that appear to fall on the boundary can be counted as one-half, triple point intersections are counted as one and one-half or two.

Then time was spent working with Carl Lombard to repeat his volume fraction tests he had done to see if the results could be repeated. The success repeating his results that proved that I could "read" volume fraction with accuracy and consistency.

Carl Lombard showed me the metallurgy lab and instructed how to operate the Reichart-Jung FeMn 30 microscope that allowed taking pictures of the highly polished samples and then observing the microstructure. The operation of the

Reichart-Jung is similar to that of a regular microscope with the exception that Polaroid or 35mm. film can be used to take pictures at different magnifications so different features of a material can be studied. The Polaroid film is a fast developing film and requires no more processing than coating the film with a substance to keep it from deteriorating. The 35mm. film requires developing, but the type of film was never used. From these pictures considerable time was spent computing volume fraction and analyzing the results.

At the end of the term a project was started that involved taking the material stability maps and showing the possible error associated with these maps. The equations for showing the error involved were written Venkot Samarathan and showed the strain and strain rate at various temperatures. The project was not finished and the work was contracted to an outside source.

I learned a great deal from my time spent at the Materials Directorate and everyone was helpful and willing to answer all my questions. I hope that I am able to go back to Wright Patterson and continue to work with the people at Wright Labs. I feel the experience was good and anyone interested in engineering and research should take advantage of the chance to learn more about the kind of work they would be doing.

Synthesis and Characterization of Polymers

Task Order No. 85
Student Support Program

Linda Wood
Wright State University

16 December 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank the following individuals with whom I have worked and who have helped me in my Task Assignment: Dr. Robert L. Crane, Dr. Tom M. Copper, Dr. Z. Tokarski, Dr. Natarajan, Dr. R. Pachter, Dr. S. Patnaik, Dr. J. Lupo and Mr. Marc Martin.

TASK ASSIGNMENT

Investigations were made into possible applications of computer software to increase lab operation effectiveness and efficiency. As a result, many programs were investigated. This knowledge is used to tutor individuals on usage and assist with questions and problems in using the software. External computer hard drives have been incorporated to improve backup efficiency and virus protection of computer systems.

Individual and small groups developed ideas that were presented during organizational meetings to develop a general research plan. The appropriate resources were obtained to investigate. Analysis results were presented in meetings. Useful elements were extracted and reported to appropriate groups.

Digitizing tablets, scanner, CD ROMs and external hard disks for computer were used in analysis as well as many different computer systems. Operation of many different pieces of hardware including; Bernoulli external hard disks; Macintosh II, Vax, Perkin Elmer, SUN computer and IBM computers were learned. Data analysis, transfers and translations were all accomplished with existing computer software and equipment.

Much was learned about research processes, computer network systems and many different computer systems. The advances made in computer systems and operations are continually becoming more important to aid the effectiveness, speed and efficiency of research. Computers are also becoming more important as research tools of molecular modeling.

In conclusion, experience that I gain from working at Wright Labs will prove invaluable to my desired career in the environmental research and development field. I also believe that my contribution to the lab is beneficial in maintaining a smooth operation.

QPA for PLD

Task Order No. 87
Student Support Program

David Tucker
Wright State University

24 April 1992

Guiding Engineer
Dr. Jim Malas
WL/MLIM

ACKNOWLEDGMENTS

I would like to thank the following for their help and support: Dr. Walter M. Griffith, Chief, Manufacturing Research Branch, Integration and Operations Division, Dr. Steven LeClair, Visiting Scientist, Dr. Jim Malas, Materials Research Engineer, Mr. Oliver Patterson, Visiting Scientist, Capt. Elizabeth Stark and Mr. Sam Laube.

I would like to express my appreciation for this student work program. I wish to thank Ms. Vogler and the staff of the SOCHE organization for giving me this opportunity and for their support of this Task.

TASK ASSIGNMENT

The PLD process is used to place a thin coating of a solid lubricating material onto the surface of an item. An example would be to coat a metal ball bearing with a low friction material such as molybdenum disulfide. A sample of the low friction material is placed in the path of a laser that vaporizes it. This vapor travels through a vacuum and is deposited on the substrate.

The problems solved in this task were to refine the existing computer program developed in the previous task and add new instruments for the computer to monitor. Since new operators would use the software, changes would be made to the user interface.

The software developed previously was split into two separate programs. This was in anticipation of the program becoming too large to fit in the memory all at one time. Refinements were made to the user interface to make it easier and faster for the operators to use. Mass spectrometer data collection was added. A second computer monitor was added to the hardware arrangement so the controls and displays had to be duplicated to show on both.

The operation can be set up and monitored from either room and new mass spectrometer data is collected. More importantly, there now exists a good framework for future development and modifications to the programs.

Qualitative Process Automation

Task Order No. 88
Student Support Program

Stephen Adams
Wright State University

31 December 1992

Guiding Engineer
Dr. James Malas
WL/MLIM

ACKNOWLEDGMENTS

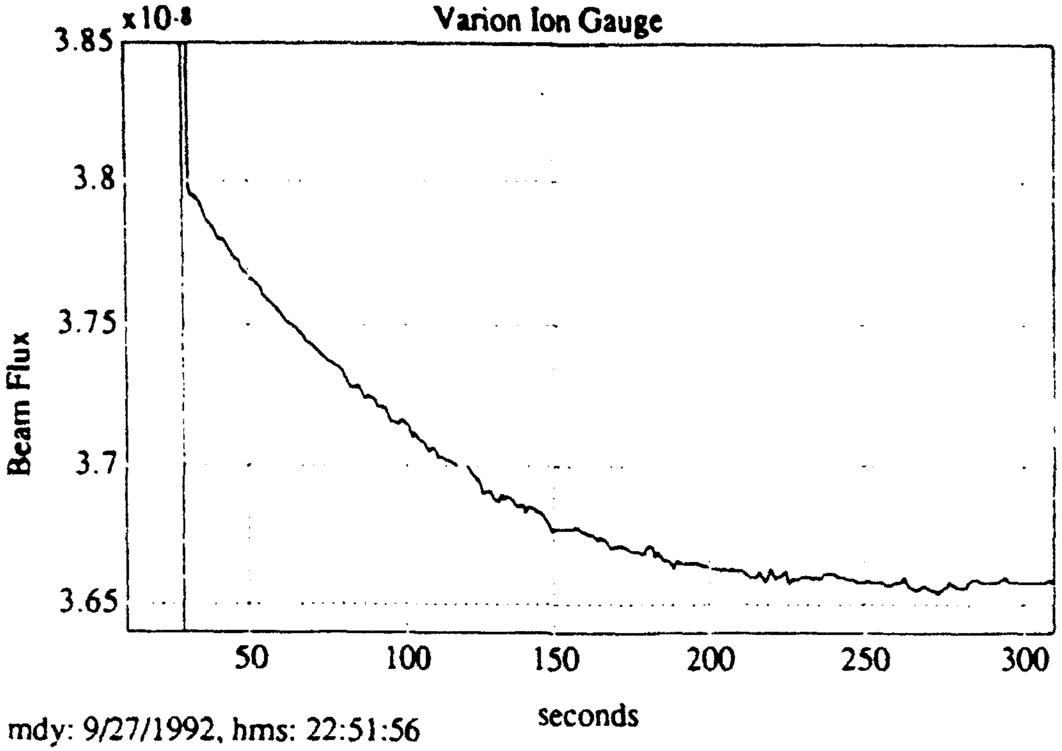
I would like to thank the following people for their help in making my second contract here very pleasant and rewarding: Dr. Walter M. Griffith, Chief, Manufacturing Research Branch, Dr. Steven LeClair, Visiting Scientist, Dr. Jim Malas, Materials Research Engineer, Mr. Oliver Patterson, Visiting Scientist, Capt. Elizabeth Stark, Mr. Jeffrey Heyob, Mr. David Tucker and Mr. Doug Moore. I also would like to thank the student support program for allowing students the opportunity to work with the Wright Laboratory of Wright Patterson Air Force Base. I have found that the people in this group are very friendly and are willing to help you out if you are having problems with your project. They also seem to go out of their way to make you a part of both the professional and the social groups that are formed at work. The exposure to technology and equipment the Air Force has to offer is an experience that I am glad to be part of and is something that has changed my out look on my future job prospects.

TASK ASSIGNMENT

Molecular Beam Epitaxy (MBE) process

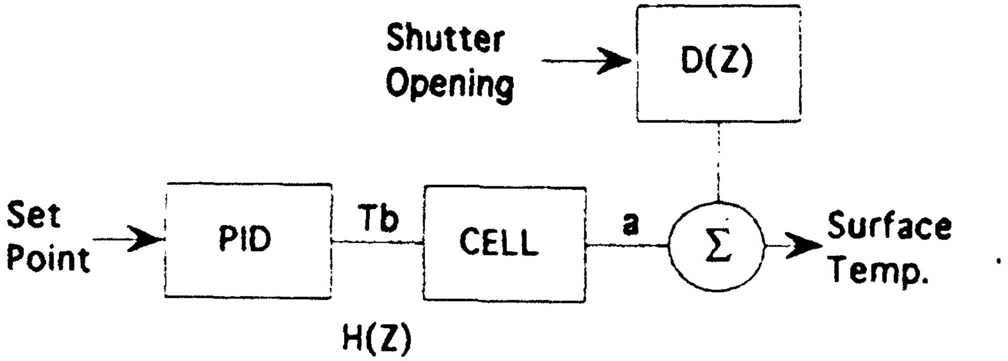
The MBE process is used to grow semiconductor thin-film on a substrate for electrical and optical applications. The process uses several materials such as: Ga, As, Al, Si and In. These materials are held in a Knudsen Cell and are heated until they are in a gaseous state. The Knudsen Cell is composed of a crucible holding the material, a shutter and a heater coil. When the shutter on the Knudsen cell is opened, a gaseous molecular beam is projected from the cell toward the substrate. When this molecular beam (e.g., flux beam) impinges on the substrate, reactions take place that result in thin film growth.

The project goal was to stabilize the flux from the cell. Under normal conditions the intensity of the flux beam tends to decrease from its initial value after shutter opening. The decrease in the flux intensity cell is caused mainly by the increased heat loss from the cell immediately after the shutter opening. A temperature controller is attached to the Knudsen cell to maintain a constant temperature within the cell. Unfortunately, this controller cannot entirely sense the increased heat loss due to shutter opening. This means that the controller cannot entirely compensate for the increased heat loss due to shutter opening. The following is an example of the droop in flux intensity after shutter opening.



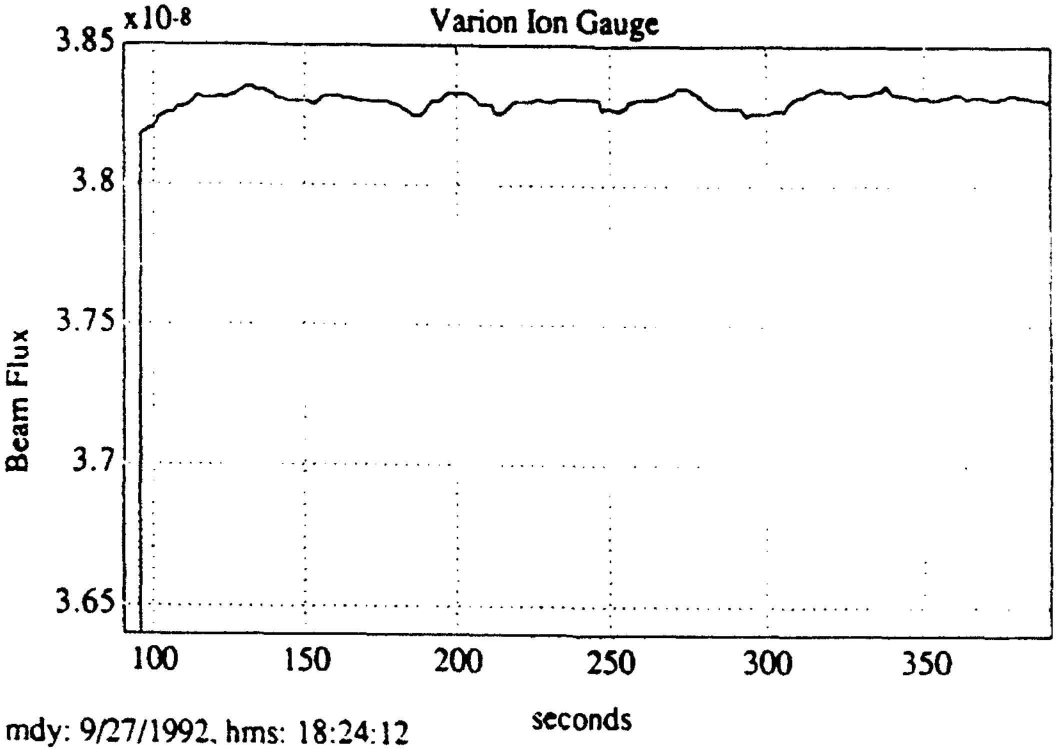
To over come this problem, feed forward flux compensation can be employed in tandem with the present temperature controller. A feed forward flux compensation signal can be summed with the normal temperature reference signal to minimize the decay of flux after shutter opening. The flux compensation signal will increase the temperature of the cell following shutter opening.

The following are new developments since my last report. A simple diagram of the model is shown below. D(z) is the equation that describes the change in the flux beam after shutter opening with the temperature of the cell held constant. The cell is the characteristic of the material that cannot be controlled directly. The PID is the temperature controller for the cell. H(z) is the equation that describes the characterists of the PID and the cell.



The information for the model is obtained by performing two tests. The first test is to set a constant temperature for the cell and then open the shutter. D(z) is then calculated using the change in the surface temperature and the constant temperature set point data. After the surface temperature has reached steady state a pulse is applied to the set point of the controller and the change in the surface temperature is recorded and H(z) is calculated. Using H(z) and D(z) it is possible to calculate the set point equation that is needed to maintain a stable surface temperature.

The results from the first test are shown below. The compensator held the Beam Flux under 0.5% change. The only problem is that the final calculated temperature was slightly greater than the actual temperature which causes the final Beam Flux to be greater then the initial value.



Characterization of Fluorinated Model Compounds

Task Order No. 89
Student Support Program

David Drake
Wright State University

31 December 1992

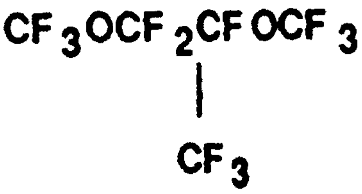
Guiding Engineer
Dr. Harvey Paige
WL/MLBT

ACKNOWLEDGMENTS

I wish to extend my sincere appreciation to Dr. Harvey Paige of the Wright Laboratory Material Directorate and Dr. Jim Liang of UDRI for the guidance and assistance they provided throughout my masters research program.

TASK ASSIGNMENT

The objective of my research is to determine the nature of impurities in a series of low molecular weight model compounds. In doing so it will be my responsibility to both obtain and interpret mass spectra and/or GC/FTIR data of samples with the following nominal structures:

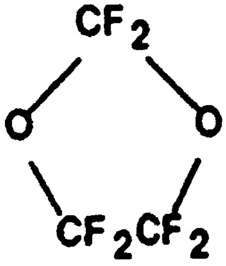


$CF_3OCF_2CF(CF_3)OCF_3$

MLO 90-721

$CF_3OCF_2OCF_3$

MLO 90-722



MLO 90-723

The numbers associated with these structures are the tracking numbers. The names of the compounds are:

MLO 90-721	Perfluoro-1,2-dimethoxypropane
MLO 90-722	Perfluorodimethoxymethane
MLO 90-723	Perfluoro-1,3-dioxolane

These compounds, which were synthesized by direct fluorination, were intended for studies of surface interactions occurring between the fluoroethers and various metal surfaces. These surface experiments are intended to give some insight into the nature of interactions between these surfaces and model fluids consisting of molecules with similar structures but of higher molecular weight. If impurities are present in the small model compounds, they may interact differently with the surface and, for this reason, their identities need to be known.

To detect the presence of impurities the following instrumentation was utilized: Hewlett Packard Gas Chromatograph/Fourier Transform Infrared Spectrometer (GC/FTIR) with sub-ambient capabilities which is necessary to obtain the separation needed for analysis and the Varian Gas Chromatograph/Mass Spectrometer (GC/MS) both Electron Impact (EI) and Chemical Ionization (CI) capability. It is preferable to use GC/MS with CI capability since with EI fragmentation occurs resulting in the absence of a molecular iron.

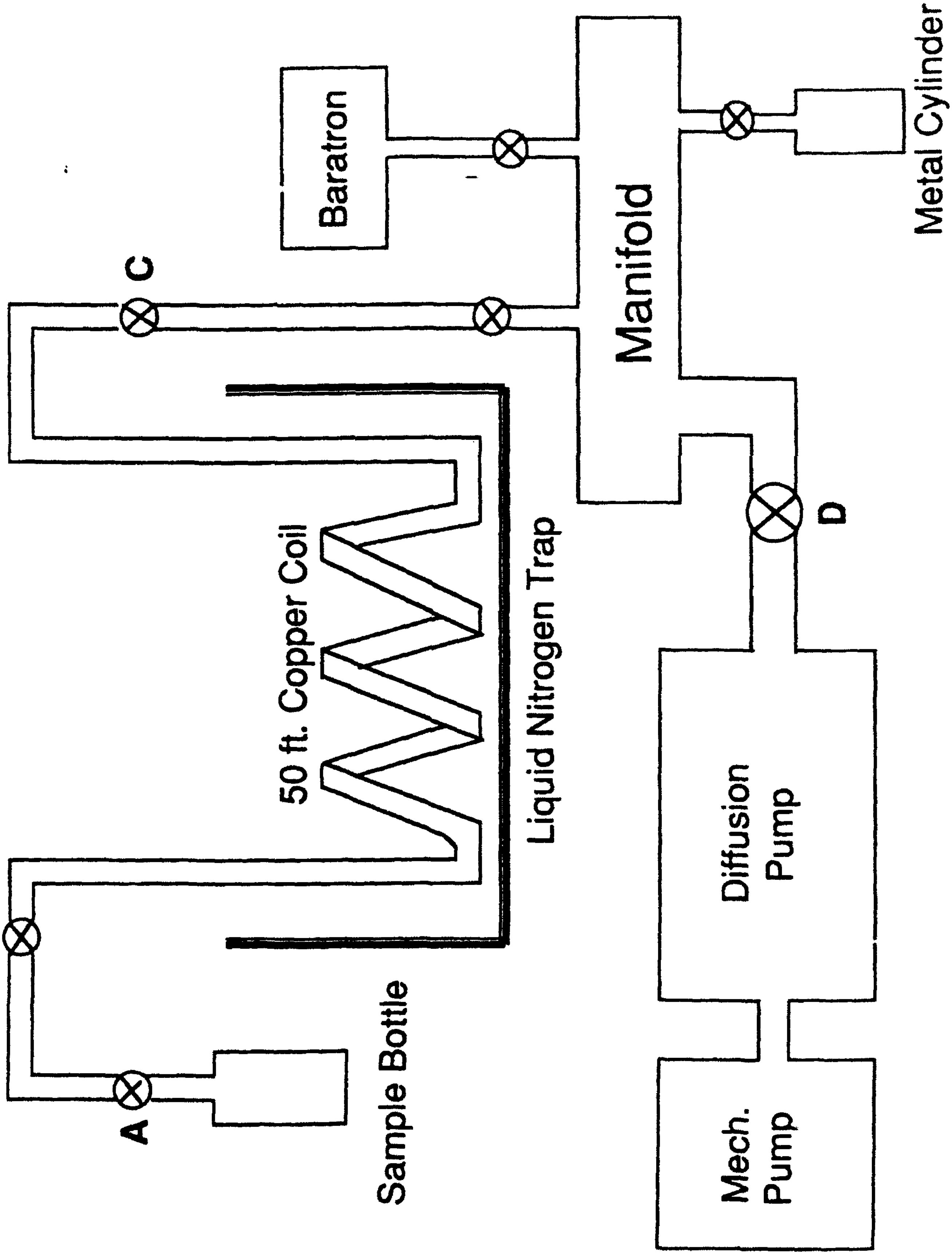
The separation of the model compounds was preformed using the apparatus shown in diagram #1. This simple setup consists of a copper coil in which the compounds travel. It is when this coil is chilled with liquid Nitrogen that the separation occurs allowing for trapping of the separate components. To aid in this trapping the vacuum manifold is utilized. Once in the manifold, cold trap distillation is used to transfer the purified sample to a sample bottle.

