



	Experiment title: An in-situ kinetic study on the (co)crystallization and phase transformations as an influence of pressure and temperature.	Experiment number: SC - 566
Beamline: ID11-BL2	Date of experiment: from: 20-4-1999 to: 26-4-1999	Date of report: 18-08-1999
Shifts: 12	Local contact(s): Anne Terry	<i>Received at ESRF:</i>
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Report:

High pressure experiments were carried out with ethylene-octene copolymers having 5.2 mole% (JW1120) and 8.0 mole% (JW1121) octene-1 content respectively.

Solid state amorphization i.e., Pressure induced amorphization at room temperature was observed in both the samples (Figure 1). A crystal to amorphous phase transition with increasing pressure at room temperature was observed. The amorphous structure resembles melt. The monoclinic crystals seem to arise as a consequence of shear and disappear on releasing pressure.

Some interesting results were observed in terms of hexagonal phase.

After treating the sample through a specific pressure-temperature cycle (pressure upto 4kbars and temperature nearly 220°C), hexagonal phase at room temperature and elevated pressure was observed (Figure 2). The (110) orthorhombic reflection splits up into a (100) hexagonal reflection. The sample crystallized into a three phase structure consisting of an amorphous part, hexagonal and orthorhombic crystals. The bulky side chains (octene-1) which otherwise tend to remain outside the crystal lattice seem to get accommodated within the crystal in hexagonal phase. Thus the introduction of defects within the main chain stabilizes the hexagonal phase at room temperature (Figure 3). With increasing mole% of octene-1 the amorphous density increases and so does the amorphization with pressure. This idea needs to be strengthened with more experiments.

In contrast to LPE where the hexagonal mesophase is metastable at room temperature, the side chain defects in ethylene copolymers could stabilize the hexagonal phase at low temperature.

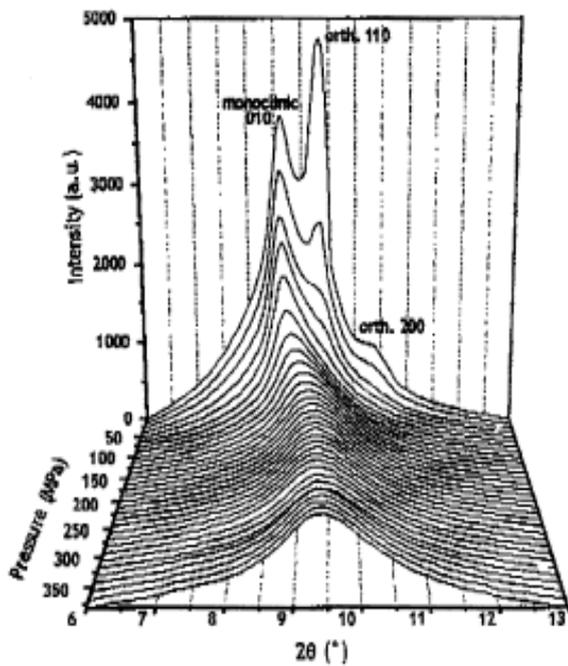


Figure 1.

Figure 1. Diffraction patterns of JW1121 during isothermal pressure increase from 5 to 380 MPa at 28°C. The orthorhombic reflections disappear around 155MPa while the monoclinic reflection shifts to higher angles as a result of compression. The applied pressure possibly destroys the initial (orthorhombic) crystalline structure and converts the crystals into an amorphous –like state obviating the need for going firstly through a “real melt state”.

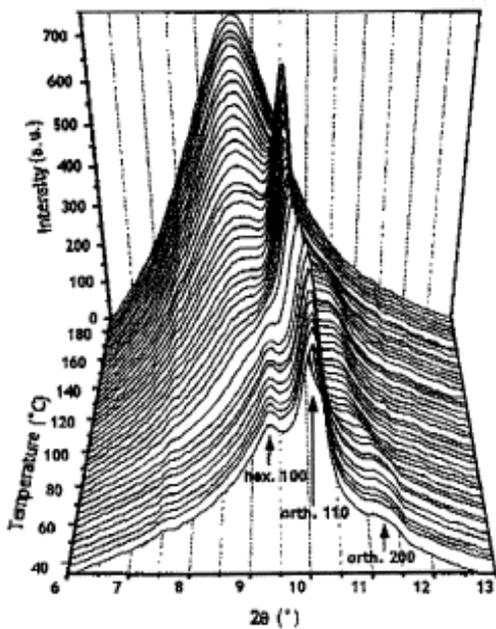


Figure 2.

Figure 2. Diffraction patterns of JW1121 during isobaric cooling at 4°C/min from 190°C to 34°C at 370 MPa. The side chains of octene-1 seem to get accommodated within the crystal in the hexagonal phase.

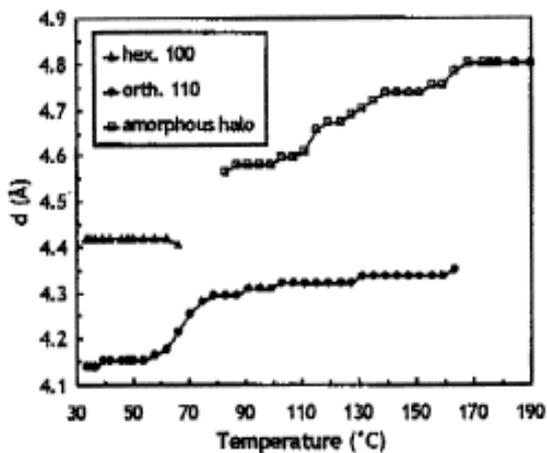


Figure 3.

Figure 3. Spacings of JW1121 during isobaric cooling at 4°C/min from 190°C to 34°C at 370 MPa. The existence of hexagonal phase at lower temperatures is observed.