

Therefore, a value of $D_{B_{600^{\circ}\text{C}}} \gg 10^{-6} \text{ cm}^2 \text{ sec}^{-1}$ must control the diffusion of lead into the iron.

The results indicate that relatively small amounts of certain second elements can change grain-boundary diffusivity to a large extent. Discontinuous precipitation is induced, if a more stable compound can be formed with the atoms which have penetrated through the surface. This process is likely to be of some practical importance, because lead-induced failures have already been reported in high strength steels [4]. It is not known as yet which other alloying elements in steels will produce similar effects.

References

1. E. HORNBOGEN, *Met. Trans.* **3** (1972) 2717.
2. *Idem*, *Acta Metallurgica* **10** (1962) 1187.
3. E. HORNBOGEN and M. ROTH, *Arch. Eisenhüttenw.* **36** (1965) 201.
4. N. N. BREYER and P. GORDON, in "The Microstructure and Design of Alloys" (The Institute of Metals and The Iron and Steel Institute, Cambridge, 1973) p. 493.

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Improved "single-crystal" texture in high-density polyethylene

Any study of the relation between structure and physical properties of crystalline polymers is greatly facilitated by using a material with as single and well-defined a texture as possible. In studies on high density bulk polyethylene it has been established that suitable combinations of deformation and heat-treatment can result in an oriented polycrystalline sample with a texture resembling that of a single crystal [1-9]. The degree of orientation obtained depends on the method used, and to date one of the most successful techniques for this has been the combination of compression-orienting followed by high pressure annealing [6, 8]. The purpose of this note is to report a modification of the above technique which has further improved the polyethylene texture.

The material used in this investigation was injection-moulded high density polyethylene (Rigidex 9) supplied in the form of granules by British Petrochemicals Limited. The material was oriented and annealed using similar apparatus to that described earlier [6], but thermistor-controlled heaters were added to the compression dies so that material could be compressed at elevated temperatures. The injection-moulded bar was first compression-oriented at $110 \pm 2^{\circ}\text{C}$ and then annealed for 15 min at $207 \pm 3^{\circ}\text{C}$ and 4 kbar pressure. The material was initially 12.5 mm thick and at the end of the process was 0.5 mm (thickness reduction of 25 times). Wide-angle X-ray photographs revealed

that prior to annealing, the compression oriented material had a good "single-crystal" texture of orthorhombic polyethylene, but there were also reflections indicating the presence of some monoclinic polyethylene. Subsequent pressure-annealing removed the monoclinic phase but also caused a slight reduction in the degree of crystallographic orientation in the specimen.

The final texture was examined by taking wide-angle pole figures using the diffractometer technique described earlier [10]. The combined

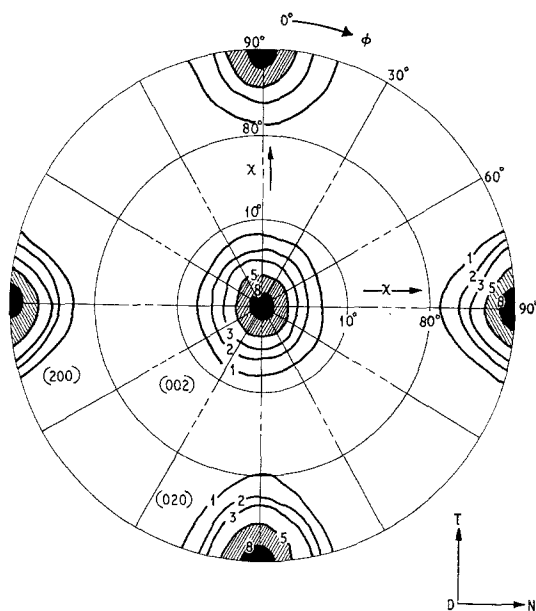


Figure 1. Combined wide-angle pole figures for the (200), (020) and (002) poles for hot compressed/pressure-annealed high density polyethylene.