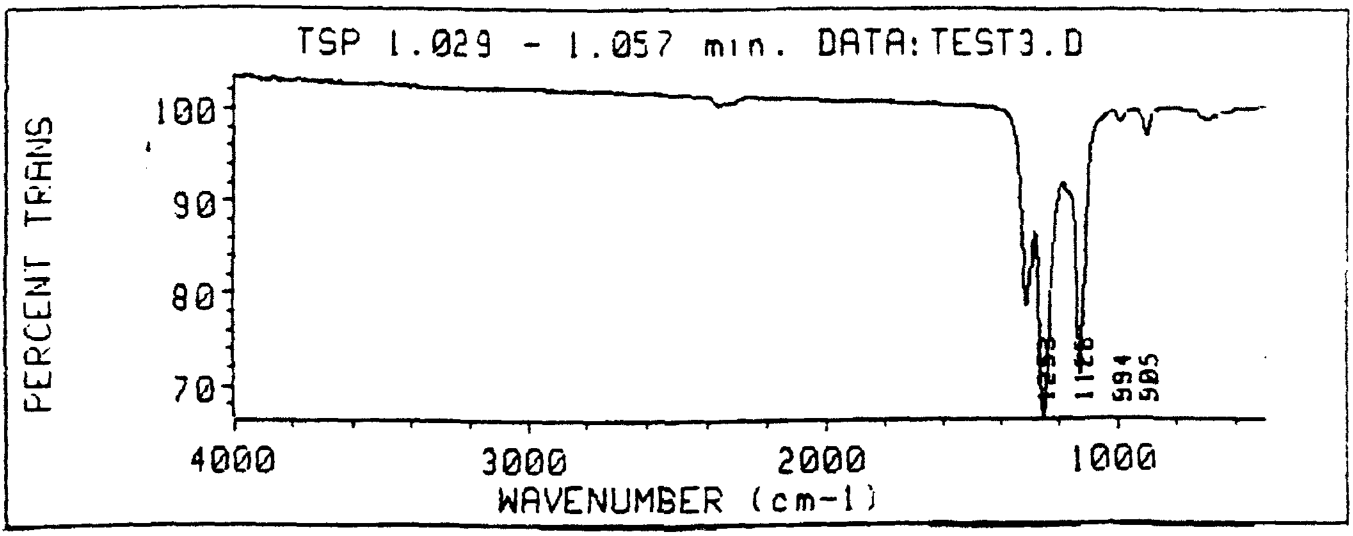
At this point the purity of the separated sample is tested using GC/FTIR. Although this technique is not an extremely sensitive technique as far as quantitation is concerned it will be sensitive enough to determine if separation was complete. If at this point it is found that the separation was not satisfactory then further separation will be performed as described above.

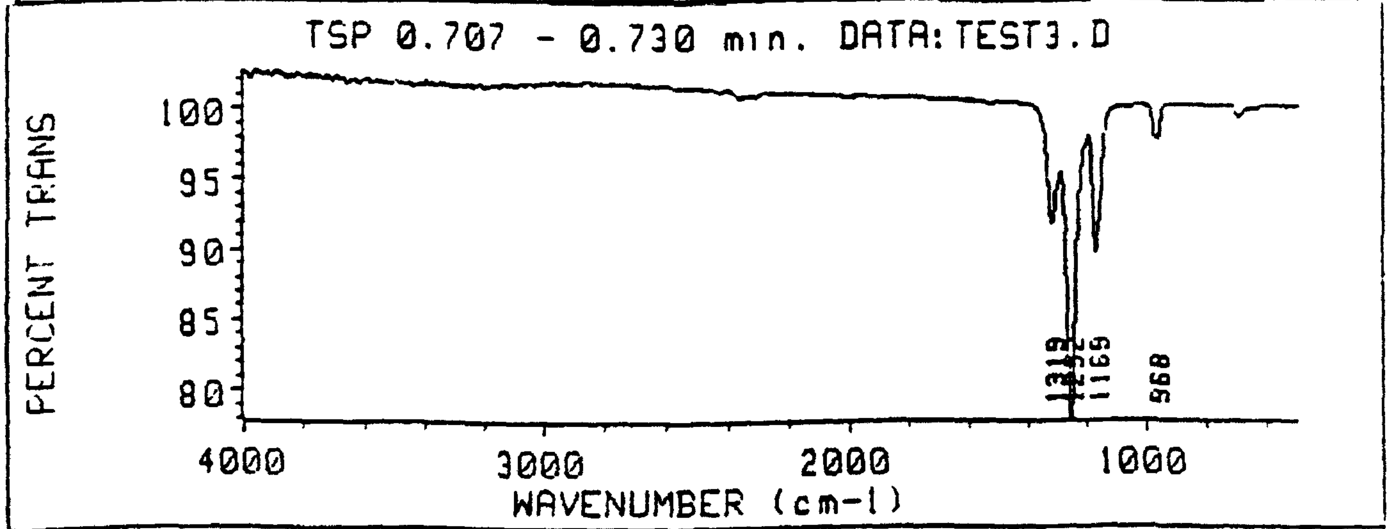
Once separation is complete, the Characterization of the Fluorinated Model Compounds can be done. To perform this characterization a Mass Spectrometer is going to be employed from which structural information will be obtained. There are several techniques which a mass spectrometer can use to obtain data but, for our purpose we chose to use only two of those, Electron Impact (EI) and Chemical Ionization (CI). With these two techniques which compliment each other some very useful structural information should be obtained.

The separation of the model compounds has been carried out with some success. An example of this is provided in diagram #2 in which the spectrum shows definite separation of the linear compound Perfluorodimethoxymethane.

To date, only the EI technique has been used for characterization producing excessive fragmentation which is shown in diagram #3. Due to this fragmentation, structural information is not very useful making it necessary to use the CI technique. Since, at this time CI capability is just becoming available, it will be necessary to continue this project to obtain any definite structural information.


Sample Bottle
A
B
50 ft. Copper Coil
Liquid Nitrogen Trap
C
Baratron
Manifold
Metal Cylinder
D
Diffusion Pump
Mech. Pump


TSP 1.029 - 1.057 min. DATA:TEST3.D
PERCENT TRANS
100
90
80
70
1253
1126
994
905
4000
3000
2000
1000
WAVENUMBER (cm-1)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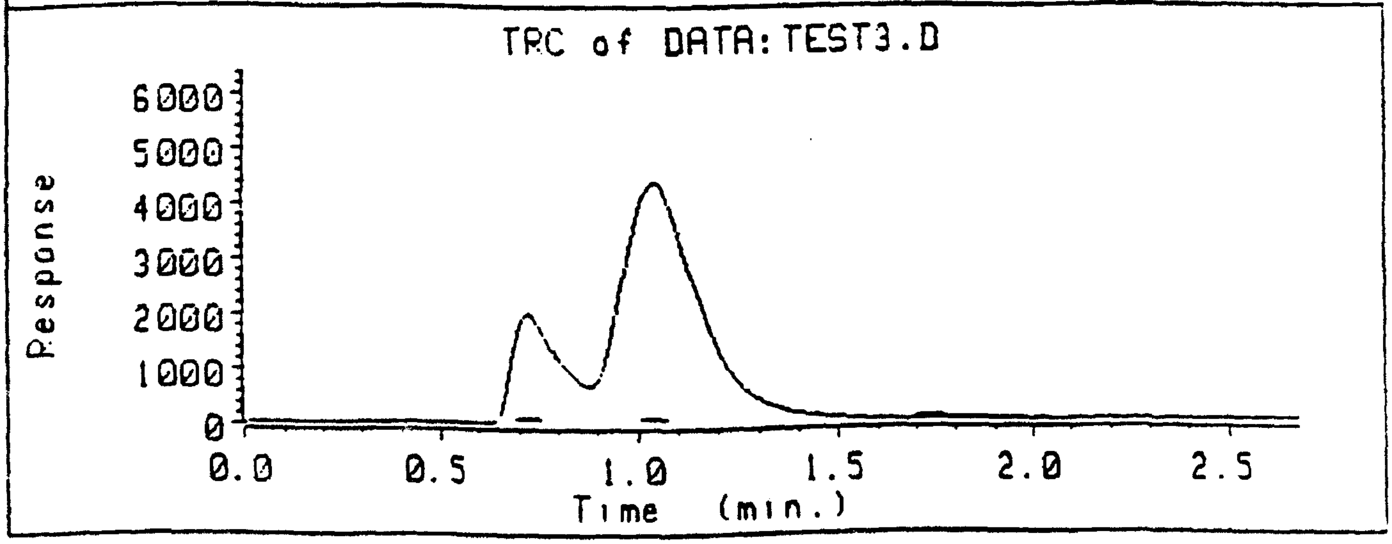
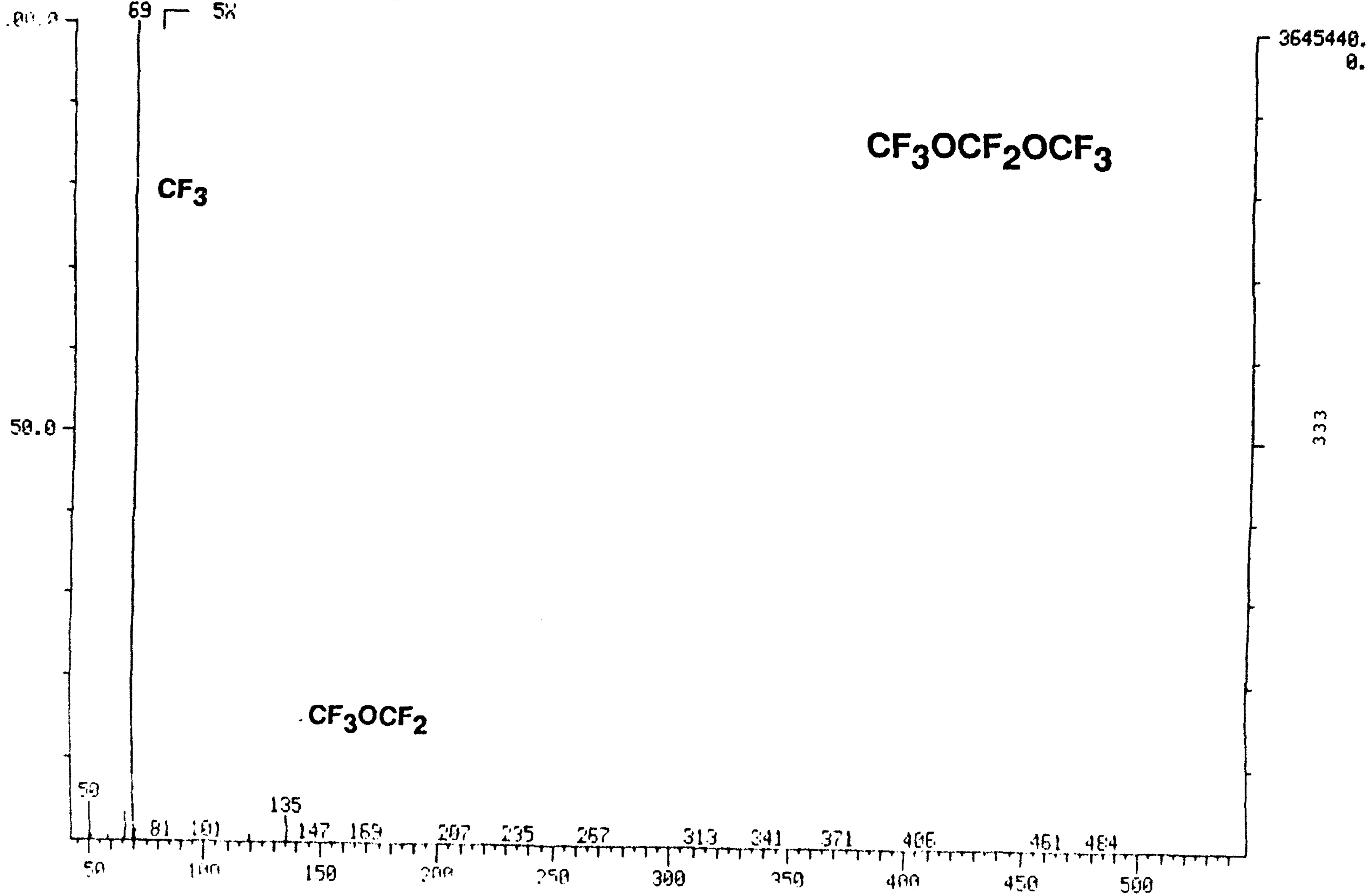
PERCENT TRANS
100
95
90
85
80
1319
1252
1165
968
4000
3000
2000
1000
WAVENUMBER (cm-1)


TRC of DATA:TEST3.D
Response
6000
5000
4000
3000
2000
1000
0
0.0
0.5
1.0
1.5
2.0
2.5
Time (min.)

MASS SPECTRUM
02/17/92 16:28:00 + 1:26
SAMPLE: BOTTLE A I
CONDS.: 40-300 SPLIT
TEMP: 40 DEG. C
#134 TO #142 SUMMED

DA[illegible] 021792RUN4 #[illegible]
CALI: 021792CAL1 #3

BASE M/E: 69
RIC: 4055040.



Graphics Support For Computations On Intermetallic Compounds

Task Order No. 90
Student Support Program

Carlos Hernandez
Wright State University

31 December 1992

Guiding Engineer
Dr. Dennis Dimiduk
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank the whole group for their support and camaraderie, which made the Task, if not easy, at least bearable. The office was one of the most friendly and pleasurable environments I have ever worked in. Special thanks to Mr. Chris Woodward for his support and expertise. I also would like to thank the people at SOCHE not only for their wonderful program, that allows students like me to gain hands on experience prior to graduation, but also for their service and help provided along the way.

TASK ASSIGNMENT

A complete graphics package was developed during the Task to display the results from the analysis and simulation of selected intermetallic compounds. In addition, several scientific programs that deal with solving and manipulating data related to research on intermetallic compounds have been initiated. The maintenance and support of the computer system was also accomplished during the task.

The graphic package developed during the Task grew twice fold over the course of the Task and has the potential to grow even further. It has an interactive interface capability as well as an interactive capability with other programs. In addition a user's guide and reference manual were developed to accompany the package. The package has the capability to display from the smallest graphic primitive, to a full scale graphic of the complete material structure. This includes the ability to display the material structure from a molecular point of view to a display of the material structure as a whole. With this package the user can analyze dislocations, stress related to materials between atoms, as well as other physical applications such as density of states etc.

The package was developed to be general in nature to provide maximum flexibility in its utilization. The necessary capabilities were built into the software by breaking it down into small pieces. General functions with changeable default settings were developed to do specific tasks. The default settings allow even the most inexperienced user to use the software package. The more experienced user has the capability to change the default settings and tailor the software to their specific needs. Every routine was written to provide the user with the ability of allowing the package to take care of everything, or let the user take full control. An attempt was made to satisfy everyone's wishes. In addition, several routines were developed that add options to provide the user with increased flexibility in the methods used to accomplish specific tasks. For instance, when plotting a function the user may want to fit a simpler curve through it or it may require a b-spline periodic/non-periodic, or even a bezier curve. Support was provided for graphical display of contour maps with several options. In fact the only restrictions of its capabilities are the restrictions of the user's imagination. The software provides the postscript output of all the graphics built in, which is currently being expanded to support the Adobe illustrator for use with the Apple system. A full set of text manipulation capabilities has also been built into the package.

The package has been tested to date with several applications, such as density of states, strain tensor, arrows plot, contours, geometric graphics, and text all with satisfactory results. The package was developed on a Sun Mycrosystem Sparc Station 1 with 16 Mb of memory, the open look X-Window System and the UNIX operating system. The software was fully implemented using the C programming language.

The open look X-window system, allows the user the capability of opening several windows at once to visualize the task from several different perspectives, or to analyze the same task from two different sets of data.

At the start of the Task a great deal of time was spent researching and reading technical manuals to become familiar with the programs available, their use and capabilities, to determine if they could be used to accomplish the task. Most of the manuals were extremely technical and not user friendly. This prompted the development of a user's guide and reference manual that was easy to understand and use, to avoid having someone else spend time trying to figure out what the software capabilities were. In doing so, a utilization example for every function call is provided to guarantee that the user does not have to run in circles trying to find a concrete example.

The task allowed the researcher to fine tune their programming skills and put their scholastic knowledge to practical application. Many frustrations were experienced along the way such as trying to define and tie down user requirements. The researcher was exposed to information on materials and scientific mathematical approaches not previously exposed to in a scholastic environment. The researcher gained hands on knowledge and experience with the X-Window library and the UNIX operating system and reviewed Fortran programming that I had not used prior to this task for at least eight years. It was necessary to maintain old code and write new code in Fortran because the group had experience with it and a lack of experience with any other language. The researcher was also exposed for the first time to the postscript language due to the need to have graphics results printed using the postscript capabilities.

The entire work experience was pleasurable and rewarding for the researcher. The experience and knowledge gained from working on the task were very worthwhile to the researcher and hopefully the contribution from the researcher was beneficial to the government.

Residual Stress Testing in Metal Matrix Composites by the Fiber-Extension Test

Task Order No. 91
Student Support Program

Andrew Johnson
University of Cincinnati

31 December 1992

Guiding Engineer
Dr.Dan Miracle
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank the following individuals with whom I have worked: Dr. Sion Pickard, Dr. Dan Miracle, Mr. Steve Russ, Mr. Bob Lewis, Mr. Eric Fletcher, Mr. Michael Scott, and Mr. Mark Dodd.

TASK ASSIGNMENTS

Residual stress is an important component in the study of metal matrix composites. Residual stresses remain in the material after cooling down from the processing temperature. Stresses are normally induced by the application of a load to a material. Residual stresses remain in a material after the load has been removed. In the case of metal matrix composites of MMCs, the residual stresses are created by a mismatch in coefficient of thermal expansion or CTE. Titanium aluminides are the metals used as the matrix in the composites studied. Silicon carbide, SCS-6 fiber, is the material used as the fiber reinforcements in the composite. Titanium aluminides have a CTE of 12×10^{-6} cm/cm*Celsius and silicon carbide fibers have a CTE of 5.3×10^{-6} cm/cm*Celcsius. Titanium aluminides have a larger coefficient of expansion than silicon carbide. This situation creates a problem during the cooling of a processed MMCs.

The matrix contracts more than the fiber during the cooling process. This places the fiber in compression and the matrix in tension. The residual stresses are created in MMCs by this situation. The magnitude of the residual stresses is not known. MMCs were developed in the past two decades. Previous testing techniques could not be applied to MMCs due to the unique geometry of the residual stresses in MMCs.

The residual stresses present in MCCs have been divided into three components along or centering at the fiber: the axial stress (σ_z), the radial stress (σ_r), and hoop stress (σ_ϕ). This particular technique, the fiber-extension test, measures the axial strain present in the fiber. By removing the matrix from the MMC, the fiber will push-out to a certain distance due to the axial stress. This characteristic of the residual stresses formed the basis for this residual stress technique. Through measurement of the residual stresses, premature failure in metal matrix composites can be avoided.

This research technique is composed of three stages: electropolishing, measurement, and analysis. A sample of a titanium aluminide composite was sectioned into piece 0.75 cm in length and 1.0 cm in width. The composite was mounted in a Triple-X mount. A Triple-X mount consists of alternating layers Konductomet and epomet. The sample was mounted with fibers perpendicular to the surface. This sample was polished to 0.05μ finish. The mounted sample was lacquered except for a central region that was electropolished. The electrolyte used for the electropolishing was sulfuric acid/ methanol solution. This sample was electropolished for eight hours to remove sufficient amounts of the matrix from the composite.

The lacquer was removed from the sample. The sample was cleaned in an IM etchant (45 ml phosphoric acid, 45 ml distilled water, 30 ml hydrofluoric acid, 15 ml nitric acid) for 30 minutes to remove the oxide layer present in the electropolished region. The etch depth for each fiber was measured optically and recorded. The etched region was photographed across the length of the etched area. The push-out distance of each fiber was measured by use of a Dektak profileometer. The Dektak reported any variations in the surface sample along a single row of fibers. The etched area was filled with a carbon paint to reduce the difference in height the Dektak had to measure. The Dektak has a 60μ surface contrast limit. The carbon paint was removed by ultrasonically cleaning the sample in an acetone solution. Push-out distance was measured from the surface profile printout (Ex. Figure 1). The unetched region of the sample provided the starting point for the measurement of the push-out distance.

