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(2)

REPORT DOCUMENTATION PAGE

Form Approved
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1a. REPORT SECURITY CLASSIFICATION

AD-A211 460

1b. RESTRICTIVE MARKINGS

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3. DISTRIBUTION/AVAILABILITY OF REPORT

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4. PERFORMING ORGANIZATION REPORT NUMBER(S)

5. MONITORING ORGANIZATION REPORT NUMBER(S)

AFOSR-TR- 89-1185

6a. NAME OF PERFORMING ORGANIZATION	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION
University of Massachusetts		AFOSR/NC

6c. ADDRESS (City, State, and ZIP Code)	7b. ADDRESS (City, State, and ZIP Code)
Amherst, MA 01003	Building 410, Bolling AFB DC 20332-6448

8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
AFOSR	NC	AFOSR-88-0046

8c. ADDRESS (City, State, and ZIP Code)	10. SOURCE OF FUNDING NUMBERS			
	PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO	WORK UNIT ACCESSION NO.
Building 410, Bolling AFB DC 20332-6448	61102 F	2303	A3	

11. TITLE (Include Security Classification)

ULTRASTRUCTURE PROCESSING AND CHARACTERIZATION OF POLYMERS (U)

DTIC ELECTE MAR 1 1989 D

12. PERSONAL AUTHOR(S)

Frank E. Karasz

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
FINAL	FROM _____ TO _____		8

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

During this period the research program contained components dealing inter alia with polymer blends, electro- and optically-active polymers, computer simulation of blend phenomena, dynamic light scattering measuring diffusion in porous media, and aspects of ultrastructural processing. New state-of-the-art instrumentation was developed, including a two-dimensional x-ray system and solid state NMR. The MIRP program constitues the core research complementing a contract which was initiated here on 1 January 1987.

In the area of polymer blends we have continued to emphasize the effect of chain microstructure on miscibility in multi-component systems. This has involved phase behavior studies of copolymer-containing blends, where the structure of the copolymer have been varied in terms of chemistry, tacticity and/or sequence distribution. We have also emphasized studies of fundamental interactions in certain high temperature blend systems which were developed

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
☒ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT ☐ DTIC USERS	UNCLASSIFIED

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL
Dr Donald R. Ulrich	(202) 767-4963	NC

DD Form 1473, JUN 86 — Previous editions are obsolete. — SECURITY CLASSIFICATION OF THIS PAGE

89 8 21 005

in the URI program. During the period, a program for the computer simulation of An/Bn mixtures on two- and three-dimensional lattices yielded valuable insights into mixing phenomena, initially in terms of equilibrium states, but more recently extended to dynamic conditions.

In the electro-active polymer area, we have concentrated on research involving poly(p-phenylene vinylene) (PPV) and its derivatives, copolymers, and blends. The electrical and, more recently, the non-linear optical properties of this family of conjugated polymers has proved to be of particular interest and potential applicability. Studies of the morphology of PPV has led to observation of the importance of ultra-structure processing in the development of maximum property sets. Finally, mention may be made of a new technique for studying diffusion in constrained media pores using quasi-elastic light scattering. This has led to advances in the theory of motion of flexible chains under hindered conditions and constitutes a novel method of examining the effect of chain microstructure and architecture on such motions.

FINAL REPORT

ULTRASTRUCTURE PROCESSING AND CHARACTERIZATION OF POLYMERS

MIRP Grant AFOSR 87-0033

1 October 1986 - 30 September, 1987

MIRP Grant AFOSR 88-0046

7 October 1987 - 30 September 1988

Frank E. Karasz
Department of Polymer Science & Engineering
University of Massachusetts
Amherst, MA 01003

FINAL REPORT

ULTRASTRUCTURE PROCESSING AND CHARACTERIZATION OF POLYMERS

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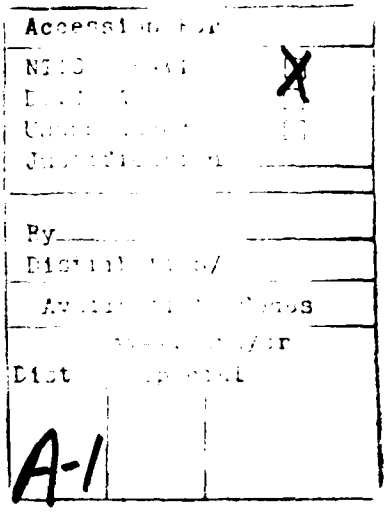

