Project #: G-41-607  
Center #: 10/24-6-R6883-0A0  
Contract#: 22325-AC7

Cost share #:  
Center shr #:  
Mod #: 

Active

Subprojects ? : N
Main project #: 

Project unit: PHYSICS  
Project director(s): HUANG T-H

Cost share
Center shr
PHYSICS
PHYSICS

OCA file #:
Work type #: RES
Document : GRANT
Contract entity: GTRC

Rev #: 1

Award period: 900201 to 920831 (performance) 921001 (reports)

Sponsor amount  
New this change  
Total to date  
Contract value 0.00 40,000.00
Funded 0.00 40,000.00
Cost sharing amount 0.00

Does subcontracting plan apply ?: N

Title: NUCLEAR MAGNETIC RESONANCE STUDIES OF CRYSTALLIZATION IN ORIENTED ...

Project Administration Data

OCA contact: Kathleen R. Ehlinger 894-4820
Sponsor technical contact

Sponsor issuing office

JOSEPH E. ROGERS, JR. (202)872-4481

AMERICAN CHEMICAL SOCIETY 1155 16TH ST., N.W. WASHINGTON, D.C. 20036

Security class (U,C,S,TS) : U
Defense priority rating : N/A
Equipment title vests with: Sponsor NONE PROPOSED.

Administrative comments - ISSUED TO REVISE DELIVERABLE SCHEDULE.
GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION
NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 11/09/92

Project No. G-41-607

Project DirectorHUANG T-H

Center No. 10/24-6-R6883-0A0

School/Lab PHYSICS

Sponsor AMERICAN CHEMICAL SOCIETY/PETROLEUM RES FUND - DC

Contract/Grant No. 22325-AC7

Prime Contract No.

Contract Entity GTRC

Title NUCLEAR MAGNETIC RESONANCE STUDIES OF CRYSTALLIZATION IN ORIENTED ...

Effective Completion Date 920831 (Performance) 921001 (Reports)

Closeout Actions Required: Y/N Submitted

Final Invoice, or Copy of Final Invoice N

Final Report of Inventions and/or Subcontracts N

Government Property Inventory & Related Certificate N

Classified Material Certificate N

Release and Assignment N

Other N

Comments

Subproject Under Main Project No. ____________

Continues Project No. ____________

Distribution Required:

Project Director Y

Administrative Network Representative Y

GTRI Accounting/Grants and Contracts Y

Procurement/Supply Services Y

Research Property Management Y

Research Security Services Y

Reports Coordinator (OCA) Y

GTRC Y

Project File Y

Other HARRY VANN FMD Y

FRED CAIN OOD Y
We have employed one- and two-dimensional CP/MAS $^{13}$C NMR techniques to study the morphology and orientational order of melt-spun (2000-5300 m/min) polyethylene (terephthalate) (PET) and ultra-high molecular weight polyethylene (UHMWPE) fibers. Multiple component features were observed for both polymer systems. The relative population of each component can be determined accurately from the characteristic changes in intensity at different cross polarization time. We found that the PET fibers compose of at least four components with varied orientational order (1), whilst the UHMWPE system consists of three components (2). The relative population of these components varied substantially with post-processing conditions such as cold drawing and annealing. By studying the morphology of melt-spun (5300 m/min) PET fibers which were quenched at 9 to 21 inches from the spinneret the evolution of these morphological components was deduced.

2-D rotor synchronized MAS (ROSMAS) technique was also employed to determine the orientational order. A linear regression algorithm was developed to determine the order parameter, thus the orientational distribution function (ODF) of the fiber systems. By performing careful deconvolution we, for the first time, were able to determine the orientational order of each morphological component. We are still in the process of analyzing these data. Initial results suggest that the amorphous components in PET fibers have orientational order which is higher than that of the crystalline component at low spinning rate. The development of the orientational order was also studied with the rapid quenched sample. We are also studying the orientational order of the PE system.