The push-out distance is plotted versus the etch depth for each fiber in a row. This plot was fitted to a zero to produce a linear plot. The slope of this linear plot was the residual axial strain present in the fiber. This technique was repeated on a variety of MCCs. A representative plot has been included (Figure 2). Each row is represented by different data markers.

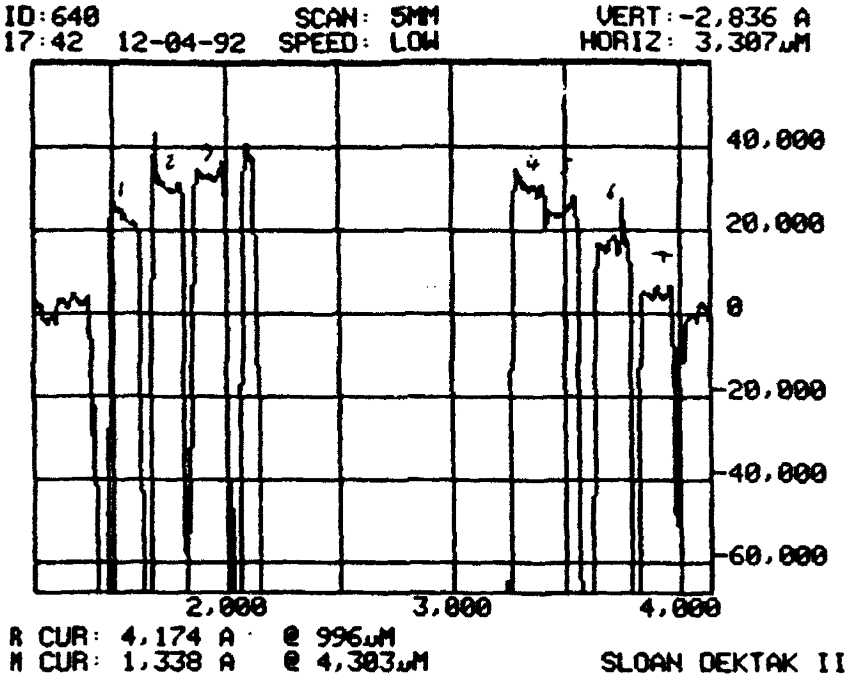
The residual axial strain measurements that were produced are converted into residual stress measurements. The stress was calculated using the formula for the perfectly elastic case:

$$\sigma_z = (E\epsilon_z)/(1- u)$$

E--modulus SiC fiber = $5.3\text{x}10^{-6}$ cm/cm* Celsius
ϵ_z--residual axial strain
u--Poisson's ratio = 0.3

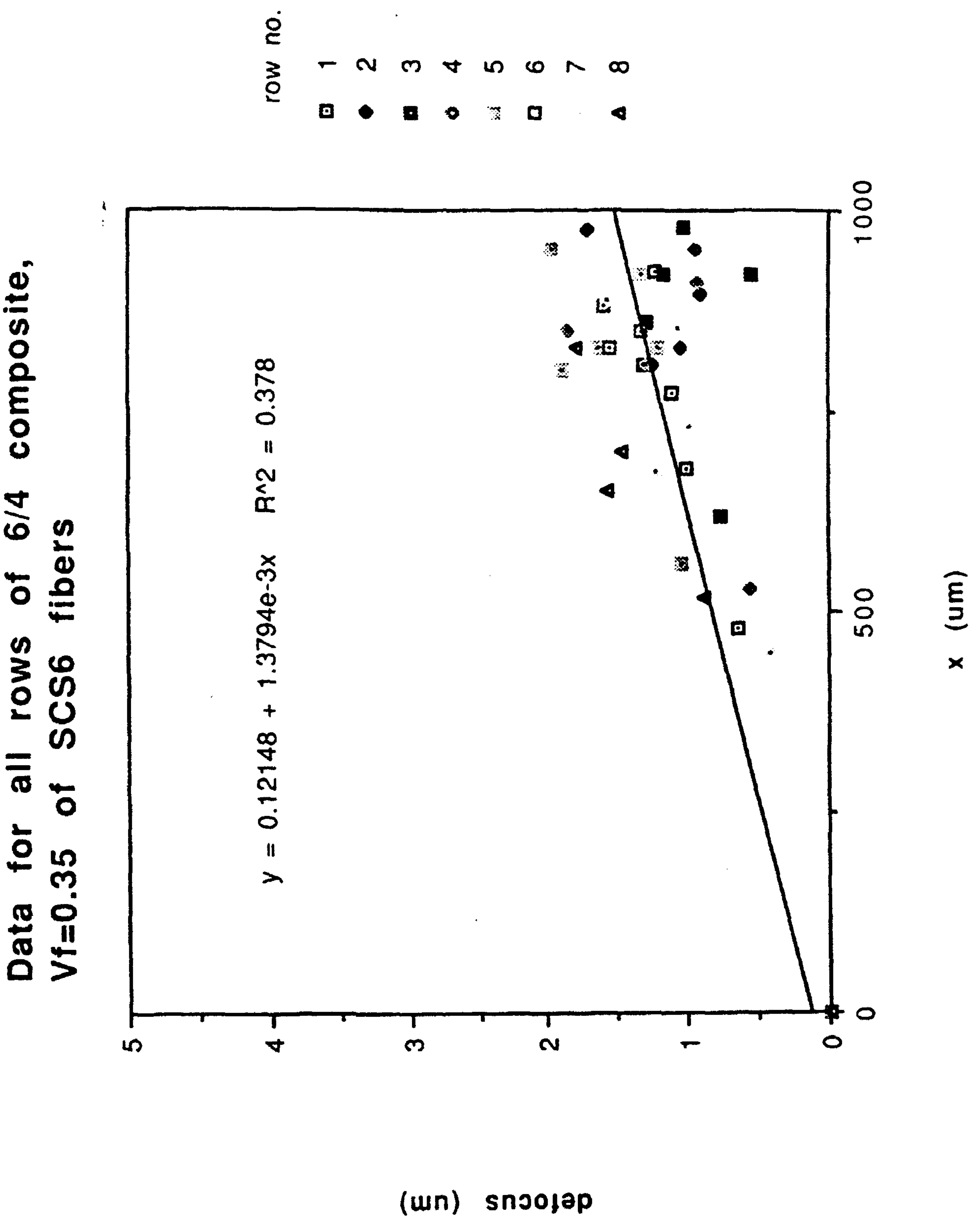
At the present the validity of this testing technique has not yet been confirmed. A variety of tests is presently being planned to confirm the accuracy of this test. X-ray diffraction is the principle confirming test. If the X-ray diffraction measurements are reasonably close to the fiber-extension test can be completed in three days. This technique has many possible applications. The fiber-extension test can determine any variations in residual stresses due to different cooling rates across a composite plate. This fact is the source of much speculation. The technique can be used with heat treatments to reduce the residual stresses present in the composites. This technique is the simplest residual stress measurement technique of ones that are being developed. This fact makes widespread use and application of this technique possible in the composites field.

Figure 1



4.

Figure 2


Data for all rows of 6/4 composite,
Vf=0.35 of SCS6 fibers
y = 0.12148 + 1.37940e-3x   R^2 = 0.378
defocus (um)
0
1
2
3
4
5
x (um)
0
500
1000
row no.
1
2
3
4
5
6
7
8

Engineering and Design Data

Task Order No. 92
Student Support Program

Brain Knapke
University of Dayton

31 December 1992

Guiding Engineer
Mr. Clayton Harmsworth
WL/MLSE

ACKNOWLEDGMENTS

I would like to thank the entire Engineering and Design Data Branch as well as the University of Dayton Research Institute technicians for their support and assistance whenever I needed it. I would especially like to thank Mr. Clay Harmsworth, Mr. Neil Ontko, Mrs. MaryAnn Phillips and Ms. Dayle Pearson for the extra time they spent with me on the ARALL-3 project.

TASK ASSIGNMENT

I have spent the year working on the ARALL-3 (aramid aluminum laminate) composite material that consisted of 6 plys of aluminum and 5 plys of Kevlar fibers. ARALL was developed by Alcoa as a material with a density comparable to that of aluminum but with much better material properties than aluminum itself, especially in the area of fatigue crack growth rate. This occurs because the high-strength Kevlar fibers inhibit the growth of a crack in the aluminum. Material property testing was necessary to verify these claims and to determine the effect of exposing the material to varying temperatures as well as high humidity. The ARALL material's proposed are in the cargo bay door of McDonnell-Douglas' cargo-transport plane, the C-17.

Tensile, compression, Rolfes compression and Iospescu shear environmental tests were performed on this material. All tests were to be performed at -65°F, room temperature, and 180°F. When the specimens were received, half the specimens had been saturated with moisture in a humidity chamber. Six specimens were tested at each temperature, humidity combination with the Kelvar fibers 0° to the longitudinal axis and with the fibers 90° to the longitudinal axis. The results of the tests were collected in the form of a load vs. strain plot, with the load being measured by a load cell attached to the testing machine, and the strain measured by either a strain gage or an extensometer.

Whenever a testing machine was not available, I taught myself AutoCAD and updated the test specimen files by redrawing them on AutoCAD, and organized them in a directory for the engineers to access them easier.

First, the ARALL specimens were to be properly cleaned on the gage surface using an airbrasive machine and Micromeasurements Conditioner A. Then the pH of the surface was neutralized with Micromeasurements Neutralizer 5. Once the surface was prepared, the Micromeasurements strain gages were applied to the tensile and Rolfes' compression specimens with M-bond 200 adhesive. In performing the tests, an Instron 4507 series mechanical testing machine was used and a Cincinnati Sub-Zero environmental chamber was used to reach the desired test conditions.

The tensile specimens were tested at a crosshead speed of 0.05 inches per minute and the data was taken in the form of two load-strain curves, one for the axial strain, the other using the lateral strain. Poisson's ratio and elastic modulus were then calculated. The strain on the tensile specimens was measured using two strain gages, one positioned parallel to the longitudinal axis and one perpendicular to the axis. The elastic modulus was calculated by the change in stress divided by the change in strain, which is the slope of the stress-strain curve. Poisson's ratio was calculated by dividing the axial strain by the lateral strain at a certain load. The units for the elastic modulus are pounds per square inch and Poisson's ratio are unitless.

Two forms of compression tests were performed, which utilized the SATEC metal compression fixture and the Rolfes' compression fixture. The strain for the SATEC fixture was measured by an MTS extensometer, and the strain for the Rolfes' fixture was measured by a strain gage. The crosshead speed used during the metal compression tests was 0.005 in/min, while the speed of the Rolfes compression tests was 0.05 in/min. To compensate for any buckling during the tests, the Rolfes' specimens had strain gages on each side of the specimen, and then the average strain was used. The compressive modulus was than calculated for both tests in the same manner that the elastic modulus was for the tensile tests.

In conclusion, the data that was collected and the values that were obtained for the modulus and Poisson's ratio were fairly precise and agreed with the expected values. As expected, the strength of the material decreased as the temperature increased or as the relative humidity increased. The data was organized and put in a database on Lotus 1-2-3. An example of the results of one of the sets of tensile tests is attached at the back of this report.

J Knapke
E Test Technician

MaryAnn Phillips
MLBE Project Engineer

ARALL-3 ENVIRONMENTAL TESTS
TENSILE TESTS
90 DEG. ORIENTATION
DRY SPECIMENS

SPECIMEN NUMBER	TEST DATE	WIDTH (INS)	DEPTH (INS)	AREA (IN**2)	LOAD (LBS)	ULTIMATE STRENGTH (PSI)
-65 F						
CAR390-1	08/04/92	0.4965	0.1115	0.0556	2,869	51,617
CAR390-7	08/05/92	0.5005	0.1115	0.0558	2,921	52,342
CAR390-13	08/05/92	0.4995	0.1120	0.0559	2,830	50,586
CAR390-19	08/06/92	0.4975	0.1120	0.0557	2,915	52,315
CAR390-26	08/06/92	0.4980	0.1120	0.0558	2,871	51,474
CAR390-32	08/06/92	0.4960	0.1110	0.0551	2,860	51,947
				S.D.	35	855
				AVG	2,878	51,714
R T =72 F						
CAR390-33	05/22/92	0.4990	0.1115	0.0556	2,721	48,905
CAR390-8	05/22/92	0.4965	0.1115	0.0554	2,681	48,429
CAR390-20	05/22/92	0.4975	0.1115	0.0555	2,709	48,836
CAR390-2	05/26/92	0.4965	0.1120	0.0556	2,743	49,327
CAR390-27	05/26/92	0.5015	0.1115	0.0559	2,744	49,073
CAR390-14	05/26/92	0.4990	0.1115	0.0556	2,736	49,175
				S.D.	24	314
				AVG.	2,722	48,957
+160 F						
CAR390-3	08/13/92	0.5000	0.1125	0.0563	2,392	42,524
CAR390-9	08/13/92	0.5000	0.1115	0.0558	2,468	44,269
CAR390-15	08/14/92	0.4950	0.1115	0.0552	2,386	43,231
CAR390-21	08/14/92	0.4940	0.1115	0.0551	2,444	44,371
CAR390-28	08/14/92	0.4970	0.1120	0.0557	2,392	42,972
CAR390-34	08/14/92	0.4965	0.1110	0.0553	2,403	43,428
				S.D	34	726
				AVG.	2,414	43,466

CROSSHEAD SPEED= 0.05 in/min.

CC: Mary Ann Phillips, Dayle Y Pearson

SPECIMEN NUMBER	AREA (IN**2)	LOAD (LBS)	AXIAL STRAIN 1 (INS/IN)	ELASTIC MODULUS.E (*1 E6 PSI)	AXIAL STRAIN 2 (INS/IN)	TRANSVERSE STRAIN (INS/IN)	POISSON'S RATIO
-65 F							
CAR390-1	0.0556	3,000	0.0080	6.74E+06	0.0040	0.00095	0.24
CAR390-7	0.0558	3,000	0.0080	6.72E+06	0.0032	0.00075	0.23
CAR390-13	0.0559	3,000	0.0079	6.79E+06	0.0040	0.00095	0.24
CAR390-19	0.0557	3,000	0.0079	6.82E+06	0.0026	0.00065	0.25
CAR390-26	0.0558	3,000	0.0078	6.89E+06	0.0026	0.00060	0.23
CAR390-32	0.0551	2,800	0.0074	6.87E+06	0.0040	0.00095	0.24
	AVG.	2,967	0.0078	6.81E+06	0.0034	0.00081	0.24
			S.D.	0.068E+06	0.00069	0.00016	0.008
R T =72 F							
CAR390-33	0.0556	2,500	0.0070	6.42E+06	0.0014	0.00040	0.29
CAR390-8	0.0554	2,500	0.0068	6.64E+06	0.0020	0.00048	0.24
CAR390-20	0.0555	2,500	0.0070	6.44E+06	0.0014	0.00035	0.25
CAR390-2	0.0556	2,500	0.0069	6.52E+06	0.0020	0.00047	0.24
CAR390-27	0.0559	2,250	0.0060	6.71E+06	0.0020	0.00046	0.24
CAR390-14	0.0556	2,500	0.0068	6.61E+06	0.0014	0.00032	0.23
	AVG	2,458	0.0068	6.56E+06	0.0017	0.00042	0.25
			S.D	0.116E+06	0.0003	0.00007	0.02
+160 F							
CAR390-3	0.0563	3,000	0.0090	5.92E+06	0.0030	0.00075	0.25
CAR390-9	0.0558	3,000	0.0092	5.84E+06	0.0040	0.00095	0.24
CAR390-15	0.0552	2,000	0.0066	5.49E+06	0.0034	0.00085	0.25
CAR390-21	0.0551	2,000	0.0060	6.05E+06	0.0030	0.00075	0.25
CAR390-28	0.0551	3,000	0.0090	6.05E+06	0.0030	0.00075	0.25
CAR390-34	0.0557	3,000	0.0090	5.98E+06	0.0030	0.00075	0.25
	AVG	2,667	0.0081	5.89E+06	0.0032	0.00080	0.25
			S.D	0.211E+06	0.0004	0.00008	0.004

Fatigue of Ceramic Composites

Task Order No. 93
Student Support Program

Jennifer Finch
Wright State University

31 December 1992

Guiding Engineer
Capt. John Pernot

ACKNOWLEDGMENTS

First, special thanks should be given to the two guiding engineers of this Task, Capt. John Pernot and Larry Zawada. They have they been mentors in all aspects of the job as well as in related school work. They offered professional opinions, took time to explain matters thoroughly, and gave advice on related homework problems. Next, the author would like to extend gratitude to every professional within the lab for lending me their time, patience and expertise. Lastly, to acknowledge the Student Support Program, thank-you for this opportunity.

TASK ASSIGNMENT

The objective of this Task was to analyze the fatigue characteristics of certain Ceramic Matrix Composite (CMC) materials. Two types of CMC materials were examined, SiC/SiC (a silicon carbide reinforced silicon carbide system), and C/SiC (a carbon reinforced silicon carbide system). The fatigue properties particular to these CMCs were assessed by subjecting test specimens to the influence of high-cycle loading.

The investigation of the fatigue life of Ceramic Matrix Composites (CMCs) is of interest to the researchers involved in this Task, since these materials show great potential for high-temperature aerospace applications. Programs such as the National Aero-Space Plane (NASP), Integrated High Performance Turbine Engine Technology (IHPTET), and the Advanced Tactical Fighter (ATF), are currently considering this class of materials to replace and supplement metallic materials in aerospace applications. Such applications as gas-turbine engines and hypersonic airframes require materials with high-temperature capabilities, as well as increased strength. The investigation of CMCs has shown them to be low density, high strength materials with high moduli and the ability to withstand increasing temperatures. These qualities found in CMCs make them an attractive alternative for those and other aerospace applications.

The fatigue characteristics of the two CMCs under investigation, were obtained from a series of tension-tension and tension-compression fatigue tests. Initial research of the subject was performed in the Wright Laboratory Technical Library located at the job site, before testing commenced. After the SiC/SiC and C/SiC specimens were logged into a laboratory notebook, they were prepared for testing by the following steps: 1) ultrasonic scanning to detect any defects, 2) measuring the length, width, and thickness, and 3) grinding the surface. Monotonic tension and compression tests were performed to determine the stress-strain behavior of both materials. From these data, maximum stress values were selected for use with the room-temperature, high-cycle fatigue tests, which then were performed on both types of materials. Approximately fifteen fatigue tests were run on each type of material with varied load ranges and cycle frequencies.

A horizontal servohydraulic test machine, in conjunction with load and displacement transducers, were utilized as the testing apparatus (see figure 1). The data obtained by the test machine and the transducers were recorded and stored on the connected IBM computer system. The data were then copied from the computer to floppy disks and formatted on a Macintosh computer. The data associated with fatigue properties were arranged and plotted so the researching engineers of this Task could analyze the results (see figure 2).

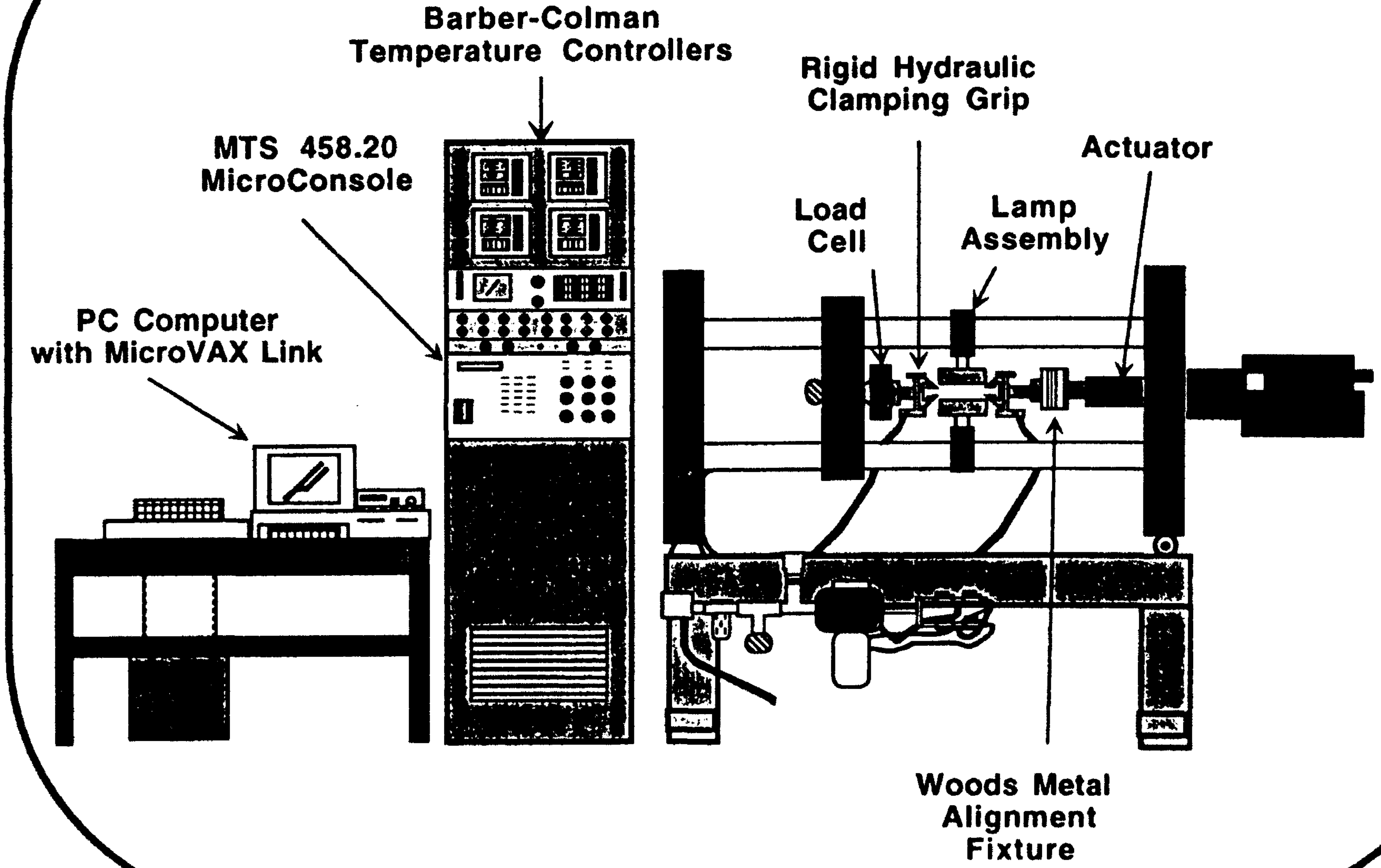
The investigation of SiC/SiC and C/SiC fiber-reinforced systems revealed significant aspects of behavior. The stress vs. cycle plots of SiC/SiC loaded at different ratios were nearly identical. That is, for a given maximum stress value, the cycles to failure in tension-tension fatigue is similar to the cycles to failure for a tension-compression test. Results from the SiC/SiC test matrices show a tendency for this material to fail in a tensile mode when cycled between tensile and compressive stresses. This may be due to the ratio of the maximum stress in tension to that in compression, which is one to three.

The fatigue limit of SiC/SiC is quite inferior to its theoretical ultimate tensile strength (UTS) as compared to the C/SiC. SiC/SiC fails consistently when loading at considerably low loads even though its UTS is higher. On the other hand, the C/SiC system was able to withstand cyclic loading at higher stresses with respect to its UTS. The fatigue limit of C/SiC in nearly 96% of the UTS.

Both the SiC/SiC and the C/SiC materials indicated that their residual strength was increased when initially fatigued at low loads. Each system was fatigued to one million cycles at increasing intervals of load, and then compared to the residual strengths of the systems after being fatigued only at high stresses. It was shown that residual strength is increased when fatigued a low loads.

As was the objective of this Task, the fatigue characteristics of some advanced Ceramic Matrix Composites were investigated. Resulting from this study, certain aspects of the fatigue properties of the fiber reinforced systems, SiC/SiC and C/SiC, were discovered. From these results, the following conclusions were made on the author's part. When cycled between tension and compression, the maximum stress in tension dictates the failure point of SiC/SiC. In addition, the mode of failure under such conditions is tensile. The tested C/SiC system is unique in withstanding fatigue at high stresses as compared with the ultimate tensile strength. Finally, the residual strength of both the SiC/SiC and C/SiC reinforced composites is increased when they are initially fatigued at low loads.

CMC Fatigue Test Station



Barber-Colman
Temperature Controllers

MTS 458.20
MicroConsole

PC Computer
with MicroVAX Link

Rigid Hydraulic
Clamping Grip

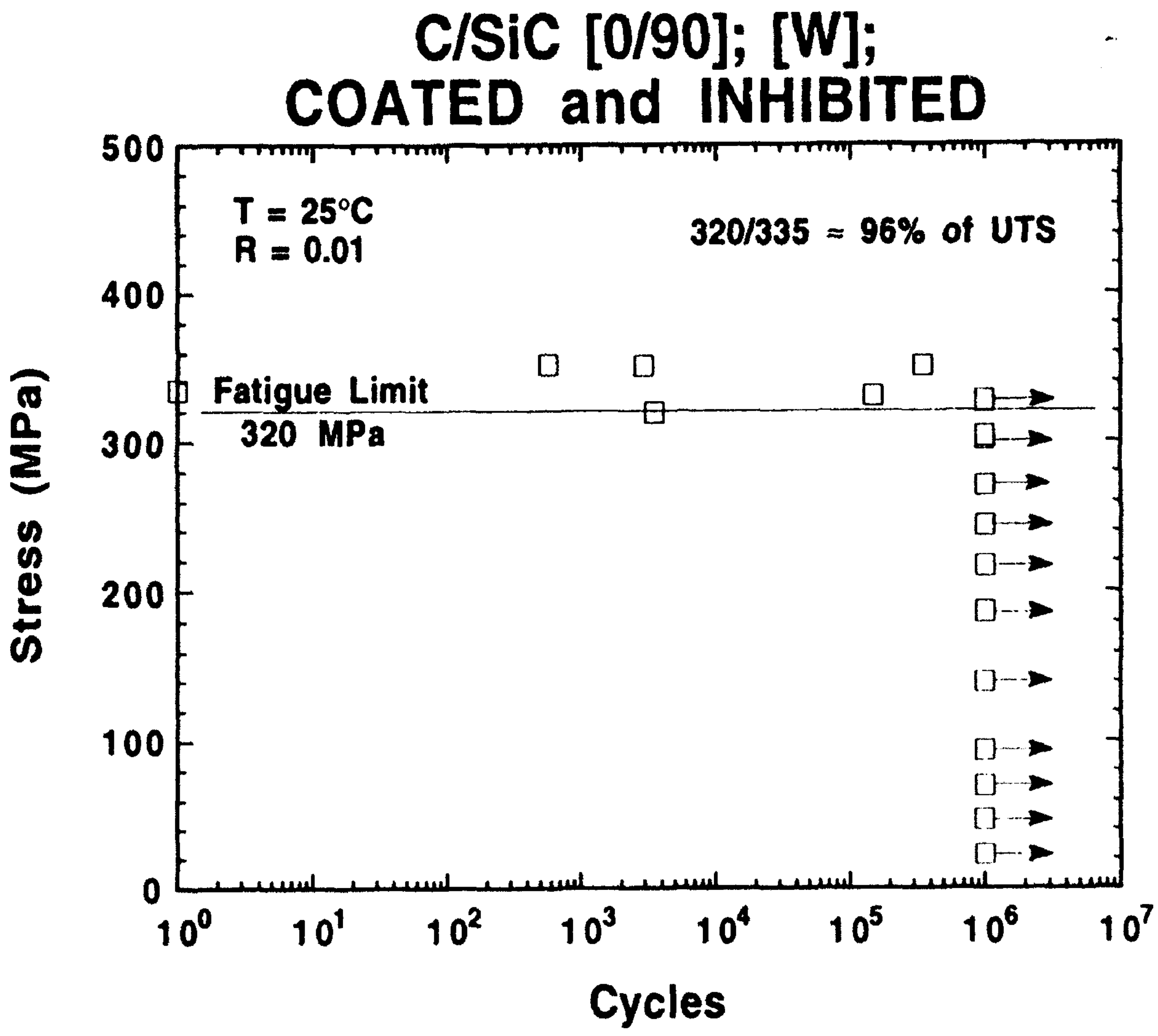
Actuator

Load
Cell

Lamp
Assembly

Woods Metal
Alignment
Fixture

(FIG 1)


C/SiC [0/90]; [W];
COATED and INHIBITED
T = 25°C
R = 0.01
320/335 ≈ 96% of UTS
Fatigue Limit
320 MPa
Stress (MPa)
500
400
300
200
100
0
Cycles
10^0
10^1
10^2
10^3
10^4
10^5
10^6
10^7

(FIG 2)

Structural Ceramic Composite Processing

Task Order No. 94
Student Support Program

William Davis
Wright State University

31 December 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank the ceramic composite group. I would also like to thank Dr. Ronald Kerans, Ms. Kristen Keller, Sankar and Mr. Charles Cooke who have guided me through the task in order to further enhance my knowledge of chemistry.

TASK ASSIGNMENT

The task objective of this assignment involves basic Material Science research at Wright Patterson Air Force Base with the Ceramic Composite group under the immediate supervision of Dr. Ronald Kerans. As a student lab aid the responsibilities of this assignment entail the production of YAG (Yttrium Aluminum Garnet) powder which is used for the development of high temperature composites used for engine parts. It involves the development of new hot pressing techniques, rotovaporation, pressure filtration, spray drying, chemical vapor deposition, modification and several other processing procedures or methods that produce dense ceramic matrix composites with very small flaw sizes. The responsibilities of the student contractor in correlation to this objective include assisting with the fabrication of these dense ceramic matrix composites. Duties which involve laying up tapes, impregnation with matrix and hot pressing in graphite die and also assisting with metallography for characterization of the composites. Other duties involve performing filtrations, rotovaporations, pressure filtrations, distillations, and hydrolysis, steps that are essential to the production process of Yttrium Aluminum Garnet powder.

Even though, the task objective and responsibilities consist mainly of techniques and procedures stated above, I have been primarily responsible for the development of YAG powder. The responsibilities with this development involve several organic, quantitative and qualitative chemistry steps which leads to the trial production of what is believed to be a course product of the YAG powder. The procedures used are very effective, however, tedious due to the physical and unpredictable nature of YAG.

I am responsible for the synthesis of solutions known as metal alkoxides which are the initial steps in the production. These metal alkoxides consists of Aluminum and Yttrium metals completely reacted or refluxed for many hours in separate flasks of Isopropyl alcohol and a catalyst of Mercuric Chloride. Once these reactions are successfully completed, provided that no traces of metal are evident, a filtration procedure follows shortly thereafter. This procedure only involves the filtration of the completely reacted Yttrium alkoxide solution. The Aluminum alkoxide solution is excluded because it is less concentrated and does not produce a sediment in contrast to the solution of Yttrium. Shortly after the filtration procedure, the filtered Yttrium Alkoxide solution is canulated or transferred and mixed with the other alkoxide solution that contains Aluminum. This final solution is known as a mixed alkoxide solution which is then heated or refluxed for a half hour in order to form a more stable product for hydrolysis (addition of water). After the solution has been hyrolyzed, another filtration procedure occurs which involves the washing of the solution with acetone and deionized water several times. The final solution is then calcined or heated in a programmable oven at a certain temperature range which leads to the formation of a solid. The solid crystals that are formed are subject to a series of tests such as X-ray crystallography to determine whether or not YAG has been produced.

During this task I have gained useful experience, knowledge and exposure to the research and development of the scientific world. Even though the task was not completely chemistry, I've been able to actually see a correlation that exists between chemistry and the material world. I've also been exposed to new laboratory techniques that include Pressure filtration, Hot pressing, Metallography and Chemical vapor deposition a method known throughout the scientific world. This task has been a valuable experience even though the objective has not changed. It has sharpened my laboratory skills and also further enhanced my knowledge of chemistry applied in the material world.

Structural Ceramic Composite Analysis

Task Order No. 95
Student Support Program

Kimberly Andrews
Wright State University

31 December 1992

Guiding Engineer
Dr. Ron Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to express my appreciation to Dr. Kerans for his guidance during this task assignment.

TASK ASSIGNMENT

Much attention has been focused on developing methods to weaken the fiber-matrix interface of a composite to increase the fracture toughness of the composite. At Wright Laboratory, one such approach has been proposed, where weakening of the interface between the fiber and matrix is accomplished by the segregation of selected dopants introduced into the composite material. If successful in this approach, ceramic matrix composites could be used as components in turbine engines.

225 ppm of each dopant was dissolved in a small amount of water and highly pure alumina powder was added. The slurry was mixed and oven dried. A polished YAG (yttrium aluminium garnet) single crystal was embedded into the doped alumina powder by hot pressing.

A thin slice of the hot pressed slug was cut and mounted on a glass slide where the sample was ground and polished. The sample was core-drilled to produce a 2mm disk that was glued to grids for interfacial support and ion-milled for transmission electron microscopy (TEM) analysis.

From TEM analysis, dopants strontium, calcium and zinc showed strong tendencies to segregate to the fiber-matrix interface. In the case of the strontium-doped material, indention tests indicated that the indentation crack grew along the interface, suggesting a weak interface between the figer and matrix. Further work was done to gain more understanding of segregation behavior, using nickel as a dopant in addition to the previously mentioned dopants. At this time, the results are being analyzed.

Structural Ceramic Composite Processing

Task Order No. 96
Student Support Program

Joy Deahl
Univeristy of Dayton

31 December 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Dr. Ronald Kerans and Dr. Randy Hay for their supervision and guidance in my research projects. My appreciation is given to Cindy Emerick for her help in preparing TEM thin foils. Thanks to Mike Scott and Eric Fletcher for their assistance in metallography. Gratitude is also extended to the entire High Temperature Ceramics Group of the Materials Directorate and all my other co-workers for making my research experience very memorable.

TASK ASSIGNMENT

The Task consisted of two major projects. The first project was the study of the kinetics of the YAP (Yttria-Alumina_Perovskite)-Alumina eutectic transformation to YAG (Yttria-Alumina-Garnet). The investigation of this solid state transformation is interesting since the reaction produces an 11% volume increase that could lead to a fiber material that would experience large compressive stresses. This is desirable in composite design. Small pieces of the YAP-Alumina eutectic were heat treated and made into TEM (transmission electron microscope) thin foils to study the effect of time and temperature on the reaction.

The second major project was that of investigating fiber/matrix interfaces in composites. Sapphire (Saphikon) fiber with various coatings was combined with a YAG matrix to produce hot pressed composite specimens. These were then heat treated and made into TEM foils for microscopy analysis. Four different systems were chosen to be studied.

The majority of time for the Task was spent preparing TEM foils. The same basic procedure is used for all samples. A sample is mounted in epoxy using the mounting press to create a one inch mount. The sample surface is then ground and polished down to a one micron finish using the mechanical polishers. The polished surface is then cleaned thoroughly and glued to a glass slide. After drying, the mount is cut with a diamond saw so there is only a thin slice remaining glued to the glass slide. The sample is now called a thin section. The thin section is ground and polished down to a thickness of less than 20 microns. The surface also should have a one micron finish. Copper grids and ovals, 3 mm. in diameter, are glued to the sample surface in the regions of interest. Heat is applied to the sample and the thin foils are removed from the glass slide making sure to remove all excess glue from the sample. The thin foils are removed from the glass slide making sure to remove all excess glue from the sample. The thin foil is now ready to be ion milled until ready for analysis in the TEM. The ion mill is operated at kV at an angle of 15° and run for several hours depending on the thickness of the sample. The goal is to obtain a great deal of thin area that can be studied in the TEM.

Approximately 30 TEM thin foils were prepared for the YAP-Alumina transformation to YAG project. The goal is to measure the YAG reaction layer thickness as a function of time and temperature to ultimately obtain the diffusion coefficient of the rate limiting species in the formation of YAG. This is done by heat treating small pieces of YAP-Alumina eutectic for known times and temperatures. Times ranged from 6 to 6000 minutes and temperatures ranged from 1200 to 1550° C. TEM thin foils were then prepared and analyzed to find where and how much reaction product is formed.

Four different systems were inspected for the composite fiber/matrix interface project. Sapphire (Spahikon) fiber with various coatings was combined with a YAG matrix to produce a 5/4 square inch composite approximately 3/16 inch thick. The overall composite was approximately 5 volume percent fiber and 95 volume percent matrix. These dimensions required a total of 32 feet of fiber in each composite. Each composite was comprised of 4 unidirectional layers of fiber that were 8 feet each. Each layer consisted of fibers cut into 80 pieces of 5/4 inch lengths and aligned parallel by hand. The matrix used was doped or undoped YAG powder. The powder was weighed out and combined alternating with the fiber layers to produce a composite with 4 fiber layers and 5 matrix layers. The composite was loaded into a 5/4 square inch die and hot pressed at 1675° C and 27.6 MPa. Four different composites were fabricated and investigated. The fiber of the first composite was Saphikon with 12 coats 30% ZrO_2/70% Al_2O_3, 1 coat Pd, 3 coats $CaO(Al_2O_3)_6$, 1 coat Pd, and 1 coat Y_2O_3. The matrix was YAG doped with 1% CaO and 1% ZrO_2. The second composite contained Saphikon fiber with 7 coats of Pd. Its matrix was undoped, pure YAG powder. The fiber of the third composite was the same as the first composite, but the matrix was changed to YAG doped with 5% CaO and 5% ZrO_2. The fourth composite contained Saphikon fibers with 3 coats Rh, 1 coat $La_2O_3(AI_2O_3)_{11}$, 3 coats Rh, 1 coat $La_2O_3(AI_2O_3)_{11}$, 3 coats Rh, 1 coat $La_2O_3(AI_2O_3)_{11}$, and 3 coats Rh. Small pieces of each composite were heat treated after the original was hot pressed. Heat treatments were performed at 1650° C, 1700° C, and 1750° C for 10 hours. TEM thin foils are to be prepared and analyzed to determine information about the fiber/matrix interface.

All heat treatments have been performed and thin foils are ready for TEM inspection for the YAP-Alumina project. However, all the specimens have not been analyzed since the TEM has not been in operating condition. TEM photographs must be studied to find a relationship between the amount of reaction, time and temperature. The fiber/matrix interface project also is not complete. More heat treatments are to be performed and TEM foils need to be prepared from them. More composites may be made from different fiber/matrix systems.

Structural Ceramic Composite Processing

Task Order No. 96a
Student Support Program

John R. Welch
Wright State University

31 December 1992

Guiding Engineer
Dr. Ronald Kerans
WL/MLLM

ACKNOWLEDGMENTS

I would like to thank Dr. Randall Hay for the opportunity to work and learn about structural ceramic composite processing. I also would like to thank the staff of the metals branch at WL/MLLM for their assistance and training in the use of the equipment and for teaching me various techniques in the preparation of T.E.M. sample slide preparation.

TASK ASSIGNMENT

This task has mainly been in the preparation of T.E.M. sections of various structural ceramic composites. Working as an aide to Dr. Randall Hay, I have learned to mount fibrous ceramic composite materials in a compound called epomet that is pressurized and heated in a mounting press. After mounting these materials, the material end of the sample is polished on diamond embedded grinding disks. A bevel is put on the cylindrical sample edge by using a rough grinding disk. From that point, the sample is washed and secured in sample holder that also acts as a weight. The samples are placed on a mechanical polisher called a vibromet, which has a one-half micron diamond solution in which the samples rotate. The samples stay on the polisher for a couple of days until the surface has become finely polished. The samples are removed and washed.

The next step in preparing the sample is to mount the sample on a glass slide by heating the sample and slide on a hot plate. When the sample has become hot enough, a wax-like glue is applied to the composite face of the sample. After cooling for a few seconds, the sample is placed on a glass slide. The sample must be pressured against the slide, for it is imperative that almost all the air bubbles that may form in the glue solution be removed. After the sample cools, it is mounted in a low speed saw and a thin piece of the sample slide is cut away.

The next step of the process is the most critical phase of preparation. The sample mounted slide is initially ground down with the diamond enmeshed grinding disks. This is done in stages, starting with a 68 micron disk, then down to a 30 micron disk, a 12 micron disk and sometimes a 6 micron disk. The purpose is to get the sample as thin as possible without losing the sample off the slide. There is a fine line between having ground the disks as much as possible and the point where the disks will pull the sample off the slide. When the sample is pulled off the slide, all the previous steps except mounting the sample in epomet must be redone.

Once the rough polishing phase is completed, the fine polishing phase begins. This is an often lengthy operation that involves polishing the slide mounted sample on a mechanical wheel that is covered with silk cloth. A slurry mixture of diamond paste and diamond suspension solution is placed on the silk. The sample is polished in the solution while the mechanical wheel is rotating at a speed of about 400 revolutions per minute. This polishing process is also done in stages. There are many different grades of the diamond suspension and paste available in the metals lab, ranging in coarseness from 45 microns to as fine as one-fourth micron solution. For the ceramic composite samples, a nine or 15 micron slurry is used first and eventually a three micron slurry is used. On occasions, a one micron paste and suspension is used, but usually it is not necessary to polish the sample with this fine of a solution. Each phase of the slurry polishing usually takes at least 10 - 15 minutes per sample. Certain samples, for reasons unknown, require a great deal more time in the polishing stage. The ultimate goal of the polishing phase is to get a sample that is approximately one grain size thick, which is usually about 10 microns.

The next phase of the operation is to glue tiny copper grid washers in areas of the sample that are thin and in relatively stable structure. The samples are viewed under a microscope and the best areas are chosen. Then the copper grid washers are placed on the areas by tweezers. The area in which they are placed is a bit of a guess. It is necessary to hold a picture of the preferred area in your mind when trying to place the grids over the areas. It is also important to use as little of the glue on the grids as possible, for the glue will cover the sample and render it useless if too much is used. Copper washers are then glued on top of the grids.

The next phase of the operation is done by Dr. Hay. The grids are removed from the slide by putting them on the hot plate. When the wax-like glue heats up, the copper grids can be removed from the slide. The grid sample is placed in an ion milling machine that shoots argon atoms at a 15 degree angle at the sample. This causes a hole to form in the sample so the interphase between the fiber and the ceramic composite can be viewed more intricately. Dr. Hay places the sample in an electron microscope, where the sample can be viewed at a magnification of 3100 times the actual size. Dr. Hay can make photographs of the sample from the electron microscope.

Unfortunately, most of the samples we have worked with to this point have shown some disturbing tendencies. The fiber in the composite cracks during the heat treating process or in some cases, there has been a reaction between the fiber and the composite in the interphase of the material sample. There is one material that does show promise. A molybdenum, zirconium, and palladium composite seems to turn out consistent samples with fibers intact which also have not reacted with the fiber.

The goal of the research is to produce a high strength, light weight ceramic composite to be used in a variety of possible applications. These applications, if the proper composite structure is developed, may even eventually include making aircraft engine turbines out of the ceramic composite material.

SYNTHESIS AND CHARACTERIZATION OF POLYMERS

Task Order No. 97
Student Support Program

Aric Rothman
Wright State University

31 December 1992

Guiding Engineer
Dr. Robert Crane
WL/WLPJ

ACKNOWLEDGMENTS

I would like to thank Jim Lupo for sharing his considerable knowledge of the Unix operating system with me. Because of Dr. Lupo's guidance, my understanding of some of the intricacies of Unix has been increased.

TASK ASSIGNMENT

The objective of this task assignment was to assist a senior chemist in the characterization of siloxane polymer materials incorporating spiropyran molecules for use as matrices for optically active materials. Results of this research could be employed in the creation of practical non-linear optical materials.

The addition of a Silicon Graphics IRIS 4D/420VGX to MLPJ has opened up several new possibilities in molecular modelling efforts. The SGI has advanced graphics hardware and is currently running AVS (Advanced Visualization System). AVS makes possible the development of imaging routines through the building of block diagrams rather than writing code. Existing applications will be converted, both commercial programs and one I have previously developed, for use with AVS. AVS will further automate the process of developing visualization systems.

Several utility programs have been developed. These include: DSPACE, used to determine the spacing of atoms in a crystalline structure given a range of geometric parameters; DP, parses e-mail messages containing sections of a large dynamic process database; FMAT, accepts the textual output from a MOPAC job containing force matrix data and translates that force matrix to a format compatible with MATLAB; R2P, translates RTF files to PDB format; and SPEC, a linear algebra program to assist in the analysis of CD spectra data.

The systems used for the majority of modeling applications are Unix based running X-Windows or a X-Windows superset. As such, they can support multiples in graphical environments. Unfortunately, the use of graphical applications on these systems has been limited to the main console terminals. Adequate terminal software for the Macintosh's commonly used by most personnel was not available, resulting in a backlog of important tasks with researchers waiting for time on these machines. Using A/UX, the Mac Unix environment, can run these programs from a Macintosh. With further work, it may be possible to increase the availability of important applications such as AVS, Quanta, Polygraf and Spartan to those who need them.

Equipment used include a Silicon Graphics IRIS 4D/420VGX, a Sun Sparcstation, various Apple Macintoshes and a DEC VT220 terminal.

I have improved my knowledge of the Unix operating system. I am currently studying AVS and will begin adapting programs to this environment.

The programs mentioned above are in use by several researchers in the laboratory. Researchers frequently have a preference of language for writing their requested program. This has resulted in an increased proficiency in C, FORTRAN and Unix shell scripts.

I have not been involved in the interpretation of data resulting from the use of programs I have developed. The nature of the data, usually concerning geometries and force interactions of molecules, is not within my field of expertise.

Synthesis and Characterization of Polymers

Task Order No. 98
Student Support Program

Karen Hyssong/Myers
Wright State University

31 December 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to thank Dr. Nat Natarajan and Dr. Tom Cooper who have directed and assisted me in developing and performing the goals of my task. I also would like to thank Tim Grinstead and Sungmee Yoon for their help in the synthesis procedures. Although she no longer works at the Materials Lab, I would like to thank Laura McGivern who demonstrated spin coating and shearing techniques for making thin films.

TASK ASSIGNMENT

The objectives of my task were to synthesize and characterize biopolymers that contained nonlinearly active molecules. These polymers are then used as matrices for laser hardening devices. This involved coupling reactions with polypeptides and nonlinear optical (NLO) compounds. Synthesis was followed by characterization procedures, which involved various measurements of the compound's properties to compare with starting materials. Using this information, conclusions about the possible NLO properties and applications of these materials can be formed.

Using coupling reactions, compounds containing non linear optical properties such as, Disperse Orange 13, 1-pyrenyl methanol and p-nitrobenyl alcohol are attached to a polypeptide backbone. The polypeptides used were poly(1-glutamic acid), poly (methyl 1-glutamate) and poly(benzyl-glutamate). The two synthesis procedures involved were carbodiimide coupling and transesterification. The reaction set up was similar for both techniques using a round bottom flask, heating mantel, stir plate and a condensor. Purification of the substituted polypeptides was preformed by consecutive precipitations until all starting materials were removed. Once synthesis and purification was complete, the substituted polypeptides were characterized. Various equipment used in my research was Fourier Transform Infrared spectroscopy (FTIR), Circular Dichroism spectroscopy (CD), UV-Vis spectroscopy, rotovap, a clean room spin coater and Dektak scanner.

The substituted polypeptides were analyzed to determine many properties. The percent substitution of the dye was determined though various methods. These were UV-Vis spectroscopy, elemental analysis and NMR spectroscopy. CD spectroscopy was used to determine the structure of the polypeptide and to determine if the dye exhibited and induced dichroism, once attached to the polypeptide. Thermal gravimetrical analysis (TGA) was performed on each polypeptide to determine at what temperature the polymer will begin to decompose. Differential scanning calorimeter (DSC) results were analyzed for melting points and other types of phase changes.

Producing thin films of various substituted polypeptides involved mostly trial and error combinations of various solvents and concentrations. Eventually, some acceptable films of p-nitrobenzyl alcohol with each of the polypetides were made in which aligning and NLO properties are being tested.

The final results for the aligning and NLO testing have not yet been reported. We hope to see a difference in the aligning due to the different polypeptide backbone. From the trends found in the systems I have synthesized, we hope will assist in the future in choosing the best system for the wanted properties.

SYNTHESIS AND CHARACTERIZATION OF POLYMERS

Task Order No. 99
Student Support Program

Scott M. Brittain
Wright State University

31 December 1992

Guiding Engineer
Dr. Tom Cooper
WL/MLPJ

ACKNOWLEDGMENTS

I would first like to thank Dr. Tom Cooper for his assistance and advice, and also would like point out that 1st Lieutenant Brian Sennett and 2nd Lieutenant Kyle Gresham have aided in my research with helpful advice and assistance.

TASK ASSIGNMENT

This task was undertaken to enhance the production of laser hardened materials for the electromagnetic materials survivability branch. Materials adequate for laser hardening purposes are still in a developmental stage, and the possibilities are of a magnanimous size. This task was initiated so that optical materials can be studied and produced so that other optical studies and developments can proceed.

The Department of Defense and the Air Force are interested in long term investigations of these optical materials, and this task involved getting the first steps of a long term experiment going. This initiation, as implied by the task description, involved the synthetic production of polypeptides (small proteins) that are usually produced by living organisms. These synthetic polypeptides are the polymers needed for further optical manipulations. The task order involved synthesizing and characterizing a peptide backbone that would retain it's characteristic biological conformations to material science situations.

As with the previous task, this involved producing synthetic peptides for eventual use in laser hardening or optical situations. This process is complex and involves many steps dealing with instrumentation and computer related problems. The actual chemistry associated with synthesizing a peptide is complicated, but is only the first step in successful production of peptides. These desired peptides are of an alpha helical secondary structure and must be extremely pure before further derivitization can be accomplished. This dependence on ultra pure products necessitates the need for many post-synthesis purification procedures, including ion-exchange chromatography, gel filtration chromatography, isoelectric focussing and reverse phase high performance chromatography. These processes are difficult, but essential to successful experimentation.

The solid phase peptide synthesis is always followed by tedious and problematic purification and characterization steps. To develop these peptides, another step in this process may involve computer modeling of the peptides so that the desired structure and shape can be acquired once the chemistry has been accomplished.

The structure of the product is also very important. Other analytical techniques must be, and have been, undertaken so one can be sure about the microsecondary structure of these materials, as predicted by the computer modeling. The research is still interesting and challenging, and the best is yet to come.

The synthesis of a 5 residue peptide (Glu-Ala-Ala-Ala-Ala) has been accomplished with minor difficulties, along with optimization of liquid chromatography techniques and electrophoretic techniques. The reverse phase chromatography initially proved to be quite problematic, but once encountered often enough, the process became a helpful tool, rather than a hindrance. Also, problems with capillary electrophoresis of our products have been worked out and again this analytical tool has proven to be an asset. Some gel electrophoresis have been performed, but still needs to be optimized, along with the isoelectric focusing.

Amino acid analysis via hydrolysis has also been qualitatively optimized with minor problems to be worked out in quantitation, but also plays an important role in determining whether our products are pure or not.

Protein sequencing is another process that has been encountered and found to be a very sensitive and time consuming process that still requires much optimization before becoming one of our trusty analytical tools. As a whole, three or four analytical techniques have been optimized and agreement between these techniques should confirm product purity with little or no doubt remaining. The productivity of the peptide chemistry laboratory as a whole has increased greatly, and many interesting research projects are feasible now. The post-synthesis studies are now being undertaken to study the secondary structure of these synthetic peptides and compare those anticipated from computer modeling data.

Different techniques, such as mass spectrometry and nuclear magnetic resonance, are being sought after to better our analytical techniques. More and more results are to be anticipated as the peptide lab becomes more productive.

Synthesis and Characterization of Polymers

Task Order No. 100
Student Support Program

Laura McGivern
University of Dayton

31 December 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to thank Dr. Zbigniew Tokarski for his time and effort in guiding me toward the goals of my task. He was very helpful in explaining the meaning and the purpose of my task as well as instructing me on how to properly use much of the equipment in the labs. Tim Bunning, Kelly Menster, Sungmee Yoon and Keith Obermeier were also helpful in assisting me in my research.

TASK ASSIGNMENT

The objective was to synthesize and characterize nonlinear optical (NLO) materials consisting of chromophores and mesogens attached to cyclic siloxane based polymers. The synthesis involves physically making the materials from other chemical compounds in the lab. The characterization process involves studying the materials with various instruments for different properties in order to draw some conclusions about the physical and optical behavior of the newly synthesized materials.

The mesogens, biphenyl and cholesterol, and the nonlinear optical chromophore, disperse red one dye, are considered side chains being attached to the polymer "backbone," which is the cyclic-penta-siloxane molecule. Initially, chromophores and mesogens were synthesized. Later they were attached to the cyclic-penta-siloxane molecules in different ratios for a total of twenty-three siloxane synthesis reactions.

The equipment used for the synthesis includes glassware for the distillation set-up, and centrifuge equipment for the purification process to recover the final product. The equipment involved in the characterization procedure is much more extensive. It includes both a Fourier Transform Infrared Spectrometer (FTIR) and a Thin Layer Chromatography (TLC) set-up for monitoring the extent of reaction, and the extent of purity of the material synthesized.

A differential scanning calorimeter (DSC) is used to do some thermal analysis on the materials and to determine where specific transitions, such as a glass transition point occur for each material. X-ray diffraction experiments are then done on each material to determine the physical structure and design of the material. Finally, thin films of the material are made, using a spincoater and a dektak scanner, so that second harmonic generation (SHG) tests can be run on them, and then the optical, in addition to the thermal and physical properties of the materials can be studied.

It was discovered during the synthesis that the higher the ratio of Disperse Red One to Cholesterol and Biphenyl, the lower the final product per cent yield would be.

Synthesis with 100% side-chain attachment, however, can be relatively easily obtained. During the DSC analysis, it was determined that a second order phase transiton (or a clearing point) in the material containing 50% cholesterol occurred 490 and 495 Kelvin indicating multiple liquid crystalline phases. The glass transition temperatures of the materials were all nearly the same around 320 Kelvin.

X-ray studies showed that the primary reflection increases, from 25 to 30 angstroms, with Disperse Red 1 concentration, and that Disperse Red 1 is larger than Biphenyl and requires larger separation distance. The packing structure of all the 50% cholesterol compounds is lamellar-like. Compounds with 50% or greater biphenyl have nematic-like structures. The thin film studies have shown that colored, glassy films of good optical quality can be produced with the siloxane materials. Studies continue in order to determine other physical and optical properties of the materials.

Synthesis and Characterization of Polymers

Task Order No. 100a
Student Support Program

David Stitzel
Wright State University

31 December 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I would like to recognize the following persons who helped guide me through my task assignment: Dr. Robert Crane, Dr. Zbigniew Tokarski, Dr. Tom Cooper, Dr. L.V. Natarajan, Tim Grinstead, Sungmee Kim and Karen Myers.

TASK ASSIGNMENT

Task Assignment 100 involved the synthesis and classification of an optical polymeric material which contained nonlinearly active molecules for use as laser hardening materials. The synthesized polymer led to a better understanding of what exactly a nonlinear optically active polymer was and how the polymer could be produced.

The synthesis of the polymer was accomplished by performing several reactions. The first of which was the difficult transesterification reaction which produced benzyl-l-glutamate (BLG) from l-glutamic acid, benzyl alcohol and HCI as the acid catalyst. The second reaction was the anhydride synthesis using triphosgene, tetrahydrofuran and the previously produced BLG. The third reaction was the synthesis of the polymerized product using dimethyformamide, NaOH (initiator) and the previously produced anhydride.

The product of each reaction, in addition to the final polymerized product, was purified and analyzed using various high technology instruments. Some of the instruments that were used includes a mass spectrometer, a fourier transfer infra-red spectrometer and a nuclear magnetic resonance spectrometer.

The data that was collected from these various high technology instruments was analyzed by the interpretation of the spectra produced. Once the spectra had been interpreted it was then possible to examine the results of the numerous reactions and the overall synthesis of the polymerized product.

After analyzing the data that was collected it was found that the transesterification reaction was very difficult and that this reaction was very sensitive to slight modifications in the less than adequate literature procedures. Once the transesterification reaction was successful in producing BLG the following reactions proceeded smoothly and the polymerized product was obtained.

Metallic Composites II

Task Order No. 101a
Student Support Program

Christopher W. Palser
Wright State University

31 December 1992

Guiding Engineer
Dr. Dan Miracle
WL/MLLM

ACKNOWLEDGMENTS

I would like to sincerely thank the persons at the Materials Laboratory who "showed me the ropes" of metallography, sample preparation and analysis namely: Jothye Menon (visiting scientist), Mike Scott (Metallography Lab supervisor), Cindy Emerick (SOCHE student). Most of all, I would like to thank Dr. Dan Miracle, who had patience with my initial frustrations, and whose "table-side lectures" taught me more useful lessons in materials and materials research than any classroom or textbook ever could.

TASK ASSIGNMENT

The objective of this Task was to determine interface properties and deformation characteristics in advanced metallic composites for high temperature applications.

The specific objective of this Task was to establish techniques to produce single fiber composites and multi-fiber composites in a range of geometries and for a number of matrix/reinforcement combinations. Additionally, the task was to produce composites for determination of interface properties and deformation characteristics in advanced metallic composites, and to conduct mechanical tests of the single and multi-fiber composites, and to analyze the data resulting from the composite production and testing.

The purpose behind this Task is to produce lighter, stronger high-temperature materials for use in jet engines and aircraft airframes and structures. A structure or engine component made of a metal-matrix composite (MMC) will be significantly stronger at both normal temperatures and at the higher operating temperatures found in jet engines, and will have a lighter density. Given these improved mechanical properties, less material of a lighter weight will be used in production to form a much lighter component, resulting in substantial savings in aircraft weight and fuel. It is obvious that advancements in the production of MMC's would enable them to be manufactured in a cost-efficient, reliable manner and therefore be widely used in aircraft production would provide long-term savings in fuel usage for aircraft using MMC's in major components.

The student's role in this research was to prepare samples, perform analyses on the prepared samples, and to understand and assist in the interpretation of the results of the analyses performed.

The bulk of the research performed on this task by the student was in the area of long-term heat-treated matrix alloys and MMC's. Long-term heat-treatment of MMC's very closely simulates the long-term exposure of MMC's to a high-temperature environment such as a jet engine. Research in this area is important, since long-term exposure to high-temperatures can drastically effect the mechanical properties of materials used.

Studies were performed on a completely new class of Titanium-Aluminide-Niobium alloys consisting of 55 atomic percent Titanium, 22 atomic percent Aluminum, and 23 atomic percent Niobium (commonly referred to as Ti-22-23), with Silicon Carbide (SiC) or SCS-6 fibers. This composite is generally known as Ti-22-23 with SCS-6 fibers, or simply, Ti-22-23 composite. Ti-22-23 matrix is composed purely of Ti-22-23 alloy, while the composite is formed of uni-directional layers of SCS-6 fibers pressed at high-temperatures and pressure between thin foils of the matrix alloy.

Samples were sectioned from the as-received sheet into approximately 1 x 2 x 0.1 cm specimens for heat treatment and subsequent analysis. Heat treatments were performed at varying temperatures and times, varying from 500-800°C and 500-1000 hours. Following heat treatment, specimens were sectioned and mounted in a thermoset mount for metallography. After mounting, metallography was performed by rough grinding through final polish for scanning electron microscope (SEM) and microprobe an analysis. Specimens for x-ray diffraction analysis were sectioned, then fine ground to remove surface oxidation.

Scanning electron microscopy was performed to analyze the phases present in the as-received and heat-treated samples so that the phase transformations that occurred during long-term exposure to high temperatures could be determined. Microprobe analysis and x-ray diffraction was performed to analyze the specific chemical composition and crystal structure of each phase present in the matrix alloy. Detailed follow-on analysis of results is yet to be performed, but initial interpretation of data was performed as data was collected.

The student had an intimate involvement in each step of the task, from sample receipt to final analysis. As the task progressed, the student became more skilled in each step of the research process, including the essential skills of sample preparation via metallography. Also learned were the operation of x-ray diffraction equipment, the scanning electron microscope, and the microprobe analysis electron microscope.

As stated, detailed analysis of the results has not been performed, since analysis of all samples is not yet complete. However, initial interpretation of data supports the suppositions made that significant phase transformations do indeed occur, but the extent of these reactions is not yet known.

Intermetallic Eutectics

Task Order No. 102
Student Support Program

Kevin L. Kendig
The Ohio State University

21 September 1992

Guiding Engineer
Dr. Dan Miracle
WL/MLLM

ACKNOWLEDGMENTS

For their support and insight during my research this summer, I would like to thank the following: Dr. Dan Miracle, Dr. Mohammad Khobaib, Dr. Karen Teal, Dr. Sion Pickard, Dr. Satish Rao, Dr. Jyothi Menon, Cameron Begg, Tony Houston, Mark Dodd, Eric Fletcher, Bob Lewis and Michael Scott.

