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DEFORMATION BEHAVIOR OF ISOTACTIC POLYSTYRENE

Julie K. Anderson, Robert E. Cohen, Ali S. Argon, Dept.
of Chemical Engineering, MIT, Cambridge, MA 02139

The deformation behavior of a model semi-crystalline polymer with a well-known rhombohedral crystal structure has been examined. As a direct consequence of its slow crystallization rate, isotactic polystyrene, the stereoregular analog of atactic polystyrene, may be prepared as an amorphous glass or as a semi-crystalline polymer with a desired morphology. Study of the crystal structure prior to and following uniaxial compression is accomplished through the use of a microdiffractometer, with a beam size small enough to permit examination of different regions of the deformed cylinder. Changes in both the mechanical deformation behavior and the crystalline scattering pattern have been examined to elucidate the effects of the crystalline component of isotactic polystyrene upon the overall properties of the polymer.

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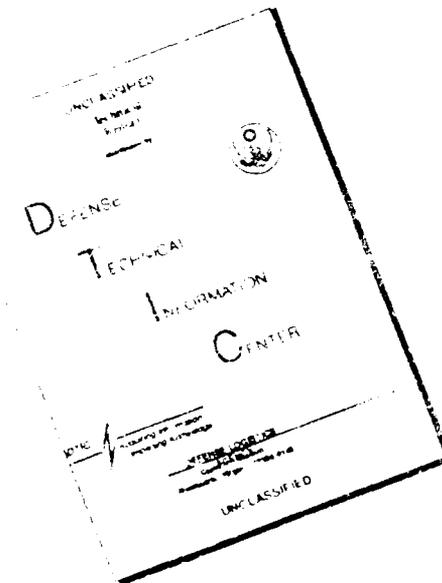
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DEFORMATION BEHAVIOR OF ISOTACTIC POLYSTYRENE

J.K. Anderson, R.E. Cohen, and A.S. Argon
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139

INTRODUCTION

The use of semi-crystalline polymers, which contain both amorphous and crystalline components, provides improved heat and chemical resistance and increased stiffness over wholly amorphous polymers. Semi-crystalline polymers such as nylon and polyethylene are produced in bulk quantities for a wide variety of uses. However, the rapid crystallization rates of these polymers, as well as the complex crystal structure of nylon, where two forms of crystallites occur simultaneously, present additional complications in the study of the effect of crystallinity upon polymer deformation behavior.

Isotactic polystyrene (iPS) was selected as a "model semi-crystalline polymer" for this study. Although its extreme brittleness under tensile loading and difficult synthesis preclude large commercial production of iPS, it is an extremely useful material for experimental study.

Even at the temperature of the maximum in the crystal growth rate curve, the growth rate for iPS is orders of magnitude less than that observed in many other semi-crystalline polymers. This offers certain advantages in the present experimental study. The slow spherulitic growth rate of iPS permits formation of a totally amorphous glass by quenching the melt to room temperature (1) and the development of samples of controlled crystalline content by the imposition of certain thermal histories. Samples with large, isolated spherulites surrounded by a glassy amorphous matrix may be prepared.

The deformation behavior of amorphous atactic polystyrene has been well characterized, but little detailed information is available on the behavior of the wholly amorphous iPS glass. The preparation of samples with desired morphologies will allow the determination of the effect of crystallinity upon deformation behavior of iPS.

In addition to the fortuitous slow growth rate of crystallites in iPS, the crystal structure itself presents several important advantages. The rhombohedral unit cell is well-characterized (1). In contrast to many polymers, iPS acts almost like a metal crystal, with several orders of reflection observable in a wide-angle x-ray diffraction pattern (2). This is an advantage in analyzing the effects of deformation upon the polymer structure, as changes in

several peaks may be examined. The high glass transition temperature of iPS (100°C) is also advantageous because imposed deformations remain fixed and are available for convenient later analysis at room temperature.

EXPERIMENTAL PROCEDURE

The atactic and isotactic polystyrenes used in this work were obtained from Polysciences, Dow Chemical Company, and by synthesis in this laboratory. The iPS was first dissolved in refluxing toluene and then precipitated into methanol to remove any impurities. After filtration, the powder was dried for 24 hours in vacuo at 100°C. Analysis by high-temperature GPC, DSC, and C¹³ NMR was conducted to characterize the iPS both before and after the precipitation.

The cylindrical samples of nominal dimensions 3 mm diameter and 4 mm height were compression molded. A temperature controlled fluidized bath (Techne, Model SBL1) was used for crystallization of the amorphous glass to desired levels. The crystalline content was measured either by DSC, density (3), or by X-ray diffraction (2). An Instron Model 1122 tensile testing machine fitted with a custom compression testing cell and equipped with an environmental chamber was used to produce the deformation. X-ray measurements were conducted both prior to and following the compression to monitor deformation-induced changes.