TABLE I Orientation functions $f_{hkl,Z}$ and mean angular deviations $\langle \xi_{hkl,Z} \rangle$

Reference	$f_{002,D}$	$\xi_{002,D}$	$f_{200,N}$	$\xi_{200,N}$	$f_{020,T}$	$\xi_{020,T}$
Present work	0.989	4.9°	0.972	7.8°	0.972	7.8°
8	0.983	6.1°	0.967	8.6°	0.969	8.2°
9	0.986	5.5°	0.897	15.1°	0.898	15.1°
7	0.994	3.6°	0.844	18.8°	0.849	18.5°

pole figures for the (200), (020) and (002) reflections are shown in Fig. 1; the specimen reference axes are labelled N (the direction of compressional deformation), D (the direction of extensional deformation) and T (the transverse direction). As with other highly oriented samples [9] it is more convenient to display the intensity distributions only over the relevant portions of the reflection hemisphere. Thus, for clarity, Fig. 1 is drawn with a discontinuous scale in the polar angle χ (angle between a pole and D); the (002) poles are then entirely mapped from 0° to 10° χ and (200) and (020) from 80° to 90° χ . It can be seen at a glance that the polyethylene texture is very close to the ideal single crystal texture, in which the unit cell directions a , b and c would be exactly parallel to N , T and D respectively. The degree of departure from ideality is assessed by calculating from Fig. 1 the orientation functions $f_{hkl,Z}$ defined [9] as $3/2 \langle \cos^2 \xi_{hkl,Z} \rangle - \frac{1}{2}$, where $\xi_{hkl,Z}$ is the angle between any of the principal poles and its direction (Z) of preferred orientation. (For the ideal single crystal, all such functions are equal to unity.) The resulting orientation functions and their equivalent mean angular deviations from ideality, $\langle \xi_{hkl,Z} \rangle$, are tabulated below for the present texture, together with values obtained from other polyethylene "single-crystal" textures [7-9].

The texture obtained here is significantly better than that found by Young and Bowden [8]. This improvement appears to be due to the fact that the hot-compressed material described here has a much larger thickness reduction (25 times) than the previous cold-compressed material (6 times). This has particularly reduced the average misalignment of chain axes (from 6.1° to 4.9°). There is also a slight improvement in the orientation of a - and b -axes (from 8.4° to 7.8° deviation). Although the "single-crystal" texture obtained by Buckley and McCrum [7] by hot-drawing and annealing has a much greater c -axis alignment (only 3.6° deviation) the orientation of a - and b -axes is less well defined, the (200) and

(020) poles being particularly spread out in the NT plane giving high (18.6°) overall angular deviations.

In conclusion, the present technique of orienting high density polyethylene (hot-compression/pressure-annealing) has resulted in an unambiguous crystallographically "single-crystal" texture with the highest degree of perfection so far reported.

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References

- (a) I. L. HAY and A. KELLER, *J. Mater. Sci.* **1** (1966) 41; (b) *idem, ibid* **2** (1967) 538.
- (a) T. SETO and T. HARA, *Repts. Prog. Polymer Phys. Japan* **9** (1966) 207; (b) T. SETO and Y. TAJIMA, *Jap. J. Appl. Phys.* **5** (1966) 354.
- A. COWKING, J. G. RIDER, I. L. HAY, and A. KELLER, *J. Mater. Sci.* **3** (1968) 646.
- J. J. POINT, G. A. HOMES, D. GEZOVICH, and A. KELLER, *ibid* **4** (1969) 908.
- C. P. BUCKLEY, R. W. GRAY, and N. G. MCCRUM, *J. Polymer Sci.* **B8** (1970) 341.
- R. J. YOUNG, P. B. BOWDEN, J. M. RITCHIE, and J. G. RIDER, *J. Mater. Sci.* **8** (1973) 23.
- C. P. BUCKLEY and N. G. MCCRUM, *ibid* **8** (1973) 1123.
- R. J. YOUNG and P. B. BOWDEN, *ibid* **8** (1973) 1177.
- R. W. GRAY, J. M. HUTCHISON, and J. F. MATTHEWS, *ibid*, **9** (1974) 503.
- E. P. CHANG, R. W. GRAY, and N. G. MCCRUM, *ibid* **8** (1973) 397.

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