TASK ASSIGNMENT

During this Task, the student worked primarily on three projects. The first was the production and analysis of single fiber composites in which the fiber's major axis was perpendicular to the direction of loading (transverse). The fiber interface was examined with both optical and electron microscopy to check for cracking caused by its processing. Successful samples were made into tensile samples. Testing of these samples has begun, but there are no results so far.

The second project was the analysis of the titanium alloy β-21S in the as-received condition and after heat treatments using x-ray diffraction, chemical analysis, optical and electron microscopy and electron microprobe. Three samples were cut for each of four specimen groups: as-received, 20 hour, 40 hour and 70 hour (heat treatments at 816°C). One set of samples was mounted and polished for x-ray analysis; and the last one was sent for chemical analysis.

The last project was measurement of radial residual stresses in a Ti-24-11 composite due to processing. This was accomplished by analyzing x-ray diffraction patterns for changes in the lattice parameter. Initial x-ray runs of both the composite and the matrix material alone were taken in order to choose a peak for closer examination. The highest 2θ peak which was not obstructed by other peaks was chosen for analysis because the expected shift from residual stress in the composite increases with increasing 2θ.

Fabrication and analysis of the transverse, single fiber composites involved hot-pressing and microscopy. A silicon carbide fiber (SCS-6) was pressed between two one by three inch Ti-6-4-foils. Consolidation of the composite was accomplished in a hot-press under vacuum at 1800 c with 2000 lbs. load for one hour. This hot-pressing schedule was selected after several experiments because it yielded an uncracked interface. Samples were radiographed after hot-pressing to check for cracking of the fiber. At this point some of the samples were cut into reduced gage tensile test specimens. The remaining samples were cut, mounted with the fiber direction perpendicular to the surface and polished. Optical and electron microscopy yielded photomicrographs which were used to evaluate the fiber-matrix interface.

The ß-21S project utilized several techniques to characterize changes in heat treated ß-21S. X-ray diffraction patterns were made for each of the four heat treatments. These were indexed and the precise lattice parameter was found using a linear regression technique to minimize error due to absorption by the material. The second set of specimens was set to be chemically analyzed to Ti, Mo., Al, Nb, Cr, O, Fe, N and Si. The third set was mounted and polished. Optical and electron microscopy of the polished specimens were done to examine any changes in microstructure with increasing heat treatment times. An electron microprobe was used to detect any variation in composition in each of the two phases (α and ß) which were found to be present; this was done for the 20 and 70 hours, and quenched into a beaker of water directly following removal form the furnace.

The residual radial stresses in a Ti-24-11 composite were measured using a Rigaku x-ray diffrectometer with computer control data collection. The initial Ti-24-11 matrix pattern from 20 to 140 degrees was 2θ was indexed. X-ray diffraction patterns were done for both a matrix and composite sample between 100 and 140 degrees 2θ at one degree per minute to get a more precise view of the area of interest. The known Young's modulus of Ti-24-11 and an approximate residual stress level was used to calculate an expected shift in the peak chosen for study. Three runs for each sample were made over a two degree range at 0.1 degree per minute to increase observed intensities of the chosen peak. The samples were held in the x-ray sample holder by cellophane tape. The possibility of a random error due to an uneven tape surface was examined by reloading each sample for each run. The peak centers were found manually instead of by the peak finding program which had proven unsatisfactory.

The transverse single fiber composite samples have not been sufficiently tested to report results. Some samples are awaiting tensile testing where acoustic emissions will be monitored in an effort to pinpoint the debonding of the fiber form the matrix. More samples are in various stages of preparation for testing.

The ß-21S heat treatment study gave several results. The precise lattice parameters were found for the BCC phase, ß, of the ß-21S samples (see table 1).

Table 1

Sample	**lattice parameter**	**wt% O**	**wt% N**
as-received	.32448 nm	.173	.013
20 hours	.32530 nm	.314	.016
40 hours	.32507 nm	.555	.030
70 hours	.32445 nm	.698	.029

There was no obvious trend in this data. Optical microscopy showed no change in average grain size, but electron microscopy showed significant coarsening of the second phase, ß, from as-received to 70 hours at temperature. The sample quenched after a 70 hour heat treatment showed that some coarsening was occurring during the air quench, but also that the phase was stable and annealing temperature and not just a precipitate formed upon cooling. Chemical analysis confirmed the expected bulk composition of the material. It was also shown that the oxygen and nitrogen contents increased with increasing time at temperature (see table 1). The effects of the increase in oxygen and nitrogen were not determined. The microprobe analysis of the two phases present in the 20 and 70 hours anneal samples showed that there was no statistically significant variation in the compositions (see table 2).

Table 2

Sample/Phase	**Ti**	**σ**	**Al**	**σ**	**Nb**	**σ**	**Mo**	**σ**	**Si**	**σ**
20 hour/ß	89.7	1.2	6.6	0.3	0.7	0.2	2.4	1.2	0.5	0.0
70 hour/ß	91.4	0.4	7.1	0.2	0.5	0.1	0.6	0.4	0.5	0.0
20 hour/α	82.7	0.6	5.1	0.1	1.8	0.1	9.7	0.7	0.6	0.0
70 hour/α	83.4	0.1	5.1	0.1	1.8	0.0	9.1	0.1	0.6	0.0

note: 5 points were analyzed per phase

The measurement of the radial residual stress in a Ti-24-11 composite gave promising results. A theoretical shift was calculated based on the 116 degree 2θ peak, a 93 GPa Young's Modulus for the matrix, and an expected 80 MPa residual stress. This value came out to 0.077 degrees θ (0.154 degrees 2θ). It was previously unclear whether the random error caused by the variability of the cellophane tape's surface would make the measurement of the residual stress' peak shift impossible. With three runs per sample of the 116 degree 2θ peak, the standard deviation was found to be within acceptable limits (see table 3).

Table 3

Peak Positions

	matrix	composite
	116.02	115.89
	116.01	115.82
	116.03	115.83
avg:	116.02	115.85
σ:	0.010	0.038

The difference in the average peak positions, 0.13, is very close to the expected value of 0.15. The 0.13 shift corresponds to a residual stress of 65.9 MPa. These preliminary results confirm that there is sufficient precision in the experimental set-up to yield useful results.

Nondestructive Evaluation

Task Order No. 103
Student Support Program

Edward D. Fisher, II
Wright State University

31 December 1992

Guiding Engineer
Dr. Curtis Fiedler
WL/MLLP

ACKNOWLEDGMENTS

I would like to thank Dr. Chris Bhagat and Dr. Curt Fiedler for giving me this opportunity to gain valuable experience in my field of computer engineering, and for their support and guidance throughout the task. I look forward to future task assignments under their leadership.

TASK ASSIGNMENT

This assignment can be divided into two major tasks and various minor tasks. The first major task was to accumulate data by way of laser scanning aluminum specimens of various thicknesses (.05" -.125") and at three laser frequencies (20 Hz, 200 Hz, 2 kHz) using a 3.5 W Argon laser. The overall objective is to use this technique for the inspection of aging aircraft for stress and metal fatigue.

"Thermal waves" are produced by the heat that results from the laser beam hitting the surface of the specimen. These "thermal waves" are then detected by a PVDF (polarized homopolymer of vinylidene fluoride) piezoelectric film transducer, which converts them into electrical signals, then sent to a lock-in device (which is designed to acquire very small signals, down to nanovolts), which amplifies and filters the signals. From the lock-in, the magnitude and phase of the signals are retrieved and fed into a computer program via an IEEE bus line; this program then converts this data into a pixel format that can be viewed on a monitor and scrutinized.

The second major task, which is still in progress, centers around the characteristics of the transducer itself. The PVDF film, which exhibits piezoelectric activity, acts as a parallel plate capacitor ($\approx$ 1pF), creating a voltage by means of the charge across its plates. The magnitude of this voltage is dependent upon the capacitance of the transducer (Ctrans), the capacitance of the coaxial cable connecting the transducer to a commercial preamplifier (Ccoax), the input capacitance of the commercial preamplifier (Camp), and any stray capacitance (Cstray). This dependence is realized by the following equation: $V = Q / (Ctrans + Ccoax + Camp + Cstray)$. Obviously, greater capacitance results in a diminished signal, thus the task is to reduce some of the capacitance, namely Ccoax, Camp, and Cstray. The result of reducing these capacitance is twofold, increasing both the magnitude of the signal and the frequency response of the transducer, which will enable us to take scans with greater detail over a broader range of frequencies.

The task is to design a unity-gain preamplifier, which will act as a buffer for the commercial preamplifier (Cin = 25pF), with little input capacitance (< 5pF) using a FET (Field-Effect Transistor) or perhaps a low input capacitance operational amplifier. This will reduce Camp from the commercial preamplifier's 25pF to my buffer preamplifier's < 5pF. The coaxial cable capacitance is easily eliminated by eliminating the cable; replaced by a short wire, while stray capacitance can be controlled by encasing the buffer preamplifier in an aluminum box. The improvement can be realized by the following equation: $V \approx Q / Ctrans$.

The minor tasks mentioned include characterizing transducers and writing various computer programs in the C and FORTRAN computer languages.

The characterization of transducers consisted of verifying the resonant frequency of commercial transducers, as well as measuring the frequency response, in order to determine the performance of each.

Some of the programs include a program to control the laser scanning by means of sending commands via an IEEE bus line to manipulate X and Y axis galvanometers: a program that receives the scanning data and translates it into pixel format for viewing, via X-Windows, on a monitor (mentioned earlier): and a program which "talks" to digitizing oscilloscope, via an IEEE bus line, for the purpose of retrieving displayed waveforms, scaling them and saving the scaled data to files that can be printed.

Characterization of Superconducting Thin Films

Task Order No. 104
Student Support Program

Benjamin Smith
Wright State University

30 September 1992

Guiding Engineer
Mr. Tim Peterson
WL/MLPO

ACKNOWLEDGMENTS

I wish to thank the Southwestern Ohio Council for Higher Education for the opportunity and support. My experience was rewarding and enriching because of many different influences. Dr. Patrick Hemenger provided me with support and encouragement. Tim Peterson, my mentor, was invaluable. Dr. Iman Maartense's advice and technical support truly served as a source of stimulation.

TASK ASSIGNMENT

The electrical resistivity of many metal alloys drops suddenly to zero when the specimen is cooled to a low temperature, most often in the liquid helium range, 4.2 Kelvin. In the last six years materials have been exhibiting this superconducting state at much higher temperatures, well beyond liquid nitrogen at 77 Kelvin. In the past three and one-half years I have been working at the Materials Lab at Wright-Patterson Air Force Base testing and evaluating these new superconductors. One method in particular is AC susceptibility.

The magnetic properties exhibited by superconductors are as dramatic as their electrical properties. The magnetic properties cannot be accounted for by the assumption that a superconductor is a normal conductor with zero electrical resistivity. In a weak magnetic field a superconductor will act as a perfect diamagnet, with zero magnetic induction in the interior. The magnetic properties of superconducting are central to the characterization of the superconducting state.

Susceptibility measurements determine the magnetic state of the entire sample, and gives a better indication of the extent to which the sample has transformed to the super conducting state. This is in contrast to the resistivity measurements which merely show when a continuous superconducting path is in place. AC susceptibility provides a better experimental indicator of the overall super conducting state, while the resistivity measurement system is a better practical guide for application purposes.

As seen in Figure 1, AC susceptibility can provide a wealth if information when used to study the high temperature superconductors. These materials are characterized by superconductors grains or inclusions which have a relativity high critical temperature. However, the weak-link inter granular coupling between the grains produces a lower critical temperature. Often two peaks in complex susceptibility are observed, corresponding to the intra-grain and inter-grain current components, respectively.

The advantage of AC susceptibility it is a quick and accurate means to characterize superconducting samples. This is a non-destructive method that can quickly screen out the samples. Since AC susceptibility scans all strengths of the superconducting phases in a sample, it is helpful in understanding the mechanisms that limit the critical current. Figure 2 is a schematic of the system used at W.P.A.F.B.

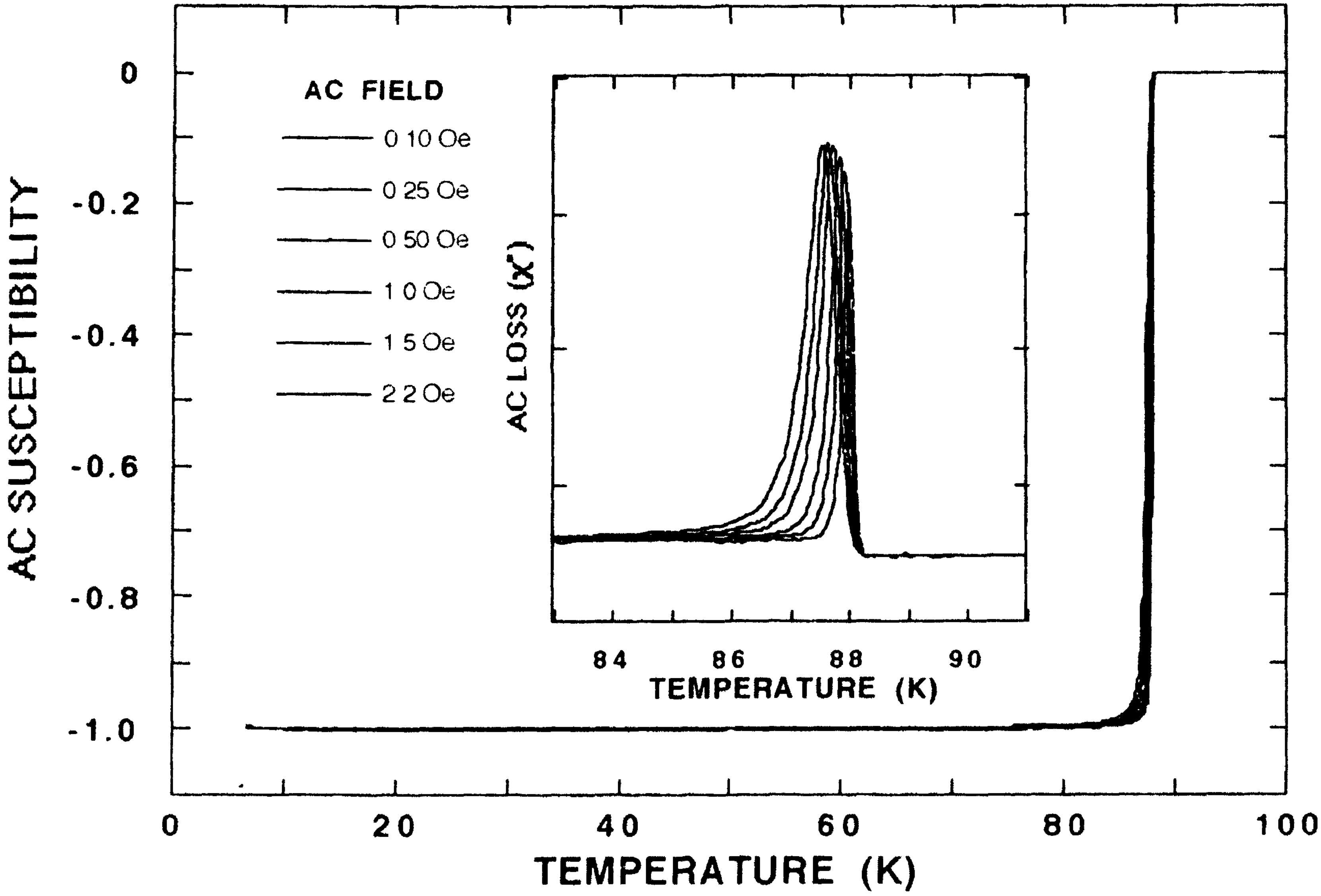
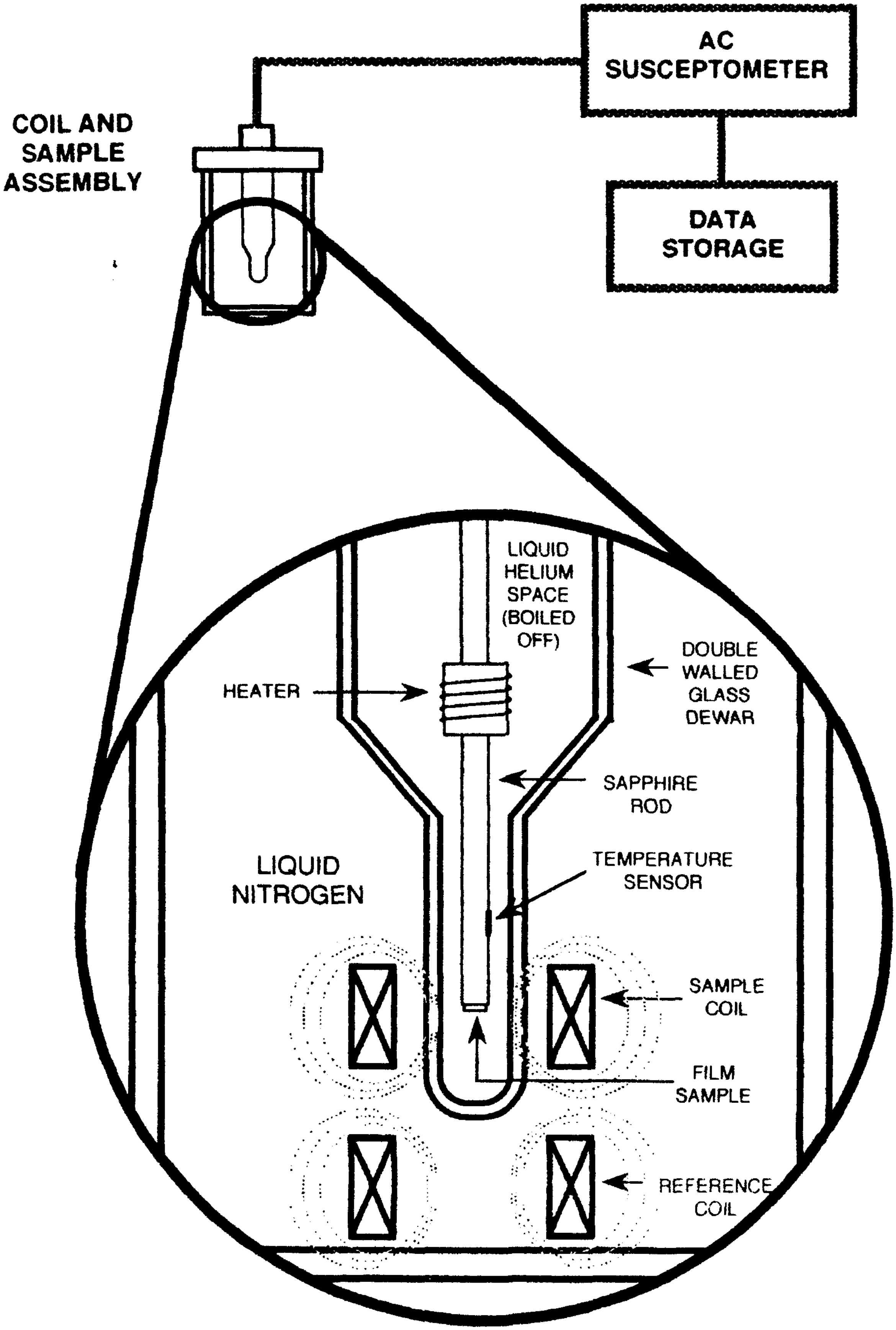

AC SUSCEPTIBILITY
0
-0.2
-0.4
-0.6
-0.8
-1.0
0
20
40
60
80
100
TEMPERATURE (K)
AC FIELD
0 10 Oe
0 25 Oe
0 50 Oe
1 0 Oe
1 5 Oe
2 2 Oe
AC LOSS (χ″)
84
86
88
90
TEMPERATURE (K)

Figure 1


AC
SUSCEPTOMETER
COIL AND
SAMPLE
ASSEMBLY
DATA
STORAGE
LIQUID
HELIUM
SPACE
(BOILED
OFF)
HEATER
DOUBLE
WALLED
GLASS
DEWAR
SAPPHIRE
ROD
TEMPERATURE
SENSOR
LIQUID
NITROGEN
SAMPLE
COIL
FILM
SAMPLE
REFERENCE
COIL

Dynamic Oxygen Absorption Study

Task Order No. 105
Student Support Program

Mark Roselius
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31 December 1992

Guiding Engineer
Dr. Harvey Paige
WL/MLBT

ACKNOWLEDGMENTS

I wish to extend my sincere appreciation to Dr. Harvey Paige and Mr. Ed Snyder of the Wright Laboratory Material Directorate for their guidance and assistance during the performance of this task and with OXAB data analysis. I also would like to thank Dr. Jim Liang of UDRI for his assistance with infrared data analysis. Finally, I would like to thank Dr. Arthur Krawetz of Phoenix Chemical Laboratory for his assistance and for providing the OXAB reference manual.