A Rigaku high brilliance rotating anode X-ray generator with a microdiffractometer attachment, capable of producing a beam of 100 microns or as small as 30 microns, was used for the majority of the diffraction studies. The small beam provided the opportunity not only to study much smaller samples than a conventional 1 cm by 1 mm beam, but to study different areas of the same sample.

X-RAY DIFFRACTION

In the microdiffractometer, the ring-shaped slit of the detector moves parallel to the incident x-ray beam. The design of this detector permits the interception of the entire Debye cone, and results in greater recording of diffracted intensity than a more traditional scintillation counter and slit apparatus. The governing equation for this setup is:

$$L = R \cot 2\theta$$

where L is the distance from the sample to the detector, R is the radius of the ring-shaped receiving slit, and 2θ is the diffraction angle.



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RESULTS AND DISCUSSION

The spherulite growth rate of the precipitated IPS was studied to establish the desired thermal history of the samples. Figure 1 is a plot of log growth rate vs. crystallization temperature. The plot shows a maximum at 175°C. At the higher temperatures, nucleation is retarded, permitting the growth of fewer, more isolated spherulites, so the range of 175 to 185°C, where the linear rate of growth is still near the maximum of the curve, was selected as the crystallization region for our work.

Compression tests on both samples of atactic and isotactic polystyrenes have been conducted and representative curves are shown in Figure 2. Stress-strain analysis of these samples indicates qualitative agreement between the behavior of atactic polystyrene and amorphous isotactic polystyrene.

A typical wide-angle x-ray diffraction trace of Intensity vs. 2θ angle is shown in Figure 3. The Miller indices for the individual planes are shown above the peaks. X-ray scattering patterns collected using the microdiffractometer have been obtained on both the uncompressed and compressed samples of semi-crystalline IPS. Analysis of the observed changes in the diffraction patterns and the implications of this upon the structure of IPS is ongoing.

Further details of the experimental findings will be presented at the symposium.

ACKNOWLEDGEMENTS

We would like to acknowledge Luigi Gazzaniga for providing some samples of IPS. The research was supported by DARPA/ONR Contract N00014-86-K-0768, and a DuPont PhD Fellowship to JKA. For the latter we are grateful to Dr. Bennett Epstein.

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Log Growth Rate vs. Temperature

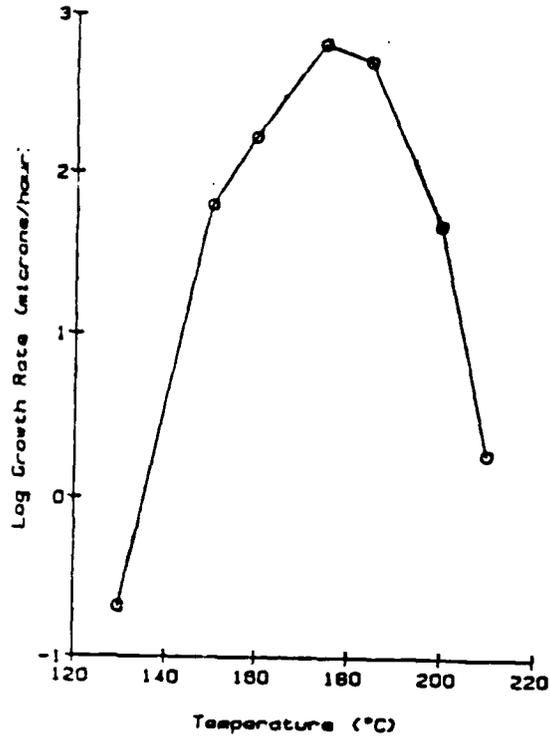


Figure 1. Log Growth Rate vs. Temperature for Spherulites of isotactic Polystyrene.

