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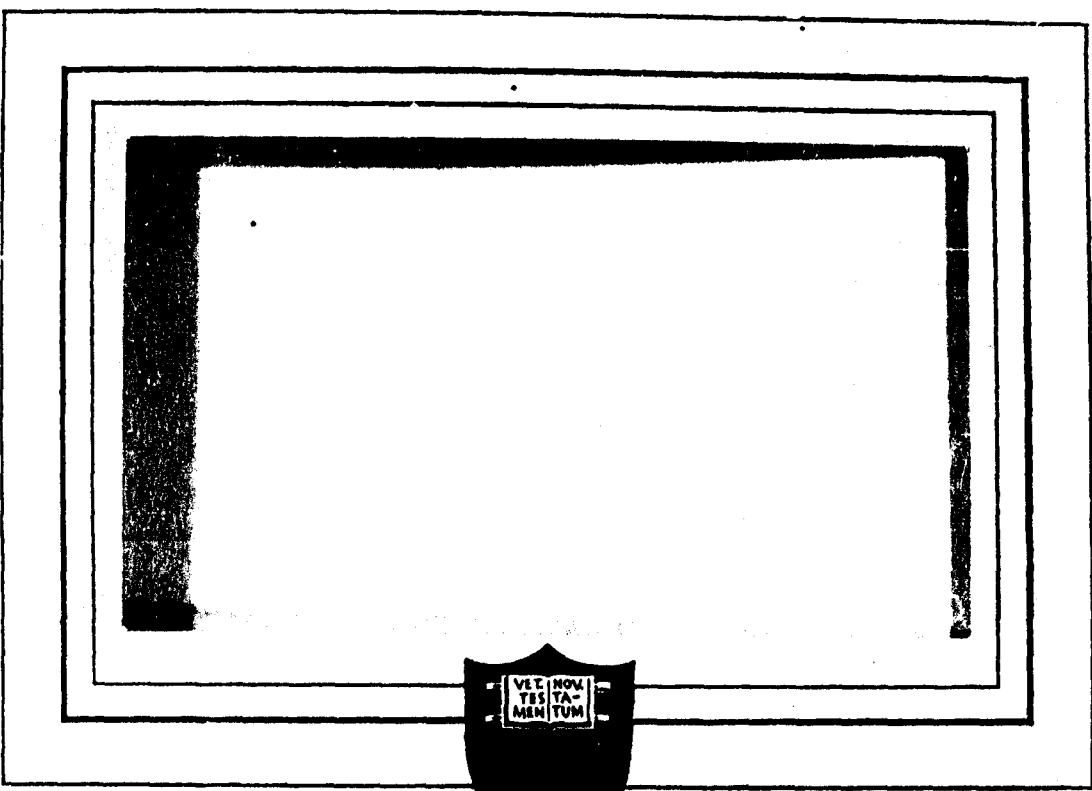


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PRINCETON UNIVERSITY
PLASTICS LABORATORY

PLASTICS LABORATORY REPORT

Number 66

Interim Progress Report

31 March 1963

Contract No. DA-31-124 ARO(D)21

The research reported here was partly
supported by the Army, Navy and
Sandia Corporation under ARO(D)21
Contract; O.N.R. Project NR 356-375

Report prepared by:
L. F. Rahm
Project Director

Contract No. DA-31-124
ARO (D)-21

31 March 1963

PLASTICS LABORATORY REPORT

Number 66

Interim Progress Report

ABSTRACT

This Interim Report covers the period
15 July 1962 to 31 March 1963 and presents:

	Page
1:0 Permanent Contract Data	1
2:0 Personnel	2
3:0 Facilities	3
4:0 Meetings	3
5:0 Project Status Reports	4
5:1 Chemistry Section	5
5:2 Dielectrics Section	7
5:3 Mechanical Section	9
6:0 Reports, Publications, Papers	10

1:0 PERMANENT CONTRACT DATA

1:1 The above referenced contract is dated 13 July 1962
and covers the period 15 July 1962 to 15 March 1963
with funds from ARO(D) and Sandia Corp.

1:2 Modification No. 1 dated 24 October 1962 extends the
termination date of the contract to 31 July 1963 with
additional funds from O.N.R.

