



	Experiment title: Deformation Behaviour and Fracture properties of Polypropylenes: Influence of Chemical Architecture	Experiment number: SC-1099
Beamline: ID-13	Date of experiment: from: 27-Apr.-05 to: 30-Apr.-05	Date of report: 10-July-06
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1. Introduction

The fracture properties of polymers are of considerable interest as they dictate service life to large extent. Consequently, many studies have attempted to connect the structure of polymers at nanometre length scales with their final properties at a macroscopic level. X-ray scattering is capable of probing nanoscopic structure during macroscopic deformation, a capability which is relatively unique. As a result, this technique is ideal for establishing structure-properties relationships within these materials. In the present study, small angle X-ray scattering is performed using a microfocused synchrotron radiation source to investigate the damage area ahead and around the crack tip in two grades of polypropylene: a typical isotactic one (PPhom), and polypropylene-ethylene copolymer (PPco). The results reveal significant plastic deformation within these regions. This is accompanied by the formation of voids whose geometry is dependent upon their position relative to the crack tip. The results also reveal significant differences in the plastic deformation behaviour between the two different polypropylene grades.

2. Experimental

Two polypropylene grades were used in the present study. The first grade was isotactic polypropylene with a density 0.900 g/cm³, a melt flow index of 0.5 g/10min (190°C, 5 kg) and 99.6% tacticity as determined by NMR. The second grade was poly(propylene-co-ethylene) copolymer with a density of 0.897 g/cm³ and a melt flow index of 0.5 g/10min (190°C, 5 kg). The NMR experiments showed that the ethylene content in the PPco was 5 mol%, and also that ethylene is randomly distributed in the polymer chain. Both polymers were pressed into disk shaped specimens with a diameter of 6 mm and a thickness of 1 mm. These were subsequently machined with two notches and a ligament length of approximately 0.9 mm. The sample was mounted on a custom-made deformation rig, details of which are provided elsewhere [1]. This was attached directly to the x/y/z translation stage of the ESRF ID13 beamline. For deformation, a 0.1 µm/s crosshead speed was used until sample fracture occurred. During stress application, a microscope coupled to a video camera collected images of the sample *in situ*.

3. Results and Discussion

During the application of load, the process and plastic deformation zones begin to form. At this time, the material experiences significant structural change, as shown by the evolution in SAXS in Figure 1. This shows selected SAXS patterns representing various deformation levels for both PPhom (Fig. 1a) and PPco (Fig. 1b), obtained sequentially from a fixed point on the sample. In the initial stages of deformation, the total scattering intensity can be observed to increase rapidly. This is particularly evident by comparing patterns C and D in Figure 2 which are both shown using the same scale. This increase initially is slightly anisotropic with a preferred orientation nearly normal to the stress field and progressively becomes more isotropic (pattern D). The high increase in intensity indicates that heterogeneities are formed in the material. Such features are consistent with the formation of cavities within the plastic deformation zone due to stress-induced negative pressure. As deformation continues and local stresses at the beam position increase, the SAXS patterns

become strongly anisotropic (oriented). This indicates that the existing cavities become elongated and oriented within the material. In these patterns the SAXS signal is perpendicular to the applied stress direction.

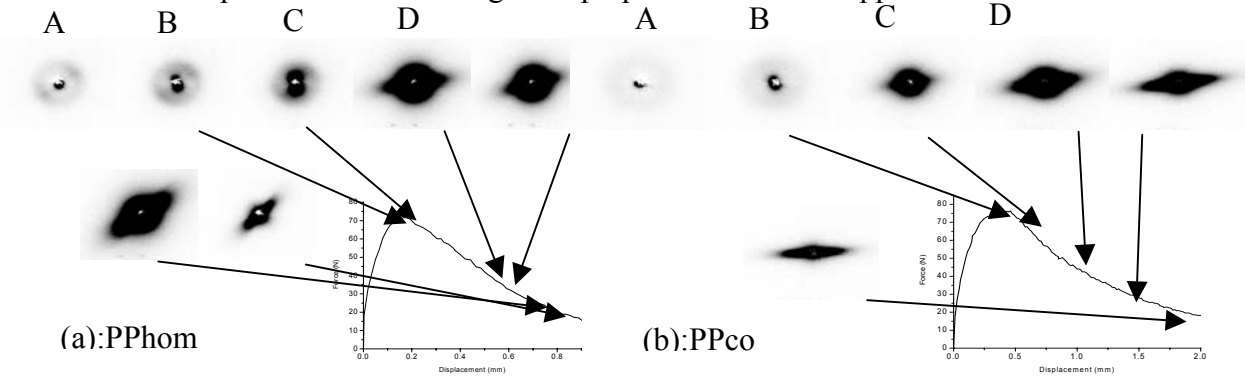


Figure 1: 2D SAXS patterns of the PP polymers as the test progresses

Figure 2 shows force-displacement diagram is combined with the total integrated intensity. As seen in Figure 2, the SAXS intensity begin to increase significantly just after the yield point for both PP grades. This corresponds to the point at which the ligament deforms plastically along its length just prior to crack initiation. The interesting aspect seen in Figure 2 is that the homopolymer yields at much lower displacements compared with the copolymer. In addition, the total integrated intensity is much higher than the one for the copolymer indicating possibly a larger density of nanocracks in the homopolymer. It should be noted that for a more quantitative description the scattering invariant needs to be calculated rather than the total integrated intensity, but such calculation is very complicated in the case of oriented SAXS patterns, such as those in the present study. Nevertheless, the total integrated intensity still provides a qualitative measure of the scattering power of the samples, and as thus, one can safely claim that the homopolymer exhibits cavitation to a greater extent than the copolymer.

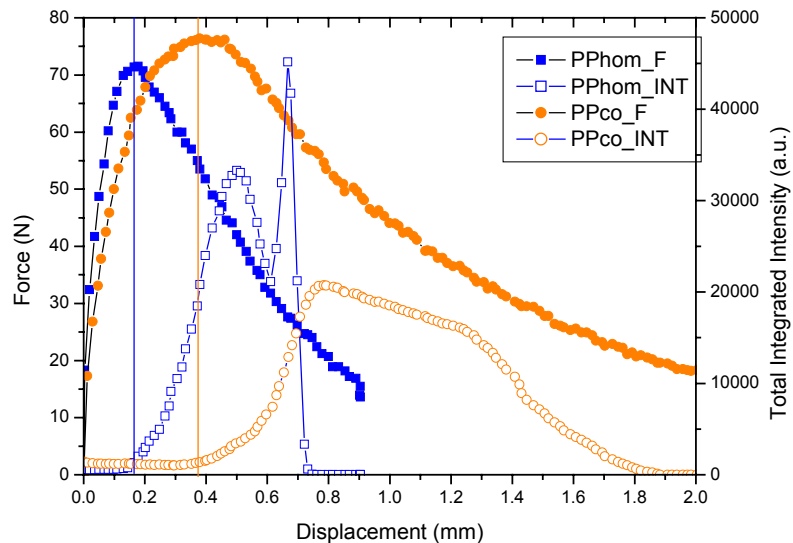


Figure 2: The Force-displacement plot coupled with the total integrated intensity-displacement plot for the two PPs. Note that the notation F (blue curves) correspond to force-displacement (left axis) while the notation INT corresponds to total integrated intensity-displacement curves (right axis)

The results of the present study have revealed that significant differences result from a slight modification of the molecular architecture in PP. In fact, only 5% mol addition of ethylene randomly distributed in the PP chain appears to suppress the formation of cavitation to a great extent without much loss of stiffness.

References

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