

ELECTRON DIFFRACTION CHARACTERIZATION OF NASCENT CONDENSATION POLYMERS

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Several years ago we developed [1], and have since applied to many condensation polymers, an apparently general technique of simultaneous polymerization/ crystallization to prepare extended chain lamellar single disclination domains and single crystals. The polymerization is done in confined thin films, usually in a melt and often at a temperature below T_m of the pure monomer. The lamellae, in most cases, are ca. 100 Å thick, regardless of the time and temperature of polymerization and thus are ideal for electron diffraction, generally yielding [001] patterns. Characterization of the crystal structure requires obtaining other zones; tilting of the crystals, epitaxial crystallization [2] and whisker growth by polymerization/crystallization in solution [3, 4] have been used. The use of sheared (fiber symmetry) samples is less satisfactory. Cerius² has then been used to model the crystal structures and calculate the diffraction patterns for comparison with those observed, with examples to be given of modifications required to obtain "agreement". Examples to be discussed include poly(*p*-oxybenzoate) [3], poly(*p*-phenylene terephthalate) [5], poly(2,6-oxynaphthoate) [4], bromo-substituted polyesters [6], (all liquid crystal polymers), poly(terephthalic anhydride) [7] (an intractable polymer), and poly(ethylene terephthalate) [8] and poly(ethylene naphthalate) [9] (both flexible backbone polymers). For the latter lamellae crystallized from the melt of pre-polymerized polymer was also used, solution grown single crystals being another appropriate sample for ED. We suggest that the crystal structures obtained from the above nascent crystals represent a "more perfect" crystal than can be obtained through any thermal and/or strain processing of pre-polymerized polymer.

References

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