References:


PERSONNEL STATEMENT

PRF# 22325-AC7  REPORTING PERIOD  02/01/90 TO 8/31/91

GRANTEE INSTITUTION: Georgia Institute of Technology
DEPARTMENT: School of Physics

PRINCIPAL INVESTIGATOR(S): Dr. Tai-Huang Huang

GRANT PROJECT TITLE: Nuclear Magnetic Resonance Studies of Crystallization in Oriented Poly (Ethylene Terephthalate) Fibers

List undergraduate, graduate, and postdoctoral co-workers receiving stipends under the above named grant:

<table>
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<tr>
<th>NAME</th>
<th>TITLE OR ACADEMIC APPOINTMENT</th>
<th>PREVIOUS EDUCATION &amp; DEGREES*</th>
<th>COUNTRY OF PERMANENT RESIDENCE</th>
<th>PERIOD OF SUPPORT (MONTHS)</th>
<th>PERCENT OF SUPPORT FROM PRF **</th>
<th>DEGREES RECEIVED (IF ANY) DURING REPORTING PERIOD</th>
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<td>Huang, Fu-Yung</td>
<td>Postdoctoral Fellow</td>
<td>University of Maine</td>
<td>R.O.C. China</td>
<td>7/1/91 - 8/31/91</td>
<td>6%</td>
<td>none</td>
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<tr>
<td>Tzou, Derlii Mike</td>
<td>Graduate Research Assistant</td>
<td>Florida Inst. of Tech</td>
<td>R.O.C. China</td>
<td>2/1/90 - 8/31/91</td>
<td>33 1/3%</td>
<td>none</td>
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List other co-workers on grant project not directly supported with ACS - PRF funds: (none)

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<th>SOURCE OF SUPPORT</th>
<th>DATES ASSOCIATED WITH GRANT PROJECT</th>
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</table>

* For graduate students, indicate the College or University attended prior to graduate work. For postdoctoral fellows, give the name of the Ph. D. granting institution.

**(during the period stated in preceding column)**

Revised 6/82
Studies of the Crystallization in Oriented Poly(Ethylene Terephthalate) Fibers.

Tai-huang, Huang, Georgia Institute of Technology

A fundamental understanding of the mechanism of fiber formation requires a detailed knowledge of fiber morphology. However, due to the limitation of current available techniques, the crystallization behavior is still not fully understood. In this work we employed solid state $^{13}$C NMR techniques to obtain detailed information on the morphology of as-spun PET and gel-spun ultra high molecular weight high performance PE fibers. The presence of multiple-components in the NMR spectra were analyzed to obtained quantitative information of morphological components. We identified the presence of four components of varied segmental order and crystallinity for the PET fibers. Whilst three components, two crystalline forms and one amorphous form, were observed for UHMWPE. The effect of post-processing on the morphological changes was also carefully monitored. We further employed 2-dimensional rotor-synchronized MAS $^{13}$C NMR (ROSMAS) technique to characterized to orientational order of these components to high order. The orientational distribution functions (ODF) calculated from these 2D spectra clearly displayed a wide range of variation. For the PET system we found that orientational order of the amorphous component can be higher than that of the crystalline components. Only at spun speed above 2000 m/min does the fiber develop high orientational order. The evolution of these morphological components was also determined by using a set rapid quenched fiber sample. We found that high degree of order and crystallinity develop at distance between 15 and 21 inches from the spinnerate. In the PE system, the two crystalline components shows very different long-range ordering. Thus, our results provide the most detailed morphological information of these two polymer systems which should be of value for the understanding of the polymer fiber systems.

References:


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References:


Tzou, D.L., Huang, T.-h.; Abhiraman, A.S. & Desai, P.

"Two-dimensional 13C NMR Studies of the Morphology and Orientation Order in Gel-Spun Ultrahigh Molecular Weight Polyethylene fibers"
Two-dimensional $^{13}$C n.m.r. studies of the morphology and orientational order in gel-spun ultrahigh molecular weight polyethylene fibres

Der-Lii Tzou, Tai-huang Huang*, A. S. Abhiraman† and Prashant Desai‡
School of Physics, †School of Chemical Engineering and ‡School of Textile & Fiber Engineering
Georgia Institute of Technology, Atlanta, GA 30332, USA
(Received 10 June 1991)

In this communication we demonstrate the feasibility of determining the orientational distribution functions of various morphological components in ultrahigh molecular weight polyethylene fibres by two-dimensional rotor-synchronized $^{13}$C cross-polarization/magic angle spinning nuclear magnetic resonance (n.m.r.) techniques. For comparative purposes we have also included a drawn polyethylene fibre of normal molecular weight. The results showed that these fibres contain two crystalline components, designated C1 and C2, and an amorphous (non-crystalline) component, A. Order parameters and orientational distribution functions of these components can be obtained through analysis of the two-dimensional $^{13}$C n.m.r. spectra.

