structure consisting of α-iron, Fe_3P and Fe_3C. At 420°C, the above phases are formed and the transformation of amorphous $Fe_{75}P_{15}C_{10}$ alloy to crystalline phases seems to be complete.

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Scanning electron microscopy observations of polyethylene spherulites

It is well known that often when polyethylene and certain other polymers are crystallized from the melt, they form banded spherulites which appear in the polarizing microscope as round or polygonal structures having a black Maltese cross and a series of concentric light and dark rings [1]. The most commonly accepted model for these to date, based almost solely on detailed analysis of the extinction patterns visible in the polarizing microscope [2-6], postulates that banded spherulites are aggregates of ribbonlike folded chain lamellae radiating outward from the centre of the spherulite while undergoing a series of full twists about the radially oriented *c*-axis. This model successfully explains the X-ray evidence [7] for a tangentially oriented *c*-axis as well as the periodic variation in refractive index along the radius necessary to produce the rings. A model of such lamellae is shown in Fig. 1, which shows a set of twisted copper strips whose edges were painted white for emphasis. All twists of the optically uniaxial lamellae must be in phase to produce the bands.

However, the optical evidence is somewhat indirect, depending heavily on the theory of optical extinction and on subtleties of the model being used. The use of the optical microscope is limited by low depth of field and insufficient resolution to see individual lamellae, and electron microscopy of replicas of spherulites [1] has not confirmed unambiguously the existence or nature of the twist. For these reasons, it was decided to use the scanning electron microscope (SEM), where these problems are to a large extent eliminated, to try to clarify the lamellar structure.

Samples 1 to 3 mm thick of melt-crystallized linear polyethylene were prepared by melting pellets of whole Marlex 6015 (M_n = 7500, M_w = 153000) on a glass slide on a hot plate and quenching the slide on an aluminium plate set on top of some crushed ice. The samples showed typical banded spherulites in the polarizing microscope. To enhance the surface relief

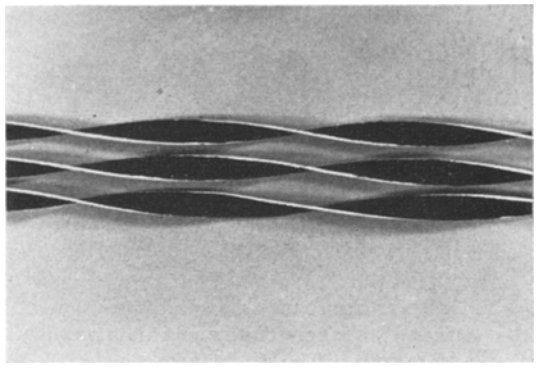

Figure 1 Schematic packing arrangement of lamellae with full helical twists.

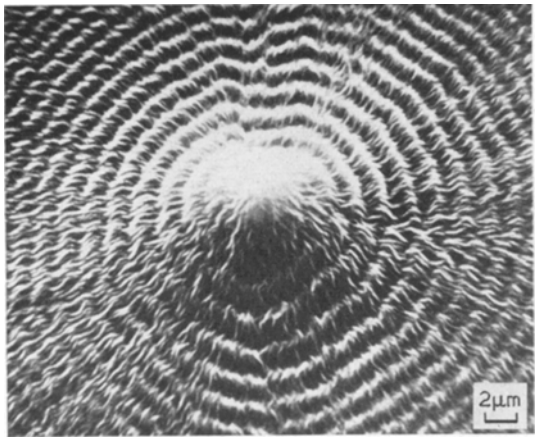

Figure 2 Scanning electron micrograph of a spherulite of melt-crystallized linear polyethylene which was ion etched for contrast. The electron beam is at 55° to the surface of the specimen.

necessary for SEM contrast, the air surfaces of the samples were etched for 4 h in a stream of argon ions with the intention of making the lamellae more visible by preferentially removing the disordered material between them. Possible effects of ion or thermal alteration of the structure were minimized or eliminated by slowly rotating the sample and inclining its surface only 15° to the beam. The accelerating potential was 6 kV and the beam current was kept at the very low value of 10 to 20 μA over 1 cm^2 of sample, again to minimize beam damage. For SEM examination the etched samples were vapour-coated with a layer of a 60% Au-40% Pd alloy less than 100Å thick.

Fig. 2 is a relatively low magnification scanning electron micrograph of a spherulite in a sample of polyethylene prepared as described above. The average diameter and band width of the spherulites as seen in the SEM matched those measured optically in the same sample.

