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OFFICE OF NAVAL RESEARCH
FINAL REPORT
SUMMARY/PUBLICATIONS/PRESENTATIONS/STUDENTS REPORT

for
Grant or Contract N00014-91-J-1475
R&T Code 4132056

**SOLID STATE NMR STUDIES OF MORPHOLOGY AND ORIENTATION IN
POLYMERS**

Prof. James F. Haw
Department of Chemistry
Texas A&M University
College Station, TX 77843

Nov 28, 1995

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Final Report for ONR Grant #N00014-91-J-1475

Summary

Our objective was to provide research training opportunities for graduate students in NMR characterization of polymers, in particular polymers for NLO applications. We collaborated with the Allcock group at Penn State and have a large paper in press in *Macromolecules* on polyphosphazene NLO polymers. The methodology was to use P-31 and C-13 MAS NMR to study in detail the temperature dependent main-chain and NLO side-group dynamics in two NLO polymers. We studied photochemical cross-linking in host polymer systems and verified the 2+2 mechanism that was central to the original proposal. This was first communicated in *Macromolecules*, and other work will be forthcoming.

Graduate Students Supported from the Grant

- | | |
|-------------------|------------------------|
| 1. Tom Krawietz | 6. Jinhau Zhang |
| 2. David Murray | 7. Sharon Taylor-Myers |
| 3. Tim Skloss | 8. Nicholas Elbaum |
| 4. Dodi Heryadi | 9. Jeffrey White |
| 5. David Ferguson | 10. Ali Kheir |

Publications

1. H. R. Allcock, C. G. Cameron, T. W. Skloss, S. Taylor-Myers and J. F. Haw, Molecular Motion of Phosphazene-Bound NLO Chromophores, *Macromolecules*, in press (1995).
2. W. Skloss and J. F. Haw, Detection of Cross-Link Formation by 2+2 Photocycloaddition in Poly(vinylcinnamate) by ¹³C Solid-State NMR, *Macromolecules*, **27**, 6998-6999 (1994).
3. J. L. White, L. W. Beck, D. B. Ferguson and J. F. Haw, Background Suppression in MAS-NMR, *J. Magn. Reson.*, **100**, 336-341 (1992).
4. N. C. Elbaum and J. F. Haw, A Robust Algorithm for Isotropic Reconstruction of Magic-Angle Spinning Solid-State NMR Spectra, *Anal. Chem.*, **64**, 2555-2561 (1992).
5. S. A. Taylor, J. L. White, N. C. Elbaum, R. C. Crosby, G. C. Campbell, J. F. Haw, and G. R. Hatfield, Morphology, ³¹P Spin Diffusion and Phase Transitions in a Representative Semicrystalline Polyphosphazene by Solid-State NMR, *Macromolecules*, **25**, 3369-3376 (1992).
6. J. F. Haw, Nuclear Magnetic Resonance Spectroscopy, *Anal. Chem.*, **64**, 243R-254R (1992).
7. S. A. Taylor, D. B. Ferguson and J. F. Haw, Chromophore Motion in Polymers for Nonlinear Optics by Solid-State NMR, *Macromolecules*, **25**, 2784-2785 (1992).
8. D. G. Gabler and J. F. Haw, Hydrolysis of Poly(dichlorophosphazene), *Macromolecules*, **24**, 4218-4220 (1991).
9. S. Maynard, T. Sharp and J. F. Haw, Thermal Degradation Chemistry of Poly(diphenoxy)phosphazene, *Macromolecules*, **24**, 2794-2799 (1991).
10. R. C. Crosby and J. F. Haw, Solid-State NMR of Phosphazene Inorganic Polymers, in *Solid-State NMR of Polymers*, L. Mathias, Ed., Plenum Press: New York, NY (1991).