(Keywords: polyethylene; morphology; $^{13}$C n.m.r.; orientational order; fibre)

Introduction

Formation of high mechanical performance fibres from ultrahigh molecular weight linear polyethylene (UHMWPE) represents an important technical advance in the quest for realizing maximum mechanical performance from organic polymers, especially those with considerable intrinsic flexibility. The morphology of these fibres is, without doubt, dominated by previously unattained degrees of chain extension and orientation in such polymers. There is general agreement that the hierarchy of organization from fibre to the polyethylene (PE) chain in UHMWPE fibres consists of macrofibrils (15 nm in diameter) of extended chains. The interfibrillar regions are believed to be extended domains of non-crystalline material and/or microvoids. There is some disagreement regarding the structure of the microfibrils themselves, with proposals ranging from perfectly crystalline fibrils (except for chain ends) to the classical synthetic fibre structure, namely alternating crystalline and non-crystalline domains in the fibril. Recent studies in our laboratories in this regard, with extensive thermorheological and X-ray scattering data, imply that the microfibrils in UHMWPE fibres consist of an alternating sequence of crystals of high and relatively lower degrees of perfection, with a low concentration of amorphous material dispersed in the interfibrillar domains. In this communication we describe preliminary results from studies with one- and two-dimensional nuclear magnetic resonance (n.m.r.) techniques to determine the nature and extent of various morphological components quantitatively, and also the orientational order in these components.

Experimental

The experiments were carried out with three PE fibres (Table 1), designated as PE-I (Allied Spectra 900), PE-II (Allied Spectra 1000) and PE-D. PE-II and PE-I are both UHMWPE fibres ($M_w > 10^6$, draw ratio > 40), with strength of 32 and 25 cN/dtex*, elongation of 3.8 and 6%, and modulus of 1940 and 750 cN/dtex, respectively. PE-D is a normal molecular weight melt-spun fibre that has been subsequently drawn ($M_w = 10200$, draw ratio = 3.0).

N.m.r. experiments were performed on a custom-built spectrometer operating at 75.5 MHz ($^{13}$C). A Doty magic angle probe (Doty Scientific, SC) with 7 mm rotors was employed to obtain the cross-polarization/magic angle spinning (CP/MAS) $^{13}$C spectra. Experimental conditions: 1 ms CP, 4 s (PE-I and PE-D) or 90 s (PE-II) recycle delay. Spectra of 50 kHz width were taken with 4 k points resolution and zero-filled to 8 k. Spectra were taken at room temperature (22°C). The relative populations of different morphological components were obtained by computer line shape simulation of the observed spectra. The intensities of these components must be corrected to compensate for the presence of difference in CP efficiency and proton $T_1\rho$. This is accomplished by simulating the relative intensities of these components for spectra obtained with contact times ranging from 50 μs to 80 ms. The absolute intensities of these components were obtained by extrapolating to zero second contact time, as described previously. Sample spinning speed was controlled by a custom-built microprocessor-controlled spinning rate controller to within ± 1 Hz. The two-dimensional rotor synchronized magic angle spectra (ROSMAS) were obtained with the pulse sequence proposed by Harbison et al. Samples for these experiments were prepared by carefully winding the fibres on a flat-spool with a precision mechanical advancing mechanism. Six layers of the fibres were glued together and allowed to dry overnight before cutting into 4 × 12 mm rectangular pieces. Six pieces of these rectangles were glued together and placed in a 7 mm MAS spinner. The fibre axis was tilted by 51° from the

*To whom correspondence should be addressed at: Institute of Biomedical Sciences, Academia Sinica, Taipei 11529, Taiwan, ROC
0032-3861/92/020426-03
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* dtex is a measure of linear density of fibres and corresponds to the mass in grams of 10000 m of fibres.
Table 1  Relative fraction and line width of the morphological components