Frank E. Karasz

Department of Polymer Science & Engineering

University of Massachusetts

Amherst, MA 01003

ACKNOWLEDGEMENT

The Principal Investigator would like to express his sincere thanks to Dr. Donald Ball, Director of Chemical Sciences, Air Force Office of Scientific Research, to Dr. Donald R. Ulrich, Senior Program Manager, Directorate of Chemical Sciences, and to other members of the Directorate, for their unfailing cooperation, help and courtesy extended to him during the period of this grant.

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I. TITLE: Ultrastructure Processing of Polymers

II. PRINCIPAL INVESTIGATOR:
Dr. Frank E. Karasz
Polymer Science & Engineering
University of Massachusetts
Amherst, Massachusetts 01003

III. GRANT NUMBER: AFOSR 86-0033 (October 1, 1986 - September 30, 1987)

AFOSR 88-0046 (October 7, 1987 - September 30, 1988)

IV. FACULTY ASSOCIATES:
Professor W.J. MacKnight
Professor R.W. Lenz

SENIOR RESEARCH PERSONNEL:
Dr. A.C. Balazs
Dr. G. Banhegyi
Dr. G. Guerra
Dr. C. C. Han
Dr. M. Jedlinska-Obrzut
Dr. D.S. Lee
Dr. J. Obrzut
Dr. D. Rice
Dr. J. Schlenoff
Dr. J. Shim

V. JUNIOR RESEARCH PERSONNEL:
M. Bishop
R. Gregorius
M. Lux
J. Machado
M. Masse
R. Stankovic
K. Kang

VI. ABSTRACT

This report is for the period 1 October 1986 to 30 September 1988 and covers the operation of an AFOSR MIRP at the University of Massachusetts. During this period the research program contained components dealing inter alia with polymer blends, electro- and optically-active polymers, computer simulation of blend phenomena, dynamic light scattering measuring diffusion in porous media, and aspects of ultrastructural processing. New state-of-the-art instrumentation was developed, including a two-dimensional x-ray system and solid state NMR. The MIRP program constitutes the core research complementing an AFOSR/DARPA URI contract which was initiated here on 1 January 1987.

In the area of polymer blends we have continued to emphasize the effect of chain microstructure on miscibility in multi-component systems. This has involved phase behavior studies of copolymer-containing blends, where the structure of the copolymer have been varied in terms of chemistry, tacticity and/or sequence distribution. We have also emphasized studies of fundamental interactions in certain high temperature blend systems which were developed in the URI program. During the period, a program for the computer simulation of A_n/B_n mixtures on two- and three-dimensional lattices yielded valuable insights into mixing phenomena, initially in terms of equilibrium states, but more recently extended to dynamic conditions.

In the electro-active polymer area, we have concentrated on research involving poly(p-phenylene vinylene) (PPV) and its derivatives, copolymers, and blends. The electrical and, more recently, the non-linear optical properties of

this family of conjugated polymers has proved to be of particular interest and potential applicability. Studies of the morphology of PPV has led to observation of the importance of ultra-structure processing in the development of maximum property sets. Finally, mention may be made of a new technique for studying diffusion in constrained media pores using quasi-elastic light scattering. This has led to advances in the theory of motion of flexible chains under hindered conditions and constitutes a novel method of examining the effect of chain microstructure and architecture on such motions.

VII. DESCRIPTION OF RESEARCH UNDERTAKEN

Some sixty-five (65) manuscripts were published or are in press in the grant period. A selection of results is presented below.

