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

The development of microstructure in PEEK and its short-fiber composites and the effects of such microstructure on mechanical failure were studied. A small-scale injection-molding machine installed in an x-ray diffractometer, was used to follow the development of crystallinity. For neat polymer, increasing the rate of injection increased the crystallization rate. Fibers sometimes slowed crystallization, due to polymer relaxation between the fibers. Fracture behavior in oriented PEEK was found to be highly anisotropy in the elastic modulus. Spherulite size was controlled by self-seeding. The fracture toughness was found to increase with spherulite size.

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FAILURE BEHAVIOR OF PEEK
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Final Report

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SUMMARY OF RESEARCH

The research was concerned generally with the development of microstructure in PEEK and its short-fiber composites and with the effect of that microstructure on mechanical failure. Three subareas were investigated: (1) the effect of flow conditions during molding on crystallization. (2) the effect of orientation on fracture. (3) the effect of spherulite-level microstructure on mechanical failure.

In the work on flow conditions and crystallization, a small-scale injection-molding apparatus was constructed and placed in the beam path of an x-ray diffractometer. Polymer or composite was injected at a given flow rate and x-ray diffraction scans taken periodically during solidification. Although no measurable polymer orientation could be observed, in the neat polymer a significant effect of flow rate on crystallization rate was measured, higher flow rates inducing higher crystallization rates. The effect of both glass and graphite fibers is to reduce the crystallization rate relative to neat resins at higher crystallization temperatures. Graphite fibers however, increase the rate of crystallization at lower temperatures. The high temperature rate reduction is attributed to melt relaxation between the fibers.

Rolltruded and melt-oriented specimens were used in the orientation study. For the neat resin, a significant increase in fracture strength and in toughness parallel to the mean fiber direction. A relatively small decrease in the transverse direction is measured. The difference in macroscopic failure measurements parallel and perpendicular to the flow are mirrored in fractographic differences. For the filled systems, an analogous difference---but here due to fiber orientation---is seen.

In the spherulite-level study, self-seeding was used to vary the mean spherulite diameter while leaving other parameters constant. The self-seeding was done by changing the highest melt temperature experienced prior to crystallization. For both relatively high and relatively low molecular weight PEEK, the fracture toughness decreases with increasing melt-treatment temperature---that is, with increasing crystallization temperature. For lower molecular weight material, large spherulites are formed. In this case, fractographs indicate that the denser core volume of a spherulite pulls out of the surrounding material. For higher molecular weight PEEK, very small, nascent spherulites form. In this case, fractographs suggest an interspherulitic fracture path.

List of Publications from DAAG29-85-K-0042

- J.-N. Chu, G.-M. Wu and J.M. Schultz, "Effects of Microstructure on Failure in PEEK and Its Short-Fiber Composites," Proc. Amer. Soc. Composites, 3rd Tech. Conf. Seattle, 1988.
- A.K. Mishra and J.M. Schultz, "Effect of Flow Rate and Temperature on Crystallization Kinetics, Crystallinity Index and Elastic Modulus of PEEK," Journal of Applied Polymer Science, in press.
- G.-M. Wu and J.M. Schultz, "Fracture Behavior of Oriented Poly (Ether-Ether Ketone) (PEEK)," Polymer Engineering and Science, 29, 405-414 (1989).
- J.-N. Chu and J.M. Schultz, "The Influence of Microstructure on the Failure Behavior of PEEK," Journal of Materials Science, in press.
- G.-M. Wu and J.M. Schultz, "Processing, Microstructure and Failure Behavior in Short Fiber Reinforced PEEK Composites," submitted to Polymer Composites.
- A.K. Mishra and J.M. Schultz, "Effect of Flow Rate on Crystallization Kinetics of PEEK," SPEIANTEC '89 (1989), pp. 1430-1433.

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