<table>
<thead>
<tr>
<th>Sample</th>
<th>$C_1^*$</th>
<th>$\Delta v_{1/2}$ (Hz)$^*$</th>
<th>$C_2^*$</th>
<th>$\Delta v_{1/2}$ (Hz)$^*$</th>
<th>$A^*$</th>
<th>$\Delta v_{1/2}$ (Hz)$^*$</th>
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<tr>
<td>PE-II</td>
<td>0.15</td>
<td>33</td>
<td>0.85</td>
<td>35</td>
<td>0.00</td>
<td>-</td>
</tr>
<tr>
<td>PE-I</td>
<td>0.06</td>
<td>35</td>
<td>0.76</td>
<td>35</td>
<td>0.18</td>
<td>120</td>
</tr>
<tr>
<td>PE-D</td>
<td>0.06</td>
<td>59</td>
<td>0.68</td>
<td>40</td>
<td>0.26</td>
<td>120</td>
</tr>
</tbody>
</table>

$^*$Relative fraction (±0.02)
$^*$Line width ±5 Hz

Separation between $C_1$ and $C_2 = 105$ Hz (1.5 ppm) and between $C_2$ and $A = 135$ Hz (1.8 ppm)

Results and discussion

Figure 1 shows a set of $^{13}$C CP/MAS spectra of PE-I, PE-II and PE-D. Each of these spectra consists of three components (labelled $C_1$, $C_2$ and $A$). Similar results were also observed by Jarrett et al. in a series of high molecular weight PE reactor powders synthesized under slurry and gas phase reaction conditions using Ziegler-Natta catalysis. The relative intensity and line width of each component can be deduced from spectral simulation. The results, after appropriate correction for CP and relaxation effects as described in the Experimental section, are given in Table 1. The accuracy in per cent population obtained from simulation is estimated to be ± 2%. The line widths are $\Delta v_{1/2} = 35 ± 5$ Hz for $C_1$ and $C_2$ and $120 ± 10$ Hz for component $A$. These data show that PE-II contains the highest population of $C_1$ (15%) and the PE sample contains the highest amount of component $A$ (29%). There is also a substantial fraction of component $A$ present in the PE-I sample (18%) but not in the PE-II sample.

Figure 2 shows one set of the two-dimensional ROSMAS spectra of the PE-II sample. The sidebands in the $N$-dimension are the normal MAS sidebands, modulated by orientational order of the fibres. Thus, the $M = 0$ spectrum is exactly the same as the normal one-dimensional CP/MAS spectrum. Each of these spectra was deconvoluted to obtain the intensities of various morphological components for all $M$-slices. The intensity variation of a given component in the $M$-dimension is employed to determine the orientational distribution functions of that component using a modified procedure similar to that employed by Harbison et al. The presence of strong intensity in the spectra with...
high $M$ values represents high orientational order. 

Figure 3 shows the intensity variation in the $M$-dimension of the three components of the $N = 0$ slice for PE-II sample. Substantial intensities can still be seen at $M = \pm 7$ for both $C_1$ and $C_2$ components. However, no intensity of the $A$ component is detectable even in the $M = \pm 1$ spectra. This is a clear indication that the $A$ component corresponds to an unoriented amorphous region of the fibres. The presence of high-order intensities for the $C_1$ and $C_2$ components suggests that both of these components are likely to be from oriented crystalline regions. This is consistent with the observed narrow line width of 35 Hz for both components and supports the assignments of Jarrett et al.\(^{(15)}\). For PE-I and PE-D samples, similar plots show that both $C_2$ and $C_1$ are ordered (results not shown), but the intensities of these components decrease faster than in the PE-II sample, suggesting that the long range order in the fibrils of PE-I and PE-D samples is lower than that of PE-II. A more detailed quantitative description of the orientation distributions, together with an analysis of the effect of processing on the morphology and orientational distribution function of these fibres, is currently in progress.

Acknowledgement

Acknowledgement is made to the donors of The Petroleum Research Fund, administered by the American Chemical Society, and the Georgia Tech Polymer Program Associates for the partial support of this research. D. L. Tzou is an American Chemical Society Petroleum Research Fund Fellow.

