

### *The influence of crystallization temperature on the deformation of polypropylene spherulites*

It has been suggested [1] that polypropylene spherulites have a crosshatched structure made up of two sets of lamellae. One set is radially oriented, as may be expected in most spherulitic polymer systems, and the other is approximately tangentially oriented with the polymer chains lying radially. The crosshatching which results can be used as the basis for explaining birefringence results [1] and deformation characteristics [2].

The incidence of growth of the tangentially oriented lamellae is considered to be dependent on the crystallization temperature. The higher the crystallization temperature, the less likelihood that a crosshatched structure will occur. The crosshatched structure may arise because of the similarity at lower temperatures between the *a* and *c* dimensions of the unit cell [3], and the physical fitting of the methyl groups at the interface of the two sets of lamellae. At the higher crystallization temperatures the epitaxial misfit is too large for this type of growth to occur. The characteristic deformation of small spherulites of polypropylene (average spherulite radius less than about 75 $\mu\text{m}$ ) with crystallization initiated at a temperature below about 140°C is, as far as is known, unique and is probably determined by this crosshatched structure [2]. It is not possible to study the deformation of spherulites grown entirely at a high temperature

(i.e. above 150°C) since the large spherulites that ensue have inherently weak boundaries invariably resulting in brittle, interspherulitic fracture.

Fig. 1 shows a deformed (in tension, strain rate  $3.28 \times 10^{-5} \text{ sec}^{-1}$ ) spherulitic structure. The polypropylene had been moulded in the following way. It was cooled from 250 to 157°C and maintained at this temperature for 13 h (melting point *ca.* 170°C). Some crystallization will occur during this period but no tangentially-oriented lamellae are expected. Before solidification was complete the material was cooled to room temperature. Final growth would therefore occur at a much lower temperature. The boundary between the initial growth caused by the final cooling is shown by the ring. On further straining (Fig. 2) it can be seen that the initial spherulite deforms in a manner analogous to, for instance, polyethylene [4]. The rest of the spherulite deforms in the manner of polypropylene crystallized by continuous cooling and this is typified by the lower spherulites in both Figs. 1 and 2.

This suggests a very different crystalline structure for the two cases. It may be that the initial high temperature grown spherulite has a coarse open structure with well-formed crystalline fibrils, whilst the later solidified spherulite has a much finer structure with many small, mechanically-interwoven fibrils. This alone may explain the difference in deformation. However, the similarity in the deformation of the initial spherulite with that of a polyethylene spheru-

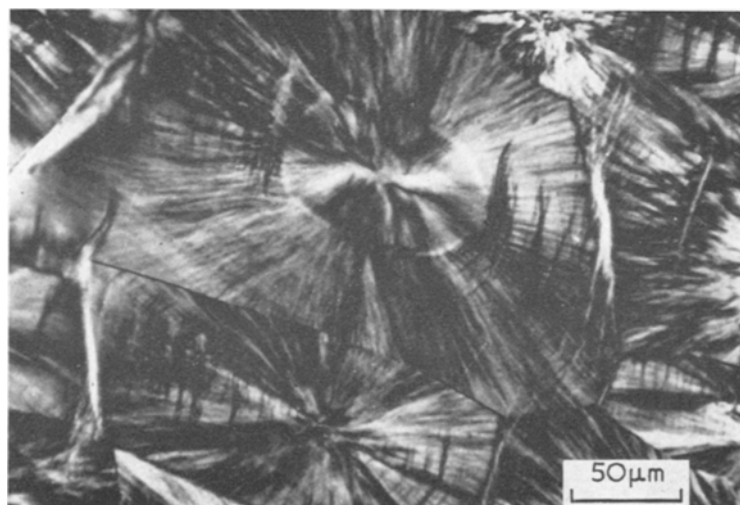


Figure 1 Transmission optical micrograph with crossed polars of deformed ringed spherulite.



Figure 2 Highly-deformed ringed spherulite.

lite suggests a similar structure, i.e. purely radially oriented lamellar growth, whilst the deformation of the final structure is characteristic of the structure peculiar to polypropylene at lower crystallization temperatures [2]. If this is the case it would tie in with the idea of a crosshatched structure which could only be formed at low crystallization temperatures, hence giving rise to the different deformation characteristics in the lower temperature grown parts of the structure.

### References

1 F. L. BINSBERGEN and B. G. M. DELANGE, *Polymer* **9** (1968) 23.

- 2 J. L. WAY and J. R. ATKINSON, *J. Mater. Sci.* **6** (1971) 102.  
 3 F. J. KHOURY, *Res. Nat. Bur. Stand.* **70A** (1966) 29.  
 4 I. L. HAY and A. KELLER, *Kolloid-Z.* **204** (1965) 43.

Received 8 June  
 and accepted 30 June 1972

J. L. WAY  
 J. R. ATKINSON  
 Department of Metallurgy  
 University of Leeds  
 Leeds, UK

### Direct observations of dislocations in splat-cooled $\text{Co}_5\text{Pr}$

Numerous attempts have been made to study crystal imperfections in metastable, rapidly quenched (splat-cooled) metal and alloy systems in an attempt to correlate their character and density with residual magnetic and/or superconducting properties [1-4]. In many prominent systems, particularly cobalt-rare earth alloys, samples prepared by rapid quenching of a molten charge directed upon a low-temperature substrate are brittle, and possess shape and thickness inhomogeneities which render them unsuitable or difficult to prepare for transmission electron microscopy. While some samples can be prepared for observations of dislocations by

thin-film transmission electron diffraction contrast [5], it has not been possible to directly observe dislocations in such thin films by moiré fringes arising from the systematic translation or rotation of overlapping foils of sufficiently large crystalline areas [6-8].

In the present investigation, the fortuitous occurrence of superimposed, parallel layers comprising thin samples of  $\text{Co}_5\text{Pr}$  splat-cooled films gave rise to moiré patterns in the transmission electron microscope which allowed dislocations in various electron-transparent regions to be directly observed.

The initial  $\text{Co}_5\text{Pr}$  alloy was prepared in a conventional arc-furnace under argon, using 99.99% Co wire and 99.9% Pr ingots as starting materials. The residual reactive impurities were