A. Polymer Blends

The dependence of kinetics of crystallization and melting behavior in isotactic polystyrene/poly-o-chlorostyrene-co-p-chlorostyrene blends on temperature, thermal history, and blend compositon has been investigated. The crystallization rate at a given temperature and copolymer composition decreases with increasing copolymer content in the blend when samples are premelted. These effects can be ascribed to the reduction of mobility of the crystallizable chains due to the presence of the copolymer and to the decrease in the number of heterogenous iPS nuclei as a result of the premelting process. The Avrami exponent values and the analysis of the blend morphology indicate that the growth mechanism of the crystals is strongly influenced by thermal treatment. There is no measurable change in the melting temperature of iPS in the blends, with composition indicating that, on the basis of the Flory-Huggins approximation of the thermodynamics of polymer mixing, the net interaction parameter at the melting temperature is close to zero. From the comparison of the phase diagram for the isotactic polystryene-containing blend with that of the atactic-containing blend, it can be concluded that in the amorphous state polystyrene with a regular configuration is slightly less compatible with the P(o-ClS-co-p-ClS) than is polystyrene with random configuration.

In another investigation copolymers of ortho(para)fluorostyrene and ortho(para)bromostyrene with a range of copolymer compositions were prepared by free radical polymerization in toluene solution using azobis(isobutyronitrile). The miscibility and phase behavior of these copolymers in blends with poly(2,6-dimenthyl-1, 4-phenylene oxide) (PPO) have been studied by differential scanning calorimetry. Of the four possible copolymer systems, miscibility was observed only for PPO/poly(o-fluorostyrene-co-p-bromostyrene) blends in which the copolymer contained between 11 and 73 mol% p-bromostyrene. High temperature phase separation in the miscible blends is a function of copolymer composition and of the thermal history. The results can be explained on the basis of the mean field theory of phase behavior for homopolymer-copolymer systems.

In the high temperature area miscibility in the amorphous and the crystalline phases of a blend of poly(oxy-1,4-phenylenoxy-1,4-phenylenecarbonyl-1,4-phenylene)[poly(ether ketone)(PEEK)] and poly(oxy-1,4-phenylenecarbonyl-1,4-phenylene)[poly(ether ketone)(PEK)] has been studied by differential scanning calorimetry (d.s.c.) and wide-angle X-ray diffraction (WAXD). Our results indicate that the polymers cocrystalline on quenching from the melt but do not cocrystallize when other thermal histories are imposed. It can be inferred from results obtained for the crystalline phase that the two polymers are miscible in the amophous phase.

B. Electro- and Optically-Active Polymers

Two new types of p-xylene bis-sulfonium chloride monomers were prepared from cycloaklylene sulfides. The polymerization characteristics of these monomers to form poly(p-xylene sulfonium chlorides), and the thermal elimination reactions of their polymers to poly(p-phenylene vinylene), were compared with those of two monomers prepared from dialkyl sulfides. The cycloalkylene sulfonium chloride monomer polymerized to higher yields and to higher molecular weight polymers, which showed more efficient elimination reactions.

The UV-visible spectrum of 200 nm thick films of poly(p-phenylene vinylene) (PPV) shows three absorption bands, with maxima at 6.12, 5.06, and 3.09 eV. The first two absorption bands are related to localized molecular states while the band at 3.08 eV has a more complicated character with a 1.5 eV bandwidth, a 2.49 eV band edge and a maximum absorption coefficient of 1.5×10^5 cm^{-1}. Semiempirical MO calculations at the INDO and PPP levels and VEH techniques have been applied in an interpretation of the electronic structure and excitations in this electrically conducting polymer. A description of the lower energy excited states of PPV has been achieved using an exciton model in which the polymer chain is treated as an ordered array of identical molecules. The 3.08 eV polymer absorption band is then attributed to an electron excitation spread over several monomer units strongly perturbed by electronic-vibrational coupling.

In addition a versatile technique for preparing electrically conducting polymer blends has been developed which utilizes the soluble precursor approach to conducting polymer synthesis. The water-soluble polymeric sulfonium salt

precursor to poly(p-phenylene vinylene) (PPV), a conducting polymer, has been used to prepare blends of PPV with polyacrylamide (PAcr). These phase-separated blends yield flexible and transparent films of good mechanical properties which can be oxidized with strong electron acceptors (e.g. AsF_5) and thereby exhibit greatly enhanced electrical conductivity. The ultimate conductivity shows an atypical gradual composition dependence, which lies between that of the pure components. These blends can also be thermally stretched in the manner reported for pure PPV. Stretching of the PPV/PAcr blends results in highly efficient PPV chain orientation and greatly augmented conductivity along the stretch axis.