References

5. Rotzinger, B. P., Chanzy, H. D. and Smith, P. *Polymer* 1989, 30, 1814
Tzou, D. L. (PH.D. Thesis)

"Solid State $^{13}$C NMR Studies of the Morphology and Orientation Order of Polymer Fibers"
Solid State $^{13}$C NMR Studies of the Morphology and Orientational Order of Polymer Fibers

A THESIS
Presented to
The Faculty of the Division of Graduate Studies

By

Der-Lii M. Tzou

In Partial Fulfillment
of the Requirements for the Degree
Doctor of Philosophy
in the School of Physics

Georgia Institute of Technology
September 1991

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SUMMARY

A fundamental understanding of the mechanism of fiber formation requires a detailed knowledge of fiber morphology. However, due to the limitation of current techniques, the crystallization behavior is still not fully understood. Modern solid state NMR techniques enable one to approach this subject from different aspects. In this work, we conducted detailed studies of the morphology of as-spun PET (poly[ethylene terephthalate]) and gel-spun PE (poly[ethylene]) fibers by $^{13}$C CP/MAS (Cross Polarization/Magic Angle Spinning) NMR technique. The presence of multiple-component resonance lineshapes allow one to calculate the true population of various morphological components, after correcting for spin relaxation effects. From these NMR measurements, two different models for PET and PE polymer systems have been proposed. We have also employed and further developed the advanced two-dimensional rotor synchronized MAS (2D ROSMAS) technique to study the orientational order of various morphological components. A wide range of orientational order is clearly displayed on 2D spectra for samples under different fabrication conditions. A regression simulation program has been used to deduce the order parameter used in computing the orientational distribution function (ODF) for each component. Our results for the first time provide unique information about orientational order and morphology at the molecular level, thereby providing a much more complete basis for theoretical modeling of polymer system.
PERSONNEL STATEMENT

PRF# 22325-AC7 REPORTING PERIOD 9/1/91 TO 8/31/92

GRANTEE INSTITUTION Georgia Institute of Technology

DEPARTMENT School of Physics

PRINCIPAL INVESTIGATOR(S) Dr. Tai-Huang Huang

GRANT PROJECT TITLE Nuclear Magnetic Resonance Studies of Crystallization in Oriented Poly (Ethylene Terephthalate) Fiber

List undergraduate, graduate, and postdoctoral co-workers receiving stipends under the above named grant:

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<tr>
<td>Huang, Fu-Yung</td>
<td>Postdoctoral Fellow</td>
<td>University of Maine, Ph.D.</td>
<td>R.O.C. China</td>
<td>9/1/91 to 9/30/91</td>
<td>50%</td>
<td>none</td>
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<tr>
<td>Tzou, Derlii Mike</td>
<td>Graduate Research Assist.</td>
<td>Florida Inst. of Tech</td>
<td>R.O.C. China</td>
<td>9/1/91 to 12/31/91</td>
<td>33 1/3%</td>
<td>Ph.D</td>
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List other co-workers on grant project not directly supported with ACS - PRF funds:

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</tbody>
</table>

* For graduate students, indicate the College or University attended prior to graduate work. For postdoctoral fellows, give the name of the Ph. D. granting institution.

** (during the period stated in preceding column)
CURRICULUM VITAE - Tai-huang Huang

Date of Birth: August 22, 1945. Marital Status: Married with two children

Educational Background:

B. S. 1969 National Taiwan Normal University.
Ph.D. 1979 Brandeis University, Waltham MA (with A. G. Redfield)

Employment History:

1982 - 1986 Assistant professor of Physics & adjunct assistant professor of Biochemistry, University of Maine, Orono, Maine, USA.
1982 - 1986 Associate member of the laboratory of Surface Science and Technology, University of Maine, Orono, Maine, USA.
1986 - present Associate professor of Physics, Georgia Institute of Technology, Atlanta, GA USA.
1991 - present Special Medical Research Fellow, Institute of Biomedical Sciences, Academia Sinica, Taiwan, R.O.C.

Professional Interests:

NMR Spectroscopy in Solution and Solid, Structural Molecular Biophysics (proteins and biomembranes) (Faculty member of The Research Center for Biotechnology, Georgia Tech), Polymer Physics (Faculty member of Polymer Education and Research Center, Georgia Tech), Magnetic Resonance Imaging.