2:0

PERSONNEL

2:1

Classification

Project Director

L. F. Rahm

Group Leaders

Chemistry - D. S. Trifan

Dielectrics - C. Capps

Mechanical - B. Maxwell

Research Associates - Full Time

R. F. Reinisch

C. Capps

Ph. D. Candidates

R. A. Sheldon*

R. N. Olsen*

C. G. Gogas*

T. W. Huseby*

R. P. Chartoff*

M.Sc. Candidates

N. J. Curto*

J. A. Krebs*

A. C. Hart*

D. P. Thomas*

W. V. Uwakaneme

* On Fellowships; No charge to contract;

Technician

W. Rightmire

Secretary

Mrs. Joyce Cohen

2:2

Allocation by Tasks

Chemistry Group

Total

Trifan, Reinisch, Sheldon, Olsen (1/2)

3-1/2

Dielectrics Group

Capps, Krebs (1/2), Thomas (1/2)

2

Mechanical Group

Maxwell (1/2), Gogas (1/2) Hart (1/2),

Chartoff (1/2), Huseby (1/2), Uwakaneme (1/2) 3

Unallocated Personnel

Rahm (1/2), Rightmire (1/2), Mrs. Cohen (1/2)

1-1/2

Full time equivalent

10

Actual Personnel

16

2:3

Personnel Status

Five former M.Sc. students were readmitted as Ph.D. candidates in September 1962, Gogas, Olsen, Chartoff, Sheldon and Huseby. Four new M.Sc. candidates Curto, Hart, Krebs, Uwakaneme were admitted in September 1962 and one, Thomas, in February 1963 making a total graduate student population of 10 as compared to 12 one year ago. Contract budget permits support of only one of these students; all others are on Fellowships.

Professor Maxwell was appointed as Assistant Dean of Engineering effective 1 February 1963. He will be available for research on a quarter-time basis on the contract and will continue as Group Leader of the Mechanical Section.

3:0

FACILITIES

A LiF prism and adapter for a Perkin-Elmer I.R. machine, originally purchased with Signal Corps funds in 1957 was returned to the Signal Corps 18 March. An old high-pressure hydrogenation bomb was donated to Princeton. This clears the Plastics Laboratory of all government owned or government furnished equipment.

More important, after three years of planning and building at a cost of eight-and-one-half million dollars, the new Engineering Quadrangle was completed during the summer of 1962 and was occupied in August. In this extensive facility the Plastics Program occupies the 4th floor of Unit D, Duffield Hall, otherwise the Mechanical Engineering Unit, adjoining both Chemical Engineering, Unit A, and Electrical Engineering Unit B. Both the office and laboratory space for Plastics has been increased by about 50% while the services in being modern, are greatly improved over the former laboratory, in safety, appearance, utility and maintenance. This permits 2 modules for Mechanical Studies, 5 modules for Processing Laboratory, 3 modules for Dielectrics and 5 modules for Chemistry. Furthermore, other activities in materials research in the Engineering School have been located in the same or adjacent units with the expectation that materials as a whole may receive more attention in the future.

4:0

MEETINGS

4:1 A meeting of the Services Technical Committee was held at Princeton 1 November 1962.

4:1 (cont.) Present for Meeting:

E. M. Beekman, Signal Corps.	C. Cappas, Princeton
P. S. Greer, ARO (D)	B. Maxwell, Princeton
H. Fox, O.N.R.	D. S. Trifan, Princeton
A. J. Quant, Sandia Corp.	L. F. Rahm, Princeton

Professor Rahm reported that in extending the contract through Modification No.1 the termination date had been accidentally made 31 July 1963, instead of 15 July 1962 as expected. With an initially tight budget and an approved overhead rate now higher than that used in estimating, there is some doubt that the available funds will cover the extra half-month but every attempt will be made to meet the extended date with the original budget.

ARO (D) announced that it would no longer consider supporting a blanket contract in chemistry, dielectrics and mechanical studies such as the Plastics Program has had since 1946. More specifically the Chemistry Division of ARO (D) is not prepared to support work here beyond \$20,000 per year, although this does not preclude other divisions from supporting similar individual contracts if they are so disposed. As a consequence of this decision Trifan is submitting a proposal for his future work to ARO (D), A.E.C. and NASA. Rahm is submitting a proposal in dielectrics studies to O.N.R., NASA and Sandia Corporation. Maxwell is filing no proposal at this time.