C. Computer Simulation of Mixing

Phase transitions from miscibility to immiscibility were observed in simulations of binary polymer mixtures on a planar square lattice using reptation sampling techniques. The relationship between the phenomenological interaction parameter, χ, and the true molecular interaction energy was followed and the dependence of the number and distribution of heterocontacts in the mixture on the applied heterosegment interaction energy was determined. Deviations from the results of mean-field treatments, which overestimate the number of heterocontacts, were observed even for athermal mixtures. Kinetically driven hysteresis governed by a temperature equilibration time scale was examined for the phase transitions. An important prediction of expansion of the polymer chains in the miscibility region can be made on the basis of the results of chain end-to-end distance calculations.

In addition the behavior of a binary polymer mixture was simulated on a

cubic lattice over both the miscibility and immiscibility regions. The number and distribution of interactions in the mixture were found to be different from the mean-field picture; however, the observed phase behavior agrees with that predicted by the mean-field theory and is not affected by the observed concentration fluctuations. The relationship between the phenomenological x parameter and the heterosegmental interaction energy was investigated. Polymer chains show nearly ideal behavior, even for strongly interacting mixtures; this simplifies the theoretical treatment of polymer mixtures analogous to homopolymer melts.

D. Some Other Studies

The molecular weight dependence of the translational diffusion coefficient of a flexible polymer in a highly interconnected porous glass material has been studied by using dynamic light scattering. Porous silicas, each with different pore radius R_p and porosity Φ, were saturated at equilibrium with dilute solutions of linear polystyrene in a thermodynamically good solvent and examined in a light-scattering spectrometer. Each measurement of diffusion was made entirely inside a single fragment of saturated porous material, thus eliminating effects due to movement of polymer from unbounded solution into the porous material. Diffusion behavior was investigated as a function of the dimensionless variables $\lambda_H \equiv R_H/R_p$ (the size of the polymer relative to the pores) and qR_p (essentially the ratio of pore size to the length over which diffusion is probed), where R_H is the polymer hydrodynamic radius and q the scattering wave vector. Macroscopic diffusion coefficients D_∞, phenomenological coef-

ficients for diffusion over large distances in the porous glass, were obtained from data at low qR_p. The reduction in D_∞ relative to D_0, where D_0 is the diffusivity in unbounded solution, can be satisfactorily interpreted in terms of (1) hydrodynamic interactions of the polymer with the pore walls, which lead to a reduced intrapore diffusivity, D_p, relative to D_0, and (2) the tortuosity of the pore space. At high $\lambda_H(\lambda_H > 0.18)$, the dependence of D_p/D_0 on λ_H is consistent with a transition from non-free-draining to free-draining behavior as is predicted by scaling theories. For these data, the tortuosities (obtained as the inverse of D_∞/D_0 in the limit $\lambda_H = 0$) are related to the pore space geometry and are independent of λ_H.

VIII. PUBLICATIONS

Sixty-five (65) refereed publications have been published or are in press during the period of this report using AFOSR support.

1. Poly. Comm. 28, 236-240 (1987) (with Saburo Akiyama, W.J. MacKnight and Junji Nambu) "Miscibility in Polycarbonate/Poly(styrene-co-methacrylic acid) Blends and the Ternary System Involving Styrene-Butyl Acrylate Block Copolymer".

2. Poly. Comm. 28, 261-262 (1987) (with C.C. Han and R.W. Lenz) "Highly Conducting, Iodine-Doped Copoly(phenylene vinylene)s".

3. J. Polymer Sci. 25, 2531-2540 (1987) (with C. Silvestre, S. Cimmino, and W.J. MacKnight) "Crystallization, Thermal Behavior, and Compatibility in Isotactic Polystyrene/Poly (o-chlorostyrene-co-p-chlorostyrene) Blends".