UNIAXIAL COMPRESSION OF POLYSTYRENE

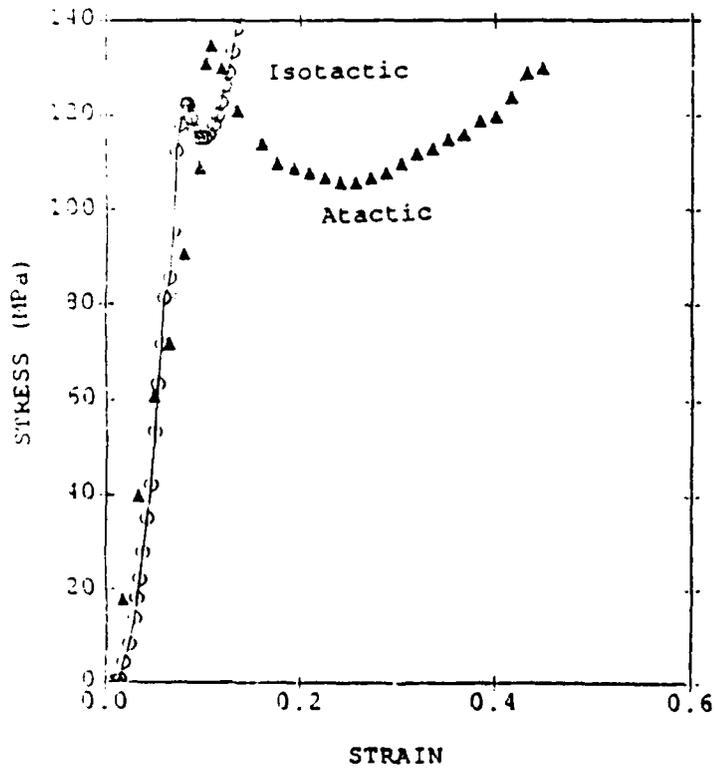


Figure 2. Stress-Strain curves for Uniaxial Compression of Isotactic and Atactic Polystyrene.

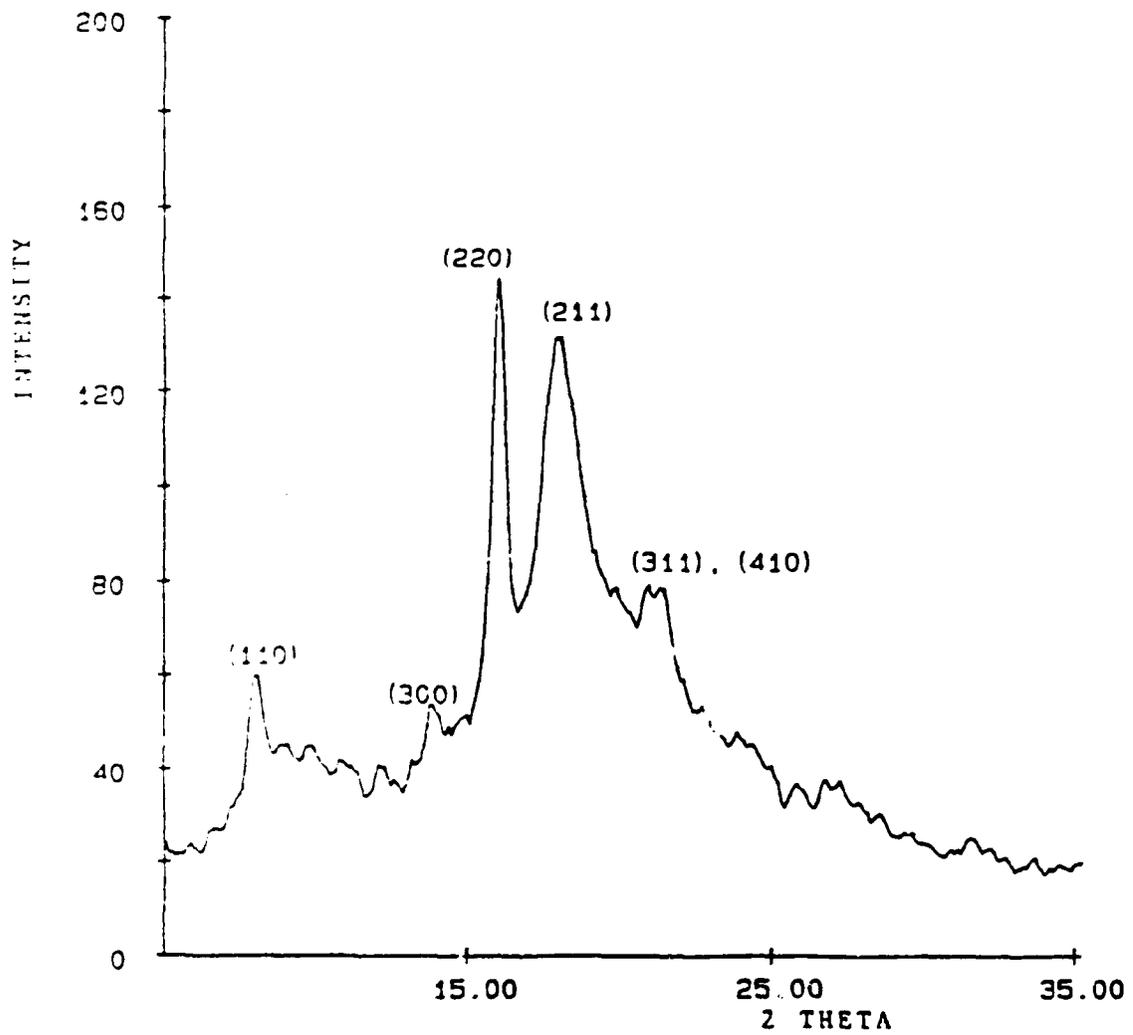


Figure 3. Wide-angle X-ray Diffraction Pattern of Semi-Crystalline Isotactic Polystyrene.