Mr. Greer requested an Interim Progress Report dated 31 March 1963, and a Final Report due one month after termination date of 31 July 1963. Written instructions since the meeting request that (with some additions and changes) the former distribution list be used for Interim, Final and any Technical Reports.

The next meeting was tentatively scheduled for Thursday, 20 June 1963 at Princeton.

The group leaders Maxwell, Cappas and Trifan then reported on the progress of the work in their respective sections; See 5:3, 5:2, 5:1.

5:0

PROJECT STATUS REPORTS

5:1

Chemistry Section

D. S. Trifan

As part of a general program of research in these laboratories involving study of the polymerization reactions of nonconjugated di- and multi-olefinic monomers leading to the formation of polymer chains comprising methylene-linked monocyclic and multicyclic ring structures, considerable attention has been given during this period to the polymerization behavior of 1,4 pentadiene. Based on some earlier preliminary results obtained here on this monomer in which a large soluble fraction is produced with rather low residual unsaturation, a new polymerization mechanism was proposed which accounted for both the < 1 residual unsaturation per monomer unit and absence of any appreciable amount strained four-member ring content as evidence by I-R analysis. In view of the small tendency toward intramolecular propagation successively alternating with intermolecular propagation to give such a sterically strained structure, it was suggested that a second intermolecular propagation immediately occurs subsequent to the first, leaving a pendant vinyl group which then participates in the first of two successive intramolecular propagations to give methylene-linked 3,3,1-bicyclo-nonane polymer chain structure. Such a sequence of inter- and intramolecular propagation steps has since been independently suggested by Chang and Price for the free radical polymerization of N,N-divinylaniline, an analogous hetero 1,4-diolefinic monomer.

In the course of the present work, the effects of certain polymerization variables have been studied for this interesting Ziegler-catalyzed polymerization of 1,4-pentadiene. For the soluble fractions from polymerizations at 0.2 to 0.006 ml. monomer per ml. heptane solution, utilizing AlEt_3 - TiCl_3 mostly at Al/Ti molar ratios of 1.5, the residual β external double bond content falls from a value of 0.33 double bonds per monomer unit at the higher monomer concentrations to 0.16 for the lower monomer concentration runs. Quantitative I-R analyses are employed in these determinations. A complicating aspects of this polymerization system is the fact that internal unsaturation is also observed, varying with monomer concentration from 0.22 to 0.06 double bonds per monomer unit. For the higher monomer concentrations studied where monomer: catalyst ratios are in the normal range substantially above unit, the residual internal unsaturation is inversely proportional to the monomer concentration.

5:1 (cont.)

The internal unsaturation, not directly expected nor accountable in terms of available polymerization paths of 1,4-pentadiene, has been established by VPC methods to be due to partial isomerization of the initially pure monomer under prevailing reaction conditions to 1,3-pentadiene, which copolymerizes via 1,4-addition mode with these Ziegler catalysts. These internal double bonds are almost entirely of trans-configuration. The nature of the Ziegler catalyst as a variable in the concurrent isomerization reaction was shown by comparison of one the above runs with an AlEt_3 - TiCl_4 -catalyzed polymerization ($\text{Al/Ti} = 1.5$) under otherwise identical reaction conditions. Internal double bond content of the product polymer was reduced from 0.2 to 0.06. Other systems are being similarly compared with respect to these variables.

In accord with the above mechanism for 1,4-pentadiene polymerization, the polymer products have a large soluble fraction (ca. 50% in heptane at room temperature in 24 hours). The insoluble material has an I-R spectrum qualitatively similar to the soluble fraction and is probably lightly crosslinked through the residual isolated external double bonds, also present in the corresponding soluble fraction, as a result of some competitive intermolecular propagation intervening in the above sequence of inter- and intramolecular steps. The polymerizations were conducted at 70°C. with system and catalyst concentrations adjusted to obtain a roughly constant yield of polymer with conversions between 40 and 45%. The data suggest a first order reaction with respect to both monomer and catalyst for the system AlEt_3 - TiCl_4 at $\text{Al/Ti} = 1.5$, with $k = 56 \text{ hr}^{-1} \text{ ml}^{-1} \pm 10\%$ over a range of monomer concentrations varying by a factor of 30 and catalyst concentration by a factor of 3.