Presentations

1. 35th ENC, Pacific Grove, CA., April 1994, "NMR Instrumentation for Probing Chemical Reactions"
2. Society for Applied Spectroscopy, Rice University, January 1994, "New Applications of NMR Spectroscopy"
3. ACS 49th Southwest Regional Meeting, Austin, TX., October 1993, "Characterization of Slow Chromophore Reorientation in Nonlinear Optical Polymers by 2-D NMR"
4. University of Delaware NMR Symposium, Newark, DE, June 1993, "NMR and the Academic-Industrial Interface"

5. NMR User's Association of Brazil 9th Conference, Angra dos Reis, Brazil, May 1993, "Solid State NMR of Materials"
6. The Aerospace Corporation, El Segundo, CA, March 1993, "Solid-State NMR Studies of Morphology and Dynamics: Nonlinear Optical Polymers and Polyphosphazenes." (Presented by Ms. Sharon A. Taylor-Myers)
7. Sandia National Laboratories, Albuquerque, NM, March 1993, "Solid-State NMR Studies of Morphology and Dynamics: Nonlinear Optical Polymers and Polyphosphazenes." (Presented by Ms. Sharon A. Taylor-Myers)
8. Rohm and Haas, Bristol, PA, February 1993, "Characterizing Dynamics of Nonlinear Optical Polymers using Solid-State NMR." (Presented by Ms. Sharon A. Taylor-Myers)
9. Magnetic Resonance Imaging Short Course for Industry, Department of Chemical Engineering, Texas A&M University, November 1992, "Introduction to NMR"
10. Department of Chemistry, Purdue University, October 1992, "Future Directions in Analytical NMR"
11. Advanced Solid State NMR Workshop, Ft. Collins, CO, July 1992, "Recent Developments in MAS NMR"
12. Office of Naval Research Contractor's Meeting, Naval Research Laboratory, Isle of Palms, SC, April 1992, "NMR of NLO Polymers" (Presented by Ms. Sharon A. Taylor-Myers)
13. Sigma Xi Interdisciplinary Research Workshop, Texas A&M University, College Station, TX, April 1992, "Solid State NMR of Materials"
14. Houston Area Research Consortium, Woodlands, TX, December 1991, "NMR Applications to Materials Science"
15. ACS 47th Southwest Regional Meeting, San Antonio, TX, October, 1991, "Characterization of Nonlinear Optical Polymers Using Solid-State NMR" (Presented by Ms. Sharon A. Taylor-Myers)
16. W. R. Grace and Company, Baltimore, MD, September 1991, "Developing NMR Methods for Characterizing Orientation in Polymers"
17. Advanced Solid State NMR Workshop, Ft. Collins, CO, July 1991, "Isotropic Reconstruction: The Second Chapter"
18. Magnetic Resonance Imaging Short Course for Industry, Department of Chemical Engineering, Texas A&M University, May, 1991, "Introduction to NMR"
19. Department of Chemistry, Juniata College, February 1991, "Investigating Solid-State Chemistry with NMR"

FORM A2-2

AUGMENTATION AWARDS FOR SCIENCE & ENGINEERING RESEARCH TRAINING (AASERT)
REPORTING FORM

The Department of Defense (DOD) requires certain information to evaluate the effectiveness of the AASERT program. By accepting this Grant Modification, which bestows the AASERT funds, the Grantee agrees to provide the information requested below to the Government's technical point of contact by each annual anniversary of the AASERT award date.

1. Grantee identification data: (R & T and Grant numbers found on Page 1 of Grant)

- a. Texas A&M University
University Name
- b. N00014-91-J-1475 Grant Number
- c. 4132056 R & T Number
- d. Dr. James F. Haw P.I. Name
- e. From: 12/1/94 To: 11/30/95
AASERT Reporting Period

NOTE: Grant to which AASERT award is attached is referred to hereafter as "Parent Agreement."

2. Total funding of the Parent Agreement and the number of full-time equivalent graduate students (FTEGS) supported by the Parent Agreement during the 12-month period prior to the AASERT award date.

- a. Funding: \$ 75,000
- b. Number FTEGS: 2


3. Total funding of the Parent Agreement and the number of FTEGS supported by the Parent Agreement during the current 12-month reporting period.

- a. Funding: \$ 80,000
- b. Number FTEGS: 2.5

4. Total AASERT funding and the number of FTEGS and undergraduate students (UGS) supported by AASERT funds during the current 12-month reporting period.

- a. Funding: \$ 84,864
- b. Number FTEGS: 1
- c. Number UGS: _____

VERIFICATION STATEMENT: I hereby verify that all students supported by the AASERT award are U.S. citizens.


Principal Investigator

11/22/95
Date