Membership in Profession Societies:

Member of American Chemical Society, Biophysical Society, Sigma Xi Society, and International Society of Magnetic Resonance.

Honors, Awards or Recognitions:

Chinese Culture and Natural Science Scholarship.
Madame Yuan's Scholarship. Rosenstiel Scholarship.
Member, NIH Study Section in Biomedical Instrumentation Program, 1983, 1985.
Chair, Symposium in Modern NMR spectroscopy, 40th ACS Southeast Regional Meeting, Atlanta, GA, 1988.

Publications:


Manuscript Submitted :


"Deuterium NMR investigation of the dynamics of trimethoprim in complex with E. coli dihydrofolate reductase" Q.-X Yang, F.Y. Huang, L. Gelbaum, & T.-h. Huang, Biochemistry.


Invited Talks:

7. "Symposium on Applications of Modern NMR Spectroscopy", Academia Sinica, Taipei, Taiwan,
Contributed Presentations (After 1988):

   b. "Restricted Motion of the Phosphate Group in Crystalline 3'-CMP/RNasee Complex" T.-h Huang, W. Gilbert, G. Petsco, and R. G. Griffin.

2. 40th American Chemical Society Southeast Regional Meeting, Nov. 9, 1988, Atlanta, GA.
   Topic: "Proton Mobility in Liquid and Frozen HCLO$_4$.5.5H$_2$O: NMR and Conductivity Measurements.

   Topic: "A Microprocessor Controlled Spin-Rate Controller", T. Maier & T.-h Huang.

   Topic: "Multinuclear NMR Studies of the Structure and Dynamicsof E. coli Dihydrofolate Reductase" F.Y Huang, Q.X Yang, L. Gelbaum & T.h Huang

   Topics: a. "Multinuclear NMR Studies of Dihydrofolate Reductase" F.Y Huang, Q.X Yang, L. Gelbaum & T.h Huang

   Topic: "$^{15}$N and $^{31}$P NMR Studies of the Conformation of Dihydrofolate Reductase " F.Y. Huang, Q.X. Yang, L. Gelbaum & T.-h. Huang.

7. 32nd Experimental Nuclear Magnetic Resonance Conference, April, 1991. St. Louis, MO.

Research Grants and Contracts :

1. Research Corporation: 3/83 - 5/85 ---------------------------- $19,000
   "NMR Studies of Snake Venom Toxins"

2. NSF: (PCM 8309333) 8/14/83 - 1/31/87 -------------------------- $165,000.
   "NMR Investigations of Biological Membrane Structure and Function"
3. NSF: (PCM 8313208) 1/84 - 6/86 ................................. $170,000
"Acquisition of a NMR Spectrometer"

4. USDA: 9/85 - 9/87 ........................................... $183,000.

5. Teregenic Cooperation: ........................................... $3,000
"Feasibility Study of Magnetic Resonance Imaging of $^{11}$B"

6. Emory-Georgia Tech. Biomedical Research Center: 1/1/88 - 12/31/88
"Solution NMR Studies of Dihydrofolate Reductase" Co-PI with D. Live of Emory University ........................................... $30,000.

7. Georgia Tech Polymer Program 9/1/87 - 8/31/90 .......................... $16,000
"NMR Studies of Orientation Distribution of High-Speed Melt-Spun Poly(ethylene terephthalate) (PET) Fibers"

8. NIH: (GM39779) 4/1/88 - 3/31/92 ................................ $309,920
"Structure, Dynamics, and Function of Dihydrofolate Reductase"

9. ACS-Petroleum Research Fund (ACS-PRF #22323-SC7)
2/1/90 - 8/31/92 .............................................. $40,000
"NMR Studies of the Crystallization of Poly(ethylene terephthalate) Fibers"

Students and Postdoctoral Fellow Supervised:


Post Doctoral Fellow:
S. H. Grode NMR Specialist, The Upjohn Corp.
T. T. Ang Lecturer, Universiti Sains Malaysia, Penang, Malaysia.
F.Y. Huang, Research Scientist, Georgia Tech.
B.H. Lee, Senior Postdoctoral Fellow, IBMS, Academia Sinica, Taipei.