In the area of study concerned with the synthesis, structure, etc. of the double-stranded hydrocarbon polymer, fused 1,2,4,5-polycyclohexane, additional syntheses of this polymer have been carried out to afford material for additional planned dehydrogenation studies. A useful improvement in the synthesis of this polymer has been introduced in which the total amount of crude 1,2-polybutadiene, obtained from the initial butadiene polymerization and containing a large gelatinous fraction due to crosslinked product, is subjected to the next intramolecular polymerization step without prior separation and removal of this swollen fraction. Formerly a difficult separation representing ca. 50% of the total polymer weight, the present modification

5:1 (cont.)

conveniently provides a final product of superior yield with actual crosslinked content of only $< 5\%$, easily removed at this stage. I-R spectra of the polymer obtained from both procedures are identical. Other aspects of catalyst variation on the second polymerization step and study of corresponding effects on the detailed structure of the doubled stranded chain are also planned.

A new pressure bomb is in preparation for more extended aromatization studies of the above linear fused cyclohexane polymer. Current efforts toward milder conditions and methods for effecting dehydrogenation and aromatization of this polymer are in progress.

5:2

Dielectrics Section *C. Cappas*

The Dielectrics Section is pursuing, on several fronts, studies into the physical and electrical properties of certain types of conducting organic polymers. An investigation of the transport properties of polymers from π -excessive and π -deficient N,O-heteroaromatic monomers has been started. All π -deficient heterocycles contain nitrogen and most of them are six-membered rings. The nitrogen atom attracts electrons from the π -double layer, and this causes a strong polarization of the molecule, which in turn causes the ring carbon atoms to have low electron densities. On the other hand, π -excessive N-heteroaromatics have at least one five-membered ring. The contribution of electrons from the nitrogen atoms of the ring to the π -layer makes it highly polar, with high electron densities on the carbon atoms. The exploration of these compounds in the polymeric materials was begun to determine their effect on electrical conduction. It was realized that they would not act as electron donors or acceptors in the strict sense of the word, but that free radicals entrapped in the chain might behave in this way. One class of compounds comprised π -excessive and π -deficient N-heterocyclic molecules; a second included π -deficient N,O-heterocyclics; and a third was synthesized from π -excessive N-heterocyclics and certain aromatic molecules. With the exception of two, pyrrole with benzoquinone

$$(\rho_{25} = 8.12 \times 10^5 \text{ ohm-cm})$$

and pyrrole with α -tetralone

$$(\rho_{25} = 1.48 \times 10^8 \text{ ohm-cm}),$$

the remainder did not have interesting enough properties to warrant extensive investigation.

Recent work in this laboratory has indicated that certain of the wholly aromatic and highly conjugated and crosslinked polymers exist in the form of stable biradicals. It was also found that certain structural factors of the monomer influence the properties of the polymer. One of these, conjugation, is being examined in the system of polyphenylacetylene. The monomer can be thermally polymerized and, depending on the polymerization temperature, soluble polymers can be obtained that exhibit thermal stability. These polymers give sharp electron paramagnetic resonance spectra, indicating unpaired electrons delocalized over the conjugated chain. It is interesting and significant that as the polymerization temperature is raised, spin density per gram increases, the polymer becomes less soluble, but the resistivity remains approximately constant at about 1.3×10^{13} ohm-cm. Some Russian workers¹ have shown that the thermal polymerization of phenylacetylene is initiated by a bimolecular reaction leading to the formation of biradical dimers. One possesses the conjugation of free electrons and benzene rings as well as π -electron conjugation in the chain. In the other, free electron conjugation occurs only on one end of the radical. The properties of the polyphenylacetylene polymers are being compared with those of another class of conjugated polymers.

A different approach that is presently being tested is to start with a well-characterized polymer and chemically alter the structure in a direction that will favor an increase in the desired electrophysical characteristics. Friedel-Crafts reactions are being carried out between a pure conjugated polymer and a series of compounds, with $AlCl_3$ as catalyst. The results so far are promising, and a significant change in the properties occurs. More efforts will be expended in this direction.

1 P.S. Shantarovich and I.A. Shlyapnikova, *Vysokomolekulyarnye Soedineniya*, 3, 363-67 March, 1961.

Scrupulous attention is being directed to the purity of reagents and solvents. Eventually the effect of this important factor will be investigated quantitatively. Some of the erratic results obtained here in the past may have been due to insufficiently stringent controls in the synthetic part. Most of the polymers now under investigation are being prepared with standard reproducible techniques. However, the emphasis is being shifted towards the investigation of a few well-characterizable polymers.