4. J. Chem. Phys. 87, 6178-6184 (1987) (with J. Obrzut) "Defects in the Electronic structure of poly(p-phenylene vinylene): Electronic Spectra, Electrochemical behavior, and Molecular Orbital calculations"

5. Eur. Polym. J. 24, 123-127 (1988) (with R. Vukovic, G. Bogdanic, V. Kuresevic, and W.J. MacKnight) "Miscibility and Phase Behavior in Poly (2,6-Dimethyl-1,4-phenylene Oxide) and Poly(fluorostyrene-co-bromostyrene) Blends"

6. Macromolecules, 21, 231-234 (1988) (with Gaetano Guerra, Soonja Choe, David J. Williams, and William J. MacKnight) "Fourier Transform Infrared Sprectroscopy of Some Miscible Polybenzimidazole/Polyimide Blends"

7. Poly. Comm., 29, 14-16 (1988) (with Slavka Stankovic, Gaetano Guerra, David J. Williams, and William J. MacKnight) "Miscible Blends of Polybenzimidazole and a Diisocyanate-based polyimide"

8. J.Poly.Sci., Physics Ed., 26, 301-313 (1988) (with Gaetano Guerra, David J. Williams, and William J. MacKnight) "Miscible Polybenzimidazole Blends with a Benzphenone-based Polyimide"

9. J.Applied Polymer Science, 35, 679-693 (1988) (with Gyorgy Banhegui and Peter Hedvig) "Analysis of Temperature-Dependent AC Dielectric Loss Data"

10. Macromolecules, 21, 446-451 (1988) (with Peter Cifra and William J. MacKnight) "Distribution of Interactions in Binary Polymer Mixtures: A Monte Carlo Simulation Study"

11. Cell Biophysics, 11, 91-104 (1987) (with Anna Balazs and William J. MacKnight) "The Aggregation of Reverse Micelles: A Computer Simulation"

12. Brit.Polym.Jrnl., 20, 149-155 (1988) (with C.K. Sham, C.H. Lau, D.J. Williams, and W.J. MacKnight) "Upper Critical Solution Temperature Behavior in Poly(ether ether sulphone)/Poly(ether ether ketone-co-ether ether sulphone) Blends"

13. Nonlinear Optical and Electroactive Polymers, ed. P.N. Prasad and D.R. Ulrich, Plenum Publ. Corp. (1988), 273-280. (with Jan Obrzut) "Optical Properties of Poly(p-Phenylene Vinylene)"

14. Macromolecules 21, 1193-1194 (1988) (with E.J. Beckman, R.S. Porter, W.J. MacKnight, J. Van Hunsel, and R. Koningsveld) "Estimation of Interfacial Fraction in Partially Miscible Polymer Blends from Differential Scanning Calorimetry Measurements"

15. Polymer, 29, 1016-1020 (1988) (with C.K. Sham, G. Guerra and W.J. MacKnight) "Blends of two poly(aryl ether ketones)"

16. J. Appl. Poly. Sci., 36, 243-248 (1988) (with A. Nelson, G. Guerra, D.J. Williams and W.J. MacKnight) "Catalytic Activity of Benzimidazole in the Imidization of Polyamic Acids"

17. J. Polym. Sci., Chem. Ed., 26, 1809-1817 (1988) (with S. Antoun and R.W. Lenz) "Synthesis and Electrical Conductivity of Poly(arylene Vinylene), I. Poly(2,5-Dimethoxyphenylene Vinylene) and Poly(2,5-Dimethylphenylene Vinylene)"

18. SPIE, 878, 123-127 (1988) (with G. Williams and G.S. Attard) "Functional Polymers and Guest-host Polymer Blends for Optical and Electronic Applications"

19. Polymer, 29, 1412-1417 (1988) (with J.M. Machado and R.W. Lenz) "Electrically Conducting Polymer Blends"

20. J. Polym. Sci. Part B: Polymer Physics, 26, 2379-2383 (1988) (with P. Cifra, and W.J. MacKnight) "Computer Simulation of a Binary Polymer Mixture in Three Dimensions"