TASK ASSIGNMENT

OXAB testing for the second half of 1992 consisted of the completion of testing begun in the first six months. In addition, a series of tests were run on jet fuels as part of an informal demonstration of OXAB potential for evaluating fuel oxidative resistance. These tests were conducted on the fuels 2827 and 2747. Reagent grade hexadecane (Aldrich) was also run as a standard.

The second step of a two step polyphenyl ether evaluation was conducted during the first half of 1992 and consisted of a series of tests using a variety of metals to determine what influence they have on the oxygen absorption rate of polyphenyl ethers. The results will hopefully offer insight into the potential corrosive effects on the metal surface in contact with the fluid and suggest whether anticorrosion additives will be needed in the end use.

Finally, infrared data were collected in an effort to understand better the possible oxidation mechanisms involved in the OXAB studies.

Oxygen absorption rate measurements were conducted using the Phoenix Chemical OXAB instrument. Tests typically ran from 6 to 10 days and were conducted on 7.5-8.5 gram quantities of polyphenyl ether, 5P4E. The temperature settings were 260, 280, 300, 320 and 335°C with at least 2 runs per temperature. A flowrate of 50 ml/min was used. Data was recorded using a Rustrak Ranger Datalogger. Data analysis was performed with software provided with the datalogger and a custom software package provided by Phoenix Chemical, both run on an IBM XT PC.

For the metals analysis, the system described above was used with a temperature setting of 300°C with the addition of a metal coupon or washer of the appropriate metal to the sample fluid. These coupons were all the same dimensions. Consequently, surface areas were constant. The metals used are aluminum, copper, 410 stainless steel, titanium and M50 (a tool steel alloy).

Infrared data was initially analyzed with a Perkin-Elmer model 1750 FTIR. This proved to be limited in the amount of useful information that could be generated from the results. As a result, infrared analysis was shifted to a Hewlett Packard GC/FTIR and analysis confined to ambient trap products.

For the fuels testing, the OXAB temperature setting was set to 180°C +/-1. This value was determined by the Propulsion Lab, (WL/POSF) Building 490, based on tests that would be used for comparison. The flowrate was maintained at 50 ml/min. and a 5.0 ml +/-0.5 sample volume was used.

The investigation of the influence of various metals on the oxidation process has opened up a new area of OXAB testing. While this investigation is not complete at this time, preliminary results of data collected through 140 hours revealed that aluminum has no discernable influence on the oxidation rate of 5P4E. M50, on the other hand, shows a substantial increase in the rate of oxidation during the first 24 hours before it falls off to a rate consistent with the baseline obtained with the virgin fluid. The copper test produced a substantial increase which appears to be sustained through 140 hours. Unfortunately, an external system failure interfered with the data collection. As a result, copper will have to be rerun. Titanium and 410 stainless steel were to have been examined but testing of polyphenyl ethers was suspended due to a change in emphasis to perfluoropolyalkylethers as a basestock to be considered for lubricants and hydraulic fluids.

To review, the M50 result is not yet understood. A coating on the coupon, shiny brown in color, was observed. This coating was removable with organic solvents. A possible explanation is that the products or reactants form a coating on the surface over a 24 hour period that acts as a barrier to any catalytic affect the metal might have. This time dependent reduction in surface activity would explain the drop in oxidation after the initial increase.

The aluminum coupon developed no coating and showed no visible signs of corrosion, coming out bright and shiny. The copper coupon exhibited some visible signs of corrosion and what appeared to be copper salts were found in the bottom of sample tube. No surface coating was detected.

Fourier Transform Infrared analysis of sample tube, ambient trap, and cold trap products was initially conducted using a conventional single beam instrument. Because of the thin films required, coupled with the peculiarities of the 5P4E spectrum and the fact that the products were all mixtures greatly limited what useful spectral information could be obtained. To get around this problem, a gas chromatograph with an FTIR detector was substituted. The GC performed the necessary separation of the various components of the mixture and the IR detector produced the needed spectra. In addition, the library function of the instrument was used as an aid in determining the possible structures of the products. This IR detector also could work with much smaller quantities. Due to GC column limitations, only ambient trap products were used for this analysis. Also, GC/FTIR system failures led to more delays with the main result being that analysis was confined to products from metals tests.

Initially it was thought that FTIR analysis revealed the primary products from the oxidation of 5P4E in the presence of metal ore phenol, m-bis (phenoxy) benzene and phenoxybenzene. However further examination of the data left substantial doubt as to the validity of the results. Consequently GC/FTIR results are now considered inconclusive.

Oxidation rate curves generated by the jet fuel samples proved to be substantially different than those generated with lubricants. This is to be expected since fuels are intended to oxidize. Based on the results, jet fuel has their maximum rate of oxidation at the start of the test with the rate deceasing quickly in the first eight hours. The hexadecane standard follows the same pattern. However, the OXAB clearly demonstrated that there is a marked difference in the two fuels tested.

Sample	TMAX	MAR
Hexadecane	0	15.8
2747	0	8.2
2827	0	0.6

TMAX: Time to maximum absorption rate(hrs)
MAR: Maximum absorption rate (10^{-6} mol/gram/min)

Because the curves generated are very different from those of lubricants, the absorption rate at 4 hour time intervals was examined.

Time	Hexadecane	2747	2827
4hr	10	4.4	.45
8hr	7.1	3.3	.32
12hr	5.3	2.7	.27
16hr	4.4	2.1	.31

Units for absorption rates are 10-6 mol/gram/min.

Examination of the MAR ratios reveals that 2747 absorbs oxygen approximately 10 times faster than 2827 and a half the rate of the standard, hexadecane. These ratios essentially remain consistent through 16 hours. As a result, 2827 can be considered approximately 10 times as resistant to oxidation as 2747.

CONSOLIDATION OF COMPUTER PROGRAM NDSANDS

Task Order No. 106
Student Support Program

Hugh Cullin
University of Dayton

31 December 1992

Guiding Engineer
Dr. G. P. Tandon
WL/MLBM

ACKNOWLEDGMENTS

I would like to thank Dr. G. P. Tandon for his everyday guidance. I would also like to thank Dr. N. J. Pagano and Dr. Tandon for initiating all the paperwork necessary for this task. Dr. David Chu also must be thanked immensely for writing some subroutines which aided in the overall project. I would like to thank the Student Support Program for giving me the opportunity to gain invaluable experience in problem solving which this task has encompassed.

TASK ASSIGNMENT

The program NDSANDS, originally written on an IBM compatible machine, was taken in its IBM form and transferred to the Macintosh. The problem was not just this simple, however. The software package Mactran was used for the Macintosh. The programming language was found to be slightly different on each machine. This caused some inherent problems which were not foreseen by Dr. Tandon at the outset. Dr. David Chu wrote some subroutines to help remedy this problem.

NDSANDS stands for N Directional Stiffness AND Strength. This program theoretically looks at materials called composites and determines how they will react to different stresses, shears and other forces. The strength of the different composites also can be determined from this program. A new composite also can be created by the user and it can be theoretically tested. If the user likes the results of the test, a laboratory test of the material can be performed next. If the material passes the lab test satisfactorily, perhaps that composite will be used on the next space shuttle mission, or on a new bomber. These are two possible, practical uses for composites.

The program itself encompasses many different subroutines contained in over twenty different files. The program was modified by critically examining each one of these subroutines individually. By doing the analysis in this way, problems were eliminated one at a time. A valuable tool in this process was the use of dummy subroutines set up to look at just a small portion of one subroutine. By looking at each part of each subroutine separately, problems can be analyzed much more quickly and subsequently eliminated. Once one part of a subroutine worked properly, the next section could be analyzed. This process was repeated until the entire program, with all subroutines included, was working.

The next step was to make the program look better to the user. The large data files which appear time and again throughout the program were put in even alignment. Much of the text which appears was also altered slightly to make the program more user-friendly. The ability to exit the program from more places and go to previous screens was added to make the program more user-friendly as well.

The main menu of the program includes several options. These options include the material library, the composite library, composite analysis, parametric study and the plots. The material library contains many different materials which may be used to create or to change a composite. New materials may be added or changed. The composite library contains present composites. New composites may be created while old ones may be deleted or changed. These composites can have either a parametric study conducted on them, or they may have an analysis conducted on them. Each different test runs a composite through a different set of tests which can be varied by the user. The results of each test can then be viewed. Plots of each test also can be viewed by the user.

The project is now in its final stages. The program is running well enough that the all important number crunching part now works. This number crunching is the part which tells the user how the material will react to certain conditions, or whether or not that particular composite will have a practical use. The program will be modified to make it look neater and to allow it to be more user-friendly. Several graphing subroutines remain to be modified. These subroutines will allow the user to physically view the results of a hypothetical test which was already numerically run by using the other options. All the options on the aforementioned Main Menu are working, save the graphing options, except for one error. Once this error is fixed, the entire program will be working in the same capacity that it was on the IBM compatible machine. This was the original task some two and one-half years ago. The experience gained throughout the two plus years of working on this project has been invaluable. The skill of problem solving is the basic tool of every engineer. This project has certainly helped to develop those skills. When this project is finished this software package will aid in the ever rapidly developing field of composites.

Consolidation of Computer Program NDSANDS

Task Order No. 106a
Student Support Program

Anthony D. May
Wright State University

31 December 1992

Guiding Engineer
Dr. G.P. Tandon
WL/MLBM

ACKNOWLEDGMENTS

Thanks go out to Dr. G. P. Tandon, whose guidance has been invaluable, to the technical initiator Nicholas J. Pagano, and to the many other generous scientists at the lab who have made this task an enjoyable experience.

TASK ASSIGNMENT

The purpose of this task is to create a consolidated, user friendly version of the "NDSANDS" program on a Macintosh computer. The "NDSANDS" program, whose title stands for N Directional Stiffness AND Strength, can be used to carry out complex analyses of composite materials. The program allows the user to create and modify a constituent material library. A composite library, with composites created by combining various constituent materials, is also maintained by the program. The primary function of the program is then to analyze these user created composites through mathematical modeling. Stresses, strains, displacements and strength ratios can each be calculated. These quantities are displayed in tabular format. One of the remaining goals of the task is to display this information in the form of graphs where the variables to be plotted against each other can be chosen specifically by the user. "NDSANDS" is capable of analyzing up to five composites simultaneously. Another useful feature of the "NDSANDS" program is the parametric study capability which it provides. Users are given the ability to alter any one of the variables which define the composite whether it be a property of a constituent material or an element of the geometric makeup of the composite. This option allows the user to quickly determine the effects of various modifications to a composite.

"NDSANDS" was originally developed in the FORTRAN language for use on IBM compatible computers. This task involves the conversion of this program to a Macintosh style application which utilizes the graphical environment present in the Macintosh computers. Nearly all the "number crunching" sections are identical to those in the original program. Modifications present in the current version of "NDSANDS" involve the manner of input, the interactive capabilities and the display of the results. One example of the differences between the IBM and Macintosh versions is that all information for display in the Macintosh version requires a window to be open and current, the position of the output in the window to be given, and the output to then be written to the screen. The input of data also becomes more complicated in the Macintosh version because all data must be entered in boxes call editfields. These editfields only read data in character form. Because of this additional subroutines are used to convert the strings to real number values where numerical input is needed. These editfields were the source of numerous bugs which caused serious malfunctions in the program. An editfield subroutine had been written to handle this type of input in a generalized manner. The problem arose because the variables used to store the data held in the editfields were not consistently declared. This caused unpredictable values to appear in variables which were not even involved in the input of the data using the editfields. The values which were being altered caused the program to loop incorrectly in some instances. As is the case with many programming bugs the solution to the problem was not very difficult once the cause of the problem was correctly diagnosed. Careful checking of the variable declarations eliminated most of these errors.

Work on this task included the familiarization with both the Macintosh computer and the "NDSANDS" program itself. Considering that the program was broken down into seventeen sections and was over eighty pages long this was no small task. The format of the data storage was reworked to represent the properties of the constituent materials in more appropriate units of measurement. This required the inclusion of factors in portions of the code to compensate for the change of units. All new screens to add, change and delete the constituent materials were written. In the deletion sections the data to be deleted was made to redisplay and confirmation of the deletion was requested. In the parametric study portion of the program, the section of code for changing geometry values that was not being accessed, was requested as input and made to execute. A number of other changes was incorporated as well. Many were little more than readjusting the position of some portion of text while others involved more analytic thinking to track down the problems and devise ways to correct them. The final section to be completed involves the plotting of the results of the composite analysis.

Synthesis and Characterization of Polymers

Task Order No. 107
Student Support Program

Colin McHugh
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31 December 1992

Guiding Engineer
Dr. Robert Crane
WL/MLPJ

ACKNOWLEDGMENTS

I need to recognize Dr. Tim Bunning, Dr. L. V. Natarajan, Dr. Robert Crane, Dr. Tom Cooper, Kelly Menster, Keith Obermeier, Jack Henes and Sungmee Kim for their assistance with my research at WL/MLPJ.

TASK ASSIGNMENT

This Task Assignment involved the synthesis and characterization of cyclic siloxane-based liquid crystalline compounds for use in optical data storage materials and optical notch materials. Thermal characteristics of the liquid crystalline compounds were investigated to determine trends in varying the lengths of the leader groups and product usefulness.

The leader group, 5-Penteneoxybenzoic acid, was synthesized according to the methods reported by Apfel and Bunning. Attachment of the leader group to either cholesterol or hydroxybiphenyl was performed using a Dicyclohexylcarbodiimide (DCCI) coupling reaction, with dimethylaminopyridine as catalyst, as found in Dr. Bunning's dissertation. A suitable solvent system of 50/50 methanol:toluene was used to recrystalize the product for use in a tetramethylhydro - and pentamethylhydrosilation reaction to form either Cholesterol-4- or Biphenyl-4-Penteneoxybenzoate (C4PB or B4PB). These hydrosilation reactions were carried out in toluene, under argon, at 90-100 C. Reaction times for the hydrosilation reactions varied from 48-70 hrs. Reaction completion was determined using Fourier Transform Infrared spectroscopy (FTIR), to see the disappearance of the Si-H band at $2160cm^{-1}$, Nuclear Magnetic Resonance (NMR), to determine quality, Thin Layer Chromotography (TLC), and elemental analysis to determine purity.

Differential scanning calorimetry was performed on the siloxane-based liquid crystals, using a Perkin-Elmer DSC-2C and a Mettler FP82HT hot stage to determine crystalline phases.

To understand better the packing behavior of the nematic phase of a pentamethylcyclosiloxane ring, containing mesogens of C4PB or B4PB, densitometry was performed. The X-Ray diffraction patterns of the meridional and equatorial reflections at different temperatures and frequencies were examined using an Image Quantifying package on the Molecular Dynamics Personal Densitometer, and on a Scandig-3 Densitometer utilizing a Digital VAX program called Mordor.

The alignment kinetics of a class of siloxane-based liquid crystals, under an electric field, were probed using

X-Ray radiation from CHESS (Cornell High Energy Synchrotron Source). Densitometry was performed to report on the kinetics.

The alignment times as a function of different frequencies, (1-10000 HZ), and temperatures, for a class of siloxane-based liquid crystals, were examined for structurally similar compounds. Alignment times rose exponentially with decreasing temperatures, but were relatively unaffected by frequency (Abstract submitted Nov. 92 for APS).

The knowledge obtained from our work, we hope, will assist in future siloxane-based projects.

Processing Materials of Molecular Composites

Task Order No. 108
Student Support Program

Henry Derstine
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31 December 1992

Guiding Engineer
Dr. Robert Spry
WL/MLBP

ACKNOWLEDGMENTS

I would like to acknowledge and thank Dr. Robert Spry and Dr. S. J. Bai for their help and guidance. Also I would like to acknowledge the entire polymer branch for their assistance and instruction.

TASK ASSIGNMENT

During this Task the duties involved measuring the conductivity of polymers as well as other minor tasks. The procedure followed was the conductivity tests were run first, then other tasks were done.

Dr. Bai presented a sample that would undergo testing and be prepared for measurement in Keithley S110 test system. This preparation was done by cutting a test sample, measure the effective length, place the sample in the test module, mount the module in the system and then begin the computer sequence.

A sample was cut into a strip from the larger film approximately 2 cm long by .2 cm wide using a razor blade and a straight edge. The precise dimensions for the calculation of the effective length were measured under the microscope. The effective length is equivalent to the (Width*Thickness) /Length (between the contacts). The sample was then placed in a Teflon holder that was connected to the S110 circuit. The program that controlled the current and did the calculations for the resistivity of the sample was then run. The effective length of the sample needed to be entered into the computer to allow the resistivity to be calculated. Other information was also entered for sample identification and record keeping.

Checking for a vacuum leak was also done. A modification to the VanderPhal test system that allowed clean Nitrogen to purge the sample cavity produced a vacuum leak somewhere in the system. The first step to find the leak was to check all the joints in the system of hoses to ensure a tight fit. The joints appeared sound, so a more careful check of the entire system was done. The technique used involved the use of acetone to find minor leaks. Acetone will evaporate quickly as air rushes through a hole or crack in the system. One leak was found at the valve for the Nitrogen tank and was sealed using Teflon tape. The other leaks were minor compared to the valve leak. These appeared in the joints of the hoses and were repaired using vacuum grease.

Another experiment was developed to go hand-in-hand with the conductivity experiment. This was a depletion experiment, to determine if the polymer was an ionic or electronic conductor. The first method developed was to use the S110 as a constant current and to measure the voltage as a function of time. It was felt that if the voltage across the polymer varied with time, while the current was held constant, the conductivity was due to an ionic effect. However, this method was not the traditional method of doing a depletion experiment. The constant current method was done in lieu of a constant voltage source. The system appeared to be valid, but to eliminate doubts a constant voltage source, a Keithley 617, was dedicated to this experiment. The experiment was now established so that the 617 produced a constant voltage across the samples. If the samples were ionic, some kind of decay would appear in the current through the samples as a function of time.

Some time was spent on researching equipment to be purchased for current and future experiments. This was done by completing a 1348-6 form. The information required on this form depends on the expense of the equipment to be purchased. Higher priced equipment required more information compared to lower priced equipment. However, some information is required of each; unit price, manufacture's name, supplier's name, description of equipment, reason for purchase, photocopy of the page in which item is found and priority of item. The form is then copied and sent to requisition personnel.

This term much information was learned and discovered. Some of the conductivity results that seem to be so promising, should be looked at again, after a depletion test is run on the sample. A simple tool and technique such as Teflon tape and acetone, can be used to find the most problematic leaks in a vacuum system.

Quasi - Particle Polymeric Systems Based on Air Force Poly (Benzobisthiazoles) (PBX)

Task Order No. 109
Student Support Program

Melissa Gonet'
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31 December 1992

Guiding Engineer
Dr. Loon Seng Tan
WL/MLBP

ACKNOWLEDGMENTS

Thanks to Dr. L. S. Tan and S. Simko who helped me in my task assignment.

TASK ASSIGNMENT

During these past six months at Wright Patterson Air Force Base I have been doing a large number of melting points and other analysis. The Infrared Radiation Machine (IT) has been used for Dr. L. S. Tan and S. Simko to speed up their work on current synthesis projects. I also used the Nuclear Magnetic Resonance Spectrum machine (NMR) on occasion.

The use of the melting points recently finished are to help S. Simko in her analysis by distinguishing any inorganic compounds in her sample to dry it out before it will melt. The IR's ran help to determine the location of functional groups in a new sample. This verifies a compound's structure before taking it further in the processing stage. Additionally, the NMR results are used to determine the placement of hydrogen atoms in the structure of a compound. These can be tricky to do because of the sensitivity of the machine. Often many compounds do not "graph" as would be expected because of various conditions not always due to the compound's structure itself.

Another major aspect of the work completed on Base is done on the Macintosh system. This allows the work of synthesis and analysis to be visually displayed for project presentations here on Base and abroad. Currently, a project is being compiled covering research of Polybenzazole (PBX) polymers and some of their non-linear properties. The PBX polymers have been under study in this laboratory for some time. When it is time for a presentation, Dr. L. S. Tan writes rough drafts of the information, and they are then typed and "drawn" as directed.