5:3

Mechanical Section

B. Maxwell

Two individual research projects described below have been active during this report period. In addition work has continued on preparing reports on projects completed during the last academic year. Three reports on this work are now in the hands of publishers for consideration as publications. If accepted they will be distributed as technical reports in reprint form.

In addition work has been undertaken to modify the flexural dynamic test method developed some time ago in this laboratory in order to adapt it to oscillatory shear loading. By this method it is hoped that dynamic tests may be performed through T_g and T_m as a function of frequency while measuring

$$E' (T, \omega), E'' (T, \omega) \text{ and } \frac{d\alpha}{dt}$$

Preliminary apparatus has been constructed to verify the method and has proven successful. Work is in progress to design precision equipment. It is planned to use this method to study the relationship between polymer structure and dynamic response in the melt state.

5:3:1 Failure Mechanisms in Glassy State Polymers

Earlier work in this laboratory indicated that the critical strain at the onset of crazing in polystyrene varied directly with the orientation of the molecules. As a result of this work a theory was developed that accounted for the strain dependence of the initiation of crazing in the glassy state and also gave a plausible explanation of the stopping of crazing cracks prior to ultimate failure. More recent work in other laboratories has developed data which to some degree seems to contradict this theory.

The important question involves the relationship between surface area of the cracks and orientation. Studies have been initiated to obtain information on this relationship by observation of the dependence of surface area (and hence surface energy) on strain magnitude and orientation. The required techniques for imparting controlled orientation to polystyrene bulk specimens have been developed and methods of observing crazed area as a function of strain in the glassy state are under study. Results to date indicate that methods of determining crazed area from residual crazing after fracture are not reliable and the true magnitude of crazed area can only be determined by observations made under various imposed strains up to failure.

5:3:2 Crystallization Mechanisms in Isotactic Polymers

The results of several studies conducted last year indicate that in addition to the normal kinetic factors which determine the crystallization rates for isotactic linear polymers the mechanical deformation of the polymer melt also has an important effect. A several fold decrease in the time of initiation of spherulitic growth in polypropylene can be achieved by the application of shear deformation to the amorphous melt.

In order to determine the relationship of rates of crystallization and such factors as shear deformation magnitude, shear stress and the temperature history of deformation, apparatus has been prepared in which the crystallization process from the melt can be studied while known shear loadings are applied. A polarizing microscope has been equipped with two hot stages for quenching from the melt while simultaneously applying either a specific magnitude of shear deformation or a specific magnitude of shear stress. Initially our work will be concentrated on determining the factors which have the most prominent effects on rate of nucleation. Following this we will be conducting detailed studies of the relationship of shear loading to rate of crystallization and to morphological variations.

6:0 REPORTS, PUBLICATIONS, PAPERS

6:1 Reports

- 66A Alkylation of Ferrocene with Alpha-Aryl Alcohols
E.W. Neuse and D. S. Trifan 23 July 1962
JACS 84 May 1962
- 66B An Evaluation of Polypropylene by Dynamic Mechanical Tests, J. E. Heider and B. Maxwell
23 July 1962 SPE Trans. 2, 2, April 1962
- 66C The Deformation Mechanisms in Branched Polyethylene Melts, J. C. Galt and B. Maxwell
31 August 1962
- 66D Electronic Properties and Structure in Conjugated Polymeric Systems, R. P. Chartoff and C. Cappas
15 February 1963

6:2 Publications

- Velocity Profiles for Polyethylene Melt in Tubes
B. Maxwell and J. C. Galt J. Poly. Sci 62, No. 174
1962

6:3

Papers

Modifying Polymer Properties Mechanically
B. Maxwell; AICh.E. Phil. 26 March 1963

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L. F. Rahm
Project Director

31 March 1963

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Interim Progress Report 15 July 1962 - 31 March 1963		
Contract No. DA-31-124 ARO(D)21		
O.N.R. Project NR 356-375		
1. Chemical Section (a) Polymerization of nonconjugated double bond systems (b) Synthesis of multiple strand polymers. 2. Dielectrics Section (a) Studies in semi-conducting high polymers. 3. Mechanical Section (a) Failure Mechanism in glassy state polymers (b) Crystallization mechanisms in isotactic polymers.		
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