21. J. Polym. Sci. Part B: Polymer Physics, 26, 2247-2256 (1988) (with J.B. Schlenoff, J.M. Machado, P.J. Glatkowski) "Chemical and Electrochemical Doping in Poly(Paraphenylene Vinylene) Blends"

22. J. Amer. Chem. Soc. 110, No. 21, 728-729 (1988) (with P.M. Lahti, D.A. Modarelli, F.R. Denton, III, R.W. Lenz) "Polymerization of α,α'-Bis(dialkylsulfonio)-p-xylene Dihalides via p-Xylylene Intermediates: Evidence for a Nonradical Mechanism"

23. J. Polym. Sci. Part A: Polymer Chemistry 26, 3241-3249 (1988) (with R.W. Lenz, C.-C. Han, J. Stenger-Smith) "Preparation of Poly(phenylene Vinylene) from Cycloalkylene Sulfonium Salt Monomers and Polymers"

24. Coll. & Polym. Sci. 266, No. 8, 701-715 (1988) (with G. Banhegyi, P. Hedvig) "DC Dielectric Study of Polyethylene/$CaCO_3$ Composites"

25. Polymer, 29, 1940-1942 (1988) (with B.P. Singh and P.N. Prasad) "Third-Order Non-Linear Optical Properties of Oriented Films of Poly(P-Phenylene Vinylene) Investigated by Femtosecond Degenerate Four Wave Mixing"

26. Kem. Ind., 37, 347-349 (1988) "Polymer Blends

27. Macromolecules,21, 3438-3442 (1988) (with M.Masse and H.Ueda) "Miscibility in Chlorinated Polybutadiene/Chlorinated Polyethylene Blends: Effect of Chain Microstructure"

28. J.Polym. Sci: Part B Polym. Physics, 27, 199-203 (1989) (with J.Machado,J.Schlenoff, and F.R.Denton) "Analytical Methods for Molecular Weight Determination of Poly(p-Xylylidene Dialkyl Sulfonium Halide):Degree of Polymerisation of Poly(p-Phenylene Vinylene) Precursors"

29. Comprehensive Polymer Science, Volume 7 (S.L. Aggarwal ed.) Pergamon Press, Oxford p.111-130 (1989) , "Polymer Blends" (with W. MacKnight)

30. Macromolecules, 22, 458-464 (1989) (with M.R.Obrzut) "X-ray Photoelectron Spectroscopy of Neutral and Electrochemically Doped Poly(p-phenylene vinylene)"

31. J.Polym. Sci: Part B Polym. Physics, 27, 469-487 (1989) (with T. Granier, E. L. Thomas and F. Karasz) "Paracrystalline Structure of Poly(paraphenylene vinylene)"

32. Macromolecules, 22, 1220-1231 (1989) (with M. Bishop and K. Langley) "Dynamic Light-Scattering Studies of Polymer Diffusion in Porous Materials: Linear Polystyrene in Porous Glass"

33. Macromolecules, 22, 1964-1973 (1989) (with J. Machado and J. Schlenoff) "Morphology, Doping, and Electrical Properties of Poly(p-phenylenevinylene)/Poly(ethylene oxide) Blends"

34. Synthetic Metals, 29, E109-E114 (1989) (with J. Obrzut and M.J. Orbrzut) "Photoelectron Spectroscopy of Poly(p-Phenylene Vinylene)"

35. Synthetic Metals, 29, E103-E108 (1989) (with J. Obrzut and M.J. Obrzut) "Photoconductivity of Poly(p-Phenylene Vinylene)"

36. Synthetic Metals, 29, E97-E102 (1989) (with J. Grobelny and J. Obrzut) "Solid-State ^{13}C NMR Study of Neutral Insulating and Electrochemically Doped Conducting Poly(p-Phenylene Vinylene)"

37. Netsu Sokutei, 16, (2), 65-75 (1989) (with Y. Maeda and W. MacKnight) "Effect of Pressure on the Miscibility of Polymer Blends by High Pressure Differential Thermal Analysis"

38. Journal of Applied Physics, "Oscillator Model of Piezoelectricity in Hydrated Polymers" (in press).

39. *Proceedings of the Electrochemical Society, 1987, "Improved Mass Transport in Rechargeable Electrodes Employing Conducting Polymer Blends" (in press).