The winter months on base are when orders for new government equipment go through. This is another project that this position is actively involved with. After being given a preliminary list of what equipment needs ordered, they are written out and itemized to keep track of funds and quantities. When the materials ordered arrive, they must be marked received and given to the proper personnel.

Some less important areas of this position include logging chemicals when received and when empty or changing locations. Recently the branch has obtained a new system of inventory upkeep, and I have been actively involved in learning to utilize the system and help others do the same. The system is entirely computerized involving the use of bar codes on chemical jars to track usage, waste, and supply availability.

Another responsibility is doing the lab dishes on a regular basis so that glassware supplies are readily available. This job requires familiarity with different chemicals and solvents that will rinse them the best. For example, the use of acetone as a rinse removes traces of most chemicals, but sometimes when cleaning a filtering funnel, chromic acid must be used to dissolve sample residue.

As noted several various responsibilities make up this placement. Each of them works together to give a sound impression about what working in a chemistry laboratory is like. Although the goal is synthesis of a product that can be sent for future development, many diverging roads exist between the start and finish of a project. The paperwork and dishes sometimes stand in the way of research, but they are necessary aspects of the position. Synthesis of a new polymer or monomer is not difficult, but creating ones that have the attributes necessary to move forward in the stage of processing is. The analytical procedures done at this level are primitive but undeniable for verification of a structure and hopefully a green light to continue working with that particular sample.

Heterocylic Intermolecular Charge-Transfer Complexes

Task Order No. 110
Student Support Program

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31 December 1992

Guiding Engineer
Mr. Bruce Reinhardt
WL/MLBP

ACKNOWLEDGMENTS

I wish to express my gratitude and appreciation for the knowledge and guidance Mr. Reinhardt has given to me. Working under his wisdom is an enjoyable experience. I also thank Gibby Dombrowki and Marilyn Unroe for their support during this task. They are both knowledgeable scientists and enjoyable people to work with.

TASK ASSIGNMENT

Work during this task focused on the organic synthesis end of NLO materials development. The object of the task was synthesis and preliminary characterization of novel charge-transfer molecules for evaluation as potential third-order NLO materials. However, early NLO data on these charge-transfer compounds suggested that they would not exhibit sufficient NLO activity. Therefore, the task diverged from the initial objective to an objective of streamlining heptamer and pentamer synthesis procedures.

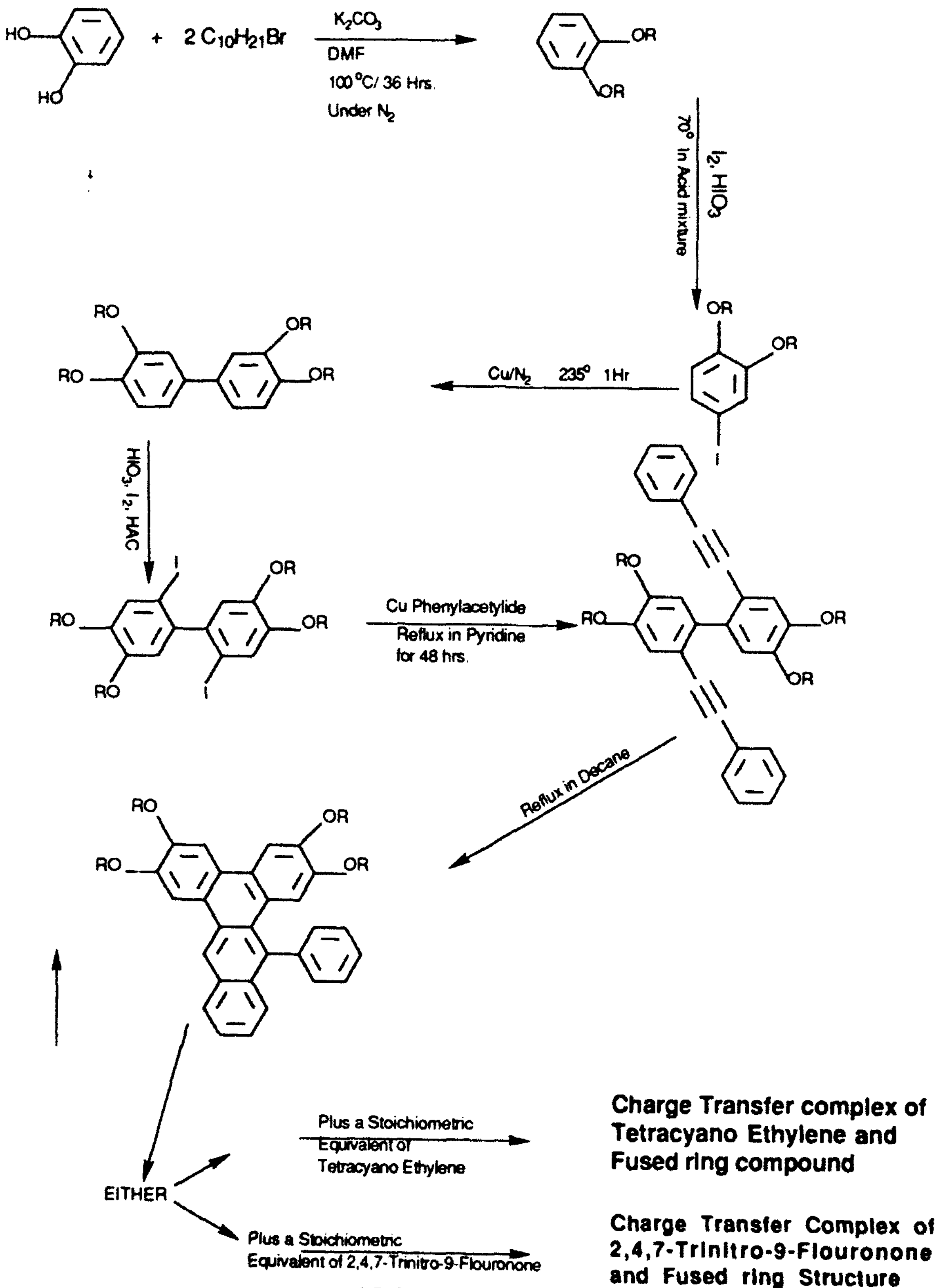
Basic task duties were as follows. Mr. Reinhardt decided what molecules he would like to synthesize for NLO test. Next, he sent the student to the library to research synthesis procedures. After obtaining the procedures, the student searched the Material Lab's computer inventory network and located all necessary reactants. The student then completed the reaction procedures and isolated the products. Typical isolation procedures included extractions, column chromatography, gravity and vacuum filtrations and recrystallizations. After isolating the product, the student characterized the product by standard methods such as infrared spectroscopy, nuclear magnetic resonance, melting point, thin layer chromatography and viscosity. Finally, the student prepared the finished product for elemental analysis, mass spectroscopy and NLO characterization.

Other task duties include basic lab maintenance, calibration of the lab's NMR and hazardous waste documentation. A number of significant new federal and state EPA regulations have recently been enacted. As a result, the student spent more of his time learning these new procedures and recording hazardous waste data.

The task contained two major sections. The first section investigated development of novel charge transfer complexes and the second section scrutinized the possibilities of streamlining heptamer and pentamer synthesis routes. In the first section two charge-transfer complexes were synthesized. Reaction routes and the charge transfer complexes for each molecule are outlined in figure one. Since these charge transfer complexes yielded relatively low third order NLO activity data, the majority of the task time was spent improving heptamer and pentamer synthesis procedures. Scientists have extensively studied the NLO activities of the heptamer and pentamer structures and have comprehensively documented the results. As a result, NLO personnel often use these compounds for NLO instrumentation calibration. However, the typical synthesis routes produced a very low yield and accordingly the price of production was relatively high. Therefore, streamlining this procedure is cost effective. Attempts at streamlining this procedure produced yields almost three times higher than previous procedures. Streamlined procedure used is located in Figure two. The Pentamer procedure is identical to the Heptamer procedure with the one exception of using a different starting material.

This task concentrated on two primary projects; synthesis of novel charge transfer complexes and streamlining of heptamer and pentamer compound synthesis procedures. The produced charge transfer complexed did not produce anticipated third-order NLO activity. The reaction sequences of the heptamer and pentamer compounds were successfully streamlined with nearly a 300% increase in final yields as compared to our old procedure. My current lab research has expanded and deepened my organic chemistry knowledge and capabilities and I look forward to continued learning in this field under the guidance of Mr. Reinhardt.

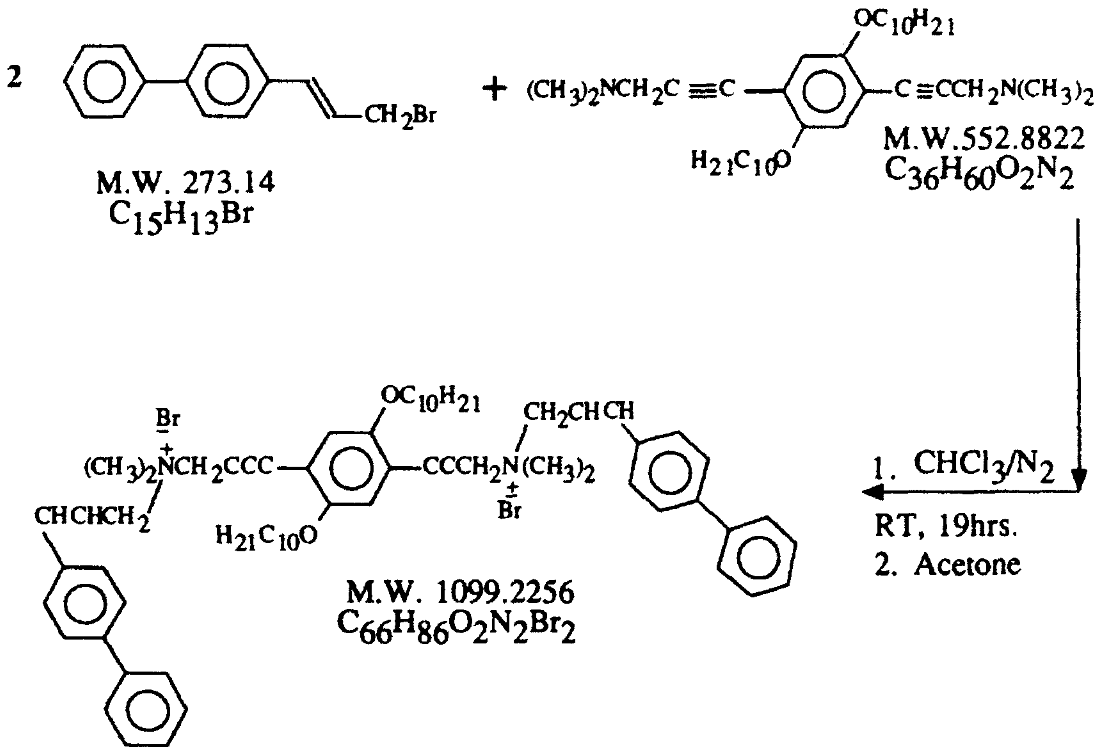
Figure one: Reaction route for charge transfer complex of fused ring compound and Tetracyano Ethylene



*OR = $OC_{10}H_{21}$

Figure Two: Heptamer Preparation

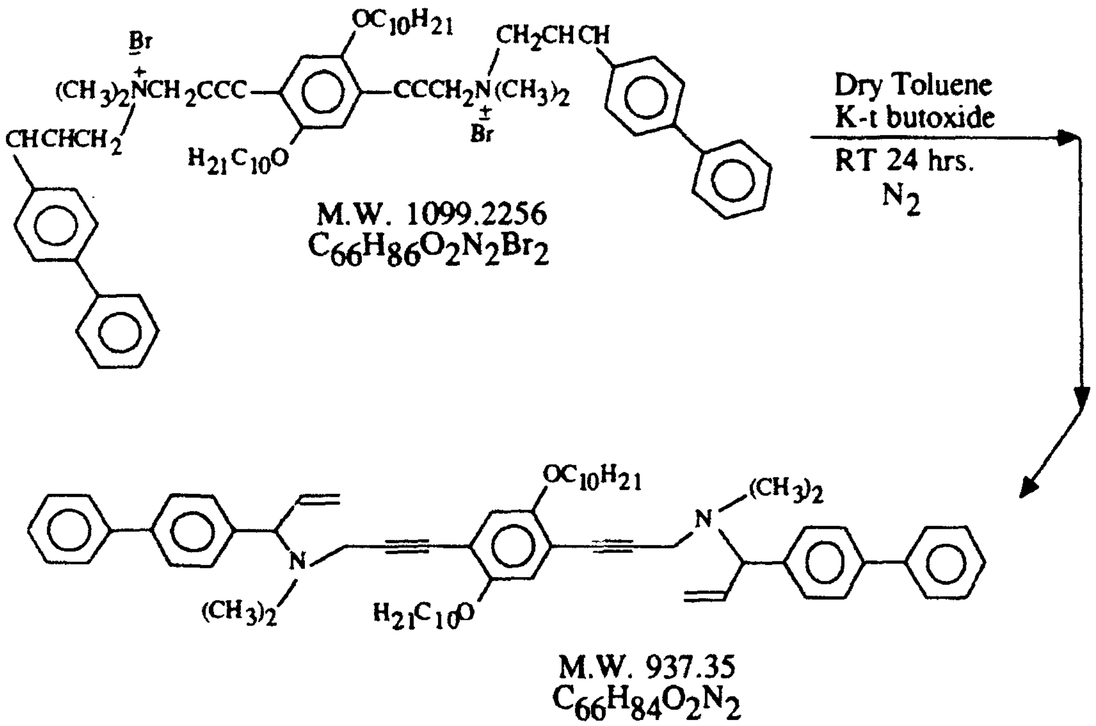
Heptamer Preparation
Step One



Procedure: A 500 ml 3NRBF was fitted with a magnetic stirrer, addition funnel, and a Nitrogen inlet line. The $C_{15}H_{13}Br$ (.04 moles, 10.93g)was added with 100ml chloroform. The $C_{26}H_{58}O_2N_2$ (.02 moles, 11.06g) was slowly added through the addition funnel over a period of one hour along with a second 100ml of chloroform. The reaction mixture stirred at room temperature for 24 hours. The chloroform was removed on the roto-vap and the resulting white solid was stirred with 100 ml of reagent grade acetone. The white solid was collected on a buchner funnel. Yields were typically in the 90% range.

Figure two: Heptamer Preparation, Continued.

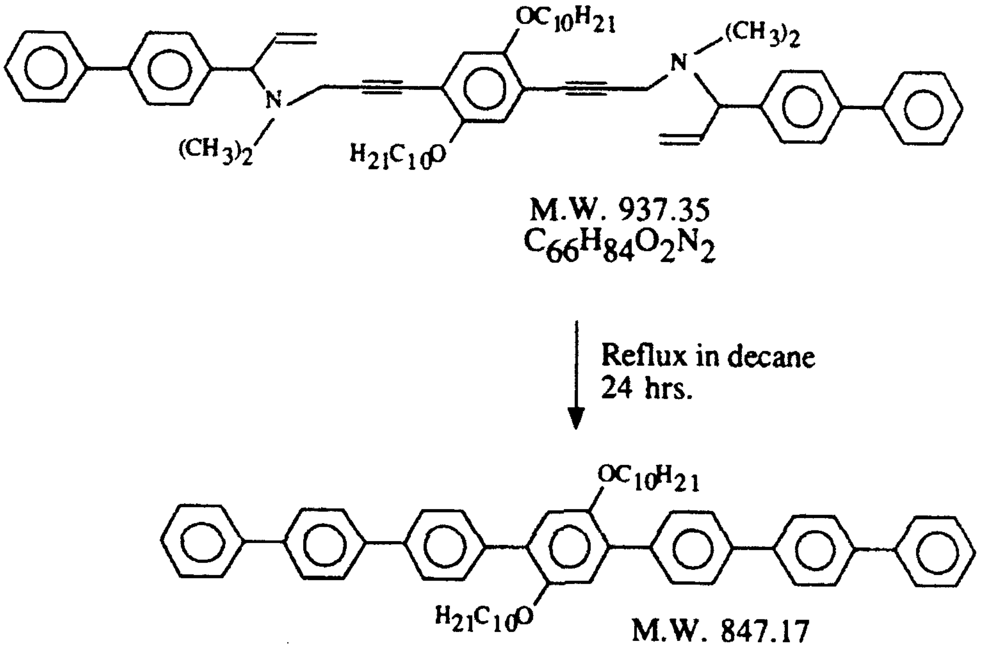
Heptamer Preparation
Step Two



Procedure: The $C_{66}H_{86}O_2N_2Br_2$ salt was placed in a 500ml 3NRBF fitted with a magnetic stirrer and gas inlet. 300ml of dry toluene was added. The reaction vessel was degassed using a firestone valve connected to a nitrogen line and the house vacuum system. K-t-butoxide was added and the reaction was allowed to proceed overnight at room temperature. After 24 hours, the toluene was removed under vacuum and 18.35g of impure product was recovered.

Figure two: Heptamer Preparation, Continued.

Heptamer Preparation
Step Three



M.W. 937.35
$C_{66}H_{84}O_2N_2$

Reflux in decane
24 hrs.

M.W. 847.17
$C_{62}H_{70}O_2$
Melting Point 190.3 - 191.6

Procedure: 18.35 g of impure C66H84O2N2 was placed in a 200ml RBF with 50ml of decane. Flask was fitted with a stir bar, condenser, and a heating mantle and then refluxed for 48 hrs. Product was poured into a 500 ml beaker while the product was still hot. After the product cooled 300 ml of methylene chloride was added and the product fell out of the methylene chloride. Product was filtered and recrystallized from 350 ml cyclohexane. A 35% yield was obtained from the impure starting material.

Mechanical Properties of Metal Matrix Composites

Task Order No. 111
Student Support Program

Alisha Hutson
The Ohio State University

31 December 1992

Guiding Engineer
Mr. Jay Jira
WL/MLLN

ACKNOWLEDGMENTS

I would like to thank Mr. Jay Jira for his patience and thorough explanations. I also would like to thank the rest of the people working in building 655. They were all quite tolerant.

TASK ASSIGNMENT

The work completed during this contract period is much the same as the last one. The necessary data reduction is completed using the MicroVAX in building 655 with a Macintosh fxII. Duties now also include digitizing the optical data for each test, having blow-ups made of the most vital pictures in each series, rereducing data to give a more meaningful representation of the fatigue data for each test, and other miscellaneous tasks.

Digitizing the optical data for each fatigue test requires looking at each picture taken during a test and measuring how long the specimen crack appears in each. This process has been automated so that each measurement takes only a few seconds. The data is read and stored by a TEK computer, and then is transferred to the MicroVAX for reduction.

The reduction step requires running the raw data taken by the computer during the test and the digitized optical data, and running both of them through a program that takes the pertinent information from both files and merges it into one file with a standard header and column labels. This file is then run through another program that generates a data set that represents the data without all the scatter. The two new files are then transferred to the Macintosh and used to generate plots that will tell the engineer most of what he needs to know about the test at a glance, i.e. whether the pictures were bad, whether there was a lot of microcracking during the test, etc.

If the optical data doesn't match the data taken during the test within about ten percent (considered a reasonable limit for error), it is assumed that some error has occurred with the data taken during the test. The data is rereduced under this assumption so it agrees with the optical in both slope and magnitude. We confirm that the optical is more accurate than the Electropotential data by taking two more measurements. The final crack length is measured from the blow-up print we have made. The specimens are tested at high temperature and the final crack length is measured from the surface of the specimen. Oxidation that occurs while the specimen is cooling looks different from that which occurs while the specimen is being tested. So by looking for a change in the appearance of the oxidation under a microscope, one can measure how long the crack in the specimen was just before it broke.

There are several other tasks assigned that allow more qualitative analysis of each specimen. Recently the government organization that prints the blow-ups have developed a method of enhancing photos to allow certain features to become visible that might be almost invisible without it. Since each photo is different, the staff that does the printing requires the customer to be present during the enhancement process, which is done by computer on Adobe Photoshop. (This program facilitates scanning, editing and printing of high resolution graphics.) By observation, it was learned how to use this program for printing purposes.

Jay also wanted to do some observation of the specimens under a Scanning Electron Microscope (SEM). Arrangements were made to have assistance in the use of the SEM until enough experience was gained to work on alone. I have now logged several hours of SEM time and experience.

During the past few months I have continued to learn new skills even though I am still working in the same lab and have the same task description. I have enjoyed working with Mr. Jira in the Materials Lab, and hope the next contract will be as educational.

☆U.S. Government Printing Office:1993—750-113/80311