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James L. White and Thein Kyu

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The present study consists essentially two major parts: (i) processing and orientation development of Kevlar fiber composites, and (ii) preparation and characterization of polyamide molecular composites. In the first part, it has been proposed to characterize the structure and orientation development of anisotropic particulates such as Kevlar chopped fibers, minerals, among others, in thermoplastic plastics. The

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effects of processing techniques and processing conditions on the fiber and matrix orientation have been established on the basis of the wide-angle X-ray (WAXD) pole figure analysis. In the second part, various molecular composites have been prepared by mixing poly-p-phenylene terephthalamide (PPTA) and various nylons. Miscibility between PPTA and nylon matrices has been examined with emphasis on the role of cross-hydrogen bonding. The interaction parameter χ has been determined for various pairs of PPTA and crystalline nylons. The phenomenon of phase segregation and its kinetics has been investigated for PPTA/amorphous nylon mixtures. The melt rheology and mechanical behavior of PPTA/nylon 6 and 66 molecular and particulate composites have been evaluated.

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Final Report

on

STRUCTURAL CHARACTERIZATION AND MECHANICS OF CHOPPED FIBER AND MOLECULAR COMPOSITES

Submitted To

U.S. Army Research Office

by

James L. White
Thein Kyu

Polymer Engineering Center
The University of Akron
Akron, Ohio 44325-0301

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PROBLEM STUDIED

The present study consists essentially two major parts: (i) processing and orientation development of Kevlar fiber composites, and (ii) preparation and characterization of polyamide molecular composites. In the first part, it has been proposed to characterize the structure and orientation development of anisotropic particulates such as Kevlar chopped fibers, minerals, among others, in thermoplastic plastics. The effects of processing techniques and processing conditions on the fiber and matrix orientation have been established on the basis of the wide-angle X-ray (WAXD) pole figure analysis. In the second part, various molecular composites have been prepared by mixing poly-p-phenylene terephthalamide (PPTA) and various nylons. Miscibility between PPTA and nylon matrices has been examined with emphasis on the role of cross-hydrogen bonding. The interaction parameter χ has been determined for various pairs of PPTA and crystalline nylons. The phenomenon of phase segregation and its kinetics has been investigated for PPTA/amorphous nylon mixtures. The melt rheology and mechanical behavior of PPTA/nylon 6 and 66 molecular and particulate composites have been evaluated.

SUMMARY OF RESEARCH ACCOMPLISHMENTS

A novel technique for determining the fiber orientation in chopped fiber reinforced thermoplastics using WAXD pole figures has been successfully developed and employed in extrudates from various dies, injection molded parts, and rolled Kevlar/polycarbonate composites. However, it was found to be difficult in the case of Kevlar/nylon fiber composites because of the similarity of their unit cell structures. When inorganic minerals such as talc or mica were compounded in poly-

ethylene, the WAXD technique can be applied to the characterization of mineral particulate orientations as a function of process conditions.

We found that the coagulation process is critical for the preparation of molecular composites. In general, most molecular composites may result in phase separation during coagulation due to the large disparity of molecular topology between stiff PPTA and flexible nylons. However, we overcome this problem from the kinetic standpoint by rapidly coagulating the molecular composites in non-solvent. The mixture thus prepared is thermodynamically unstable and undergoes thermally induced phase separation.

We also notice the occurrence of cross-hydrogen bonding between PPTA and nylons which not only depresses the melting point of nylon crystals but also acts in favor of polymer miscibility. The interaction parameter χ has been determined to be negative for polyamide molecular composites. The values range from -1 to -0.05 and vary in the order of N 6, 66, 11, and 12. However, this hydrogen bonding is not strong enough to prevent phase segregation upon heating above the crystal melting temperature.

As a consequence, the melting processing of most molecular composites yields phase separated two-phase particulate systems, except for extreme compositions, say less than 10 wt% PPTA contents. The mechanical properties of low PPTA content molecular composites, namely modulus and tensile strength, show two-fold improvement relative to those of pure nylons. Higher PPTA composites exhibit inferior properties probably due to phase segregation. Upon annealing the molecular composites at 70°C, the modulus has increased five-fold, but there is no improvement in the tensile strength.

FUTURE WORK AND STUDIES TO BE COMPLETED

We are unable to compare the structure and mechanical properties of choppcd fiber and molecular composites due to the lack of common polymer matrix. We recently obtained a series of amorphous nylons including Zytel 330, (DuPont Co.), a polycondensate of hexamethylene diamine and the mixture of terephthalic and isophthalic acids, to be suitable for both studies of chopped fiber and molecular composites. The miscibility study of PPTA and amorphous nylon is already completed. However, the specimens are too small and too thin to be used for mechanical tests. A solution processing device has been developed for preparing thicker molecular composite films which needs some modifications at the present time.

The use of amorphous nylons will allow us to directly compare the structural (and PPTA orientation) and mechanical characteristics of chopped fiber and molecular composite material. The PPTA diffraction peaks should be discernible in these materials.

SCIENTIFIC PERSONNEL

1. Dr. Tsuey I. Chen, post doctoral research fellow, permanent resident, June 1986 to March 1989, joined Goodyear in April 1989.

2. Dr. T. Kikutani, visiting scientist, Japanese, Sept. 1986 to Aug. 1987, Tokyo Institute Technology.

3. Mr. Ho-Sung Park, graduate student, U.S. Citizen, Sept. 1985 to June 1988, joined BASF (North Carolina) in July 1988 and will defend his Ph.D. Dissertation in May 1989.

4. Mr. Soon-Ho Lim, graduate student, Korean, Sept. 1987 to present, Ph.D. Candidate.

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5. "Miscibility Studies of Poly-p-Phenylene Terephthalamide/Amorphous Nylon Blends," T.I. Chen and T. Kyu, Polym. Commun., submitted.

6. "Development and Characterization of Anisotropic Disc and Fibrous Particles in a Thermoplastic Matrix," S.H. Lim and J.L. White, J. Rheology, submitted.

7. "Orientation and Morphology of Solution Processed Poly-p-Phenylene Terephthalamide/Nylons Composites," H.S. Park, T. Kyu, R. Brzoskowski, and J.L. White, in preparation.

8. "Kinetics of Phase Segregation in Poly-p-Phenylene Terephthalamide/Amorphous Nylon Blends," J.C. Yang, T.I. Chen, and T. Kyu, in preparation.