40. New Polymeric Materials, "A Continuous Process for Preparing Highly Conducting, Uniaxially Oriented Poly(Phenylene Vinylene) Film" (in press).

41. Colloid and Polymer Science, "Comment on the 'Dielectric Properties of LDPE/Ny6 Blends' by Mantia et al" (in press).

42. Macromolecules, "Computer Simulation of Copolymer-Copolymer and Copolymer-Homopolymer Mixtures with a Single Interaction Energy" (in press).

43. J. Polym. Sci., "Crystalline Phases of Electrically Conductive Poly(p-Phenylene Vinylene)" (in press).

44. Speciality Polymers, "Hydrogen Bonding in Polybenzimidazole-Polyimide Systems: A FTIR Investigation" (in press).

45. Phys. Rev. B, "Temperature Dependence of the Electrical Conductivity of AsF_5 Doped Poly(p-Phenylene Vinylene)" (in press).

46. Makromolekulare Chemie, "Structural Characterization of Vinylidene Fluoride-Vinyl Fluoride Copolymers" (in press).

47. Polym. Eng. and Sci., "Computer Vision Methods for the Study of Spinodal Decomposition in Polymer Blends" (in press).

48. Polymer, "Anisotropic Mechanical Properties of Uniaxially Oriented Electrically Conducting Poly(p-phenylene vinylene)" (in press).

49. J. Appl. Polym. Sci. "Dielectric Relaxation Properties of Polypropylene-Polyurethane Composites" (in press).

50. Polymer Composites, "Thermal and Electrical Properties of Some Epoxy Based urethane Composites" (Banhegyi) Composites" (in press).

51. Macromolecules, "Short Range Fluctuations in Spinodal Decomposition of Binary Polymer Mixture" (in press).

52. Brit. Polym. J. "Processing and Characterization of a Biaxially Oriented Conducting Polymer" (in press).

53. Biennial Polymer Symposium, "Thermal and Phase Behavior in Miscible Polybenzimidazole/Polyetherimide Blends" (in press).

54. Macromolekulare Chemie, "Miscibility Behavior in Blends of Poly(2,6-Dimethylphenylene oxide) and Random or Block Styrene-Methylmethacrylate Copolymers" (in press).

55. Materials Research Society Proceedings, "Lattice Simulation of Mixtures of Flexible and Semi-Flexible Chains" (in press).

56. J. Polymer Science, "Phototransport in Poly(p-Phenylene Vinylene)" (in press).

57. J. of Matls. Sci. "Crystal Morphology in Pristine Doped Films of Poly(p-Phenylene Vinylene)" (in press).

58. Macromolecules, "Diffusion of Weakly Confined Star and Linear Polymers and Strongly Confined Linear Polymers in a Porous Material" (in press).

59. Makromolekulare Chemie "Miscibility in Blends of Modified Polyolefins" (in press).

60. Third International Conference on Polyimides, "Thermal and Spectroscopic Behavior in Miscible Polybenzimidazole/Polyimide Blends" (in press).

61. Polymer, "Large Optical Birefringence in Poly-P-Phenylene Vinylene Films Measured by Optical Wave Guide Techniques" (in press).

62. Macromolecules, "Hydrogen Bonding in Polybenzimidazole/Polyetherimide Blends: A Spectroscopic Study" (in press).

63. European Polymer Journal, "Electrical Conductivity of Iodine Doped Poly(butadiene-co-2-vinyl pyridine) Diblock Copolymers" (in press).

64. Polymer, "The Effects of Concentration on Partitioning of Flexible Chains into Pores" (in press).

65. Macromolecules, "Hindered Diffusion of Polystyrene in Controlled Pore Glasses" (in press).

PATENTS: Applications, U.S. and Foreign

1. F.E.Karasz, et al: "High Performance Blends of Aromatic Polyimides with Aromatic Polyethersulfones"

2. F.E.Karasz, et al: "Third Order Non-Linear Optically Active Composites"