Fig. 3 is a higher magnification scanning electron micrograph of a portion of two bands of a spherulite in a different sample. The centre of the spherulite is off of the left hand side of the picture. The individual lamellae are visible with the horizontal white lines corresponding to the

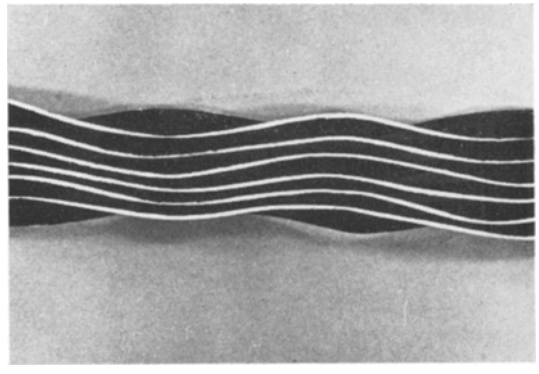

Figure 4 Schematic packing arrangement of lamellae with alternating left- and right-handed twists.

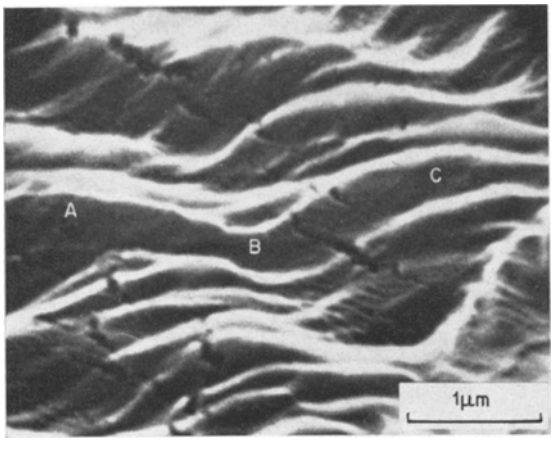

Figure 3 Scanning electron micrograph of a portion of a melt-crystallized linear polyethylene spherulite which was ion etched for contrast. The centre of the spherulite is off the left-hand edge of the picture. The beam is 45° from the surface of the specimen.

edges of the broad ribbons. The irregular black lines crossing the lamellae are cracks in the alloy coating due to differential thermal expansion under the beam between the coating and the underlying polymer. However, the most important feature of the micrograph is that the white edges appear to be continuous across the two bands. Reference to Fig. 1 indicates that this is inconsistent with the full twist model, where the edges would never be continuous across more than one band. If, on the other hand, the lamellae were to consist of alternate right- and left-handed twists, as shown by the painted copper models in Fig. 4, the present observations could be readily explained. The half-twist is particularly evident in the lamella marked with the letters in Fig. 3. The ribbon is nearly vertical at point A, dips over at B, and rises again to vertical at C. As can be seen in Fig. 4, the half-twist model also alleviates the space filling

problem presented by the fully twisted ribbons in Fig. 1, where the distance of closest approach between two lamellae twisting in phase is always determined by the ribbon width.

Such a half-twist has been observed by Geil [1] in some branched polyethylene spherulites, and by Keller and Machin [8] in crystallization of some stretched polyethylene melts. The results from the above ion etched linear polyethylene spherulites are consistent with other SEM observations made in this laboratory of unetched deeply ion-etched, and HNO_3-etched samples and on spherulites deformed to 50 to 100% elongations, which will be reported in full at a later date.

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Comments on "Constant stress creep and constant true strain-rate tensile tests of the superplastic alloy PbSn"

Recently, Baudelet and Suery [1] have reported results on true stress – true strain curves for lead–tin eutectic alloy (Pb–61.9 wt %Sn) in a superplastic condition which they believe conflict with earlier results which we have reported [2]. We reported strain hardening which was approximately a stress increase of 20% for an engineering strain of 100% (ϵ = 69%) in as-rolled lead–tin eutectic [2]. Baudelet and Suery [1] tested lead–tin eutectic which had been prepared by extrusion and then annealed "about 15 min at 130°C". They found strain independent flow, i.e., no strain hardening or strain softening. On the basis of this, they criticized our result, indicating that a possible source of error in our experiment was uncertainty in the true gauge length due to deformation in the grips [1].

In our earlier paper, we emphasized the importance of determining the actual length of the specimen which underwent deformation in creating the condition of constant applied strain-rate [2]. Our specimen was designed to provide minimal "feed-in" of material from the fillet to the gauge of the specimen. We have measured the volume of material fed into the gauge length by measuring the lateral and longitudinal profiles of the specimen near the gauge and found that this material effectively increases the gauge length by less than 2%. Baudelet and Suery [1] have calculated that a grip contribution of 20% to the initial gauge length is necessary to produce a false strain hardening effect in their material.

The explanation for this discrepancy in the results arises from differences in the conditions of the starting material used in our work [2] and that done by Baudelet and Suery [1]. Fig. 1 shows the true stress – true strain curves for lead–tin eutectic in various starting conditions.