
ID 1089

Transcrystalline Isotactic Polypropylene Studied by Synchrotron Microbeam X-Ray Diffraction

Eric Assouline^{1,2}, Ellen Wachtel², Arnold Lustiger³
Stephane Grigull,⁴ H. Daniel Wagner² and Gad Marom¹

¹Casali Institute of Applied Chemistry, The Hebrew University of Jerusalem,
Jerusalem 91904, Israel

²Department of Materials and Interfaces, The Weizmann Institute of Science,
Rehovot 76100, Israel

³ExxonMobil Research and Engineering, Route 22 East, Annandale, NJ 08801, USA

⁴ESRF, BP 220, Grenoble Cedex, F-38 043, France

Keywords: polypropylene, transcrystallinity, morphology

Fibers inserted in thin films of isotactic polypropylene (iPP) may nucleate and induce a crystalline morphology at their surface, which is different from that in the bulk: the transcrystalline (tc) layer. Here we focus on the detailed morphology of the monoclinic α iPP tc layer induced by aramid fibers and on its morphological changes under tensile stress.

The Morphology of α Polypropylene Transcrystallinity

We used a finely collimated X-ray beam ($8 \mu\text{m} * 40 \mu\text{m}$) on beamline ID11 at ESRF. X-ray patterns were recorded with $8 \mu\text{m}$ steps sampling the tc layer in the radial direction from the fiber surface to the bulk. The positions of the centers of the arcs reveal a progressive change of the orientation of the crystallites as a function of the distance from the fiber. Very close to the fiber (Fig. 1a), the (110) reflection consists of two meridional arcs whereas the (040) arcs are equatorial. In Fig. 1b, it is clearly seen that the (110) arcs rotate from the meridian to the equatorial plane while the (040) arcs rotate from the equatorial plane to the meridian. In Fig. 1c, the rotation of the reciprocal lattice vectors saturates at large distance from the fiber. In Fig. 1d the X-ray pattern performed in the bulk consists of full, uniform intensity rings.

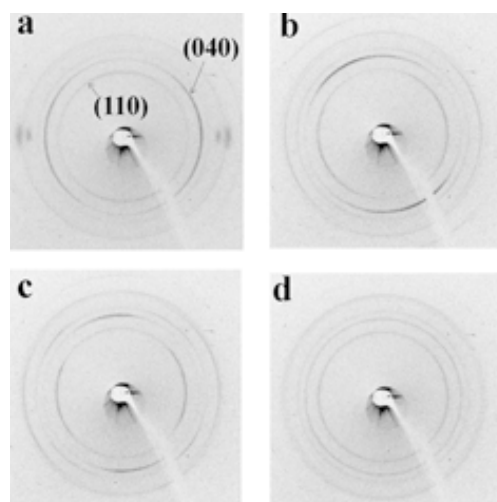


Fig.1 X-ray diffraction of α iPP transcrystallinity (a) initial position, near the fiber surface; (b) $16 \mu\text{m}$ from the initial position; (c) $32 \mu\text{m}$ from the initial position; (d) $56 \mu\text{m}$ from the initial position, in the bulk. Fiber position vertical

X-ray diffraction experiments on aramid / α iPP microcomposites described here demonstrate a dramatic change in the orientation of the polypropylene lamellae as they grow radially outwards from the fiber surface. We suggest that the parent lamellae nucleate at the fiber surface with the crystallite c - axes parallel to the fiber axis, twist one quarter turn about the parent a^* - axis within an approximate distance of $25\ \mu\text{m}$ and then continue to grow without further twisting. The two end-conformations are shown in Fig. 2.

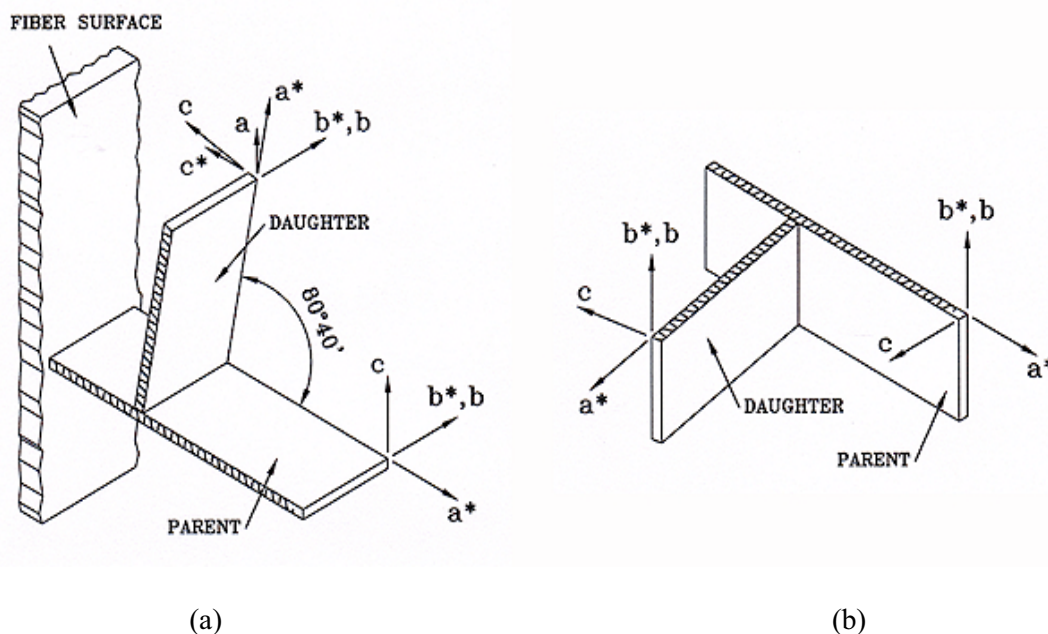


Fig. 2 Models of transcrystalline morphology (a) close to the fiber; (b) at a distance greater than $25\ \mu\text{m}$ from the fiber after twisting of the lamellae around the parent a^* -axis

α Polypropylene Transcrystallinity Under Tensile Stress

The tc sample was held in an Instron 25kN servohydraulic testing machine. For each increment in strain, X-ray diffraction patterns were recorded at $5\ \mu\text{m}$ steps sampling in the direction perpendicular to the fiber axis. Scanning proceeded from close to one tc boundary towards the other tc boundary across the fiber, 20 X-ray diffraction patterns in all (each with an exposure time of 30 s). At a given distance from the fiber, the X-ray patterns remained unchanged, meaning that the crystalline phase is insensitive to the applied tensile stress. The dimensions of the crystal lattice are not affected by the load and orientation of the lamellae is not modified, probably because the polymer chains are affixed to the fiber. The strain apparently takes place mainly in the amorphous phase. The chain axes in the parent lamellae in the vicinity of the fibre thus remain parallel to the fiber. This should improve the tensile properties of the composite, especially at high volume fraction of fibers, as has been shown for Nylon 66 based composites.

Similar experiments performed with spherulites gave different results: under tension the lamellae acquire orientation since they can react to the amorphous flow.