Letters

The Use of a Scanning Electron Microscope to Examine Whisker Growth on an Iron-Aluminium Alloy

In the course of experiments on secondary recrystallisation in iron-aluminium alloys, performed with a view to growing coarse-grained sheet with a magnetically favourable preferred orientation, it became clear that the recrystallisation process is acutely sensitive to minute traces of surface contamination. A dynamic vacuum of 10^{-4} torr still permitted contamination to a degree which prevented the growth of large grains; the growth of such grains in silicon iron is known to be controlled by the surface energy [1], which in turn is affected by contamination.

The present work was done with an alloy containing 4.6 wt % aluminium. The contamination problem was reduced by surrounding the specimen by a box made of the same alloy; this form of "self-gettering" increased the number of successful recrystallisation runs. Some further improvement was effected by improving the vacuum to 10^{-6} torr. This better vacuum was created in a small chamber, which necessitated the use of a small furnace with a steep temperature gradient. It was discovered that under these experimental conditions, copious whiskers grew on the surface of the alloy strip. Whiskers formed on those parts of the surface which were between 975 and 1250° C (the maximum temperature).

The whiskers were photographed in a "Stereoscan" scanning electron microscope (Cambridge Instrument Co). In this novel instrument, a focused electron beam explores a tilted surface, the secondary electrons emitted are collected and their measured instantaneous density is used to modulate the beam of a cathode ray tube which forms the image. The microscope combines the advantages of high resolution and great depth of focus [2], so that, for instance, long whiskers can be focused sharply along their entire length.

Figs. 1 and 2 show whiskers at low magnification. Secondary recrystallisation has not begun. The shapes of the whiskers differ widely, and most are distinctly faceted. Fig. 3 shows in more detail one part of fig. 1, where two

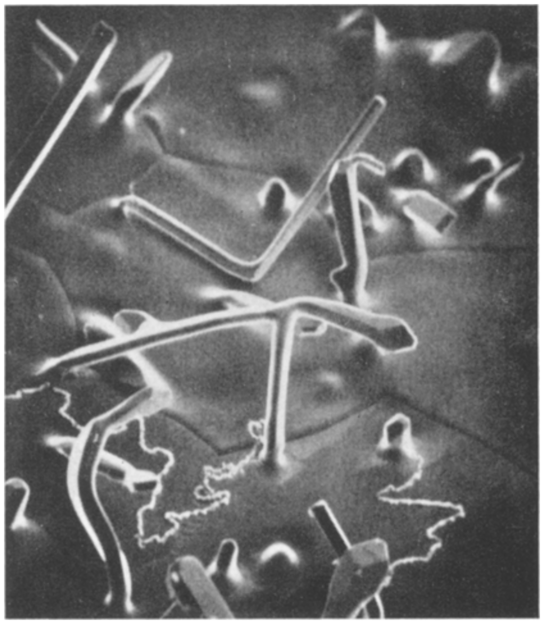

Figure 1 Whisker growth at about 1150° C. The nature of the twisted threads growing from two of the whisker roots is unknown (×250).

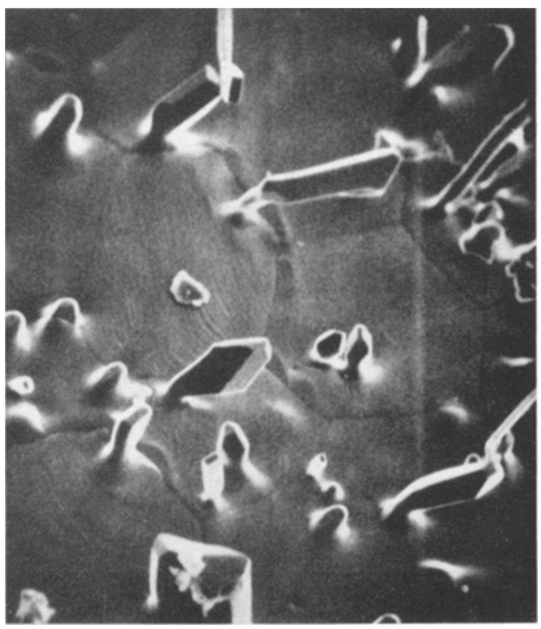

Figure 2 Whisker growth at about 1150° C (×250).

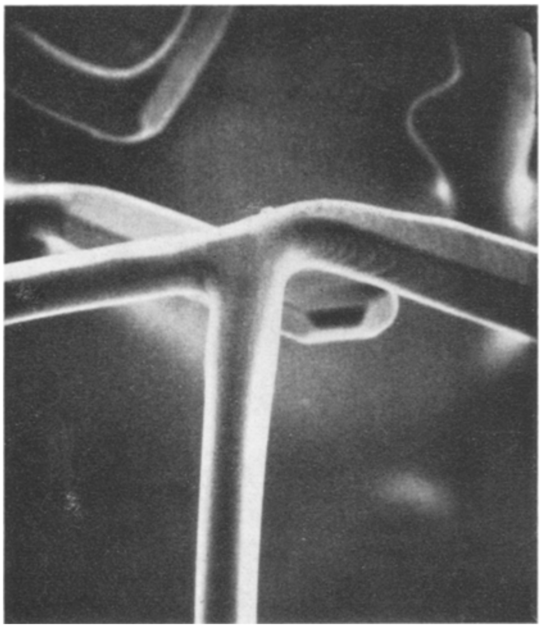

Figure 3 Part of field of fig. 1 (×800).

whiskers have joined smoothly and continued to grow in a new direction.

We believe that the whiskers formed by condensation from the vapour phase. Their composition is unknown; they may be pure iron or a concentrated alloy. The observation that metal whiskers formed on the alumina block which separated the alloy strip from its "gettering box" could only be interpreted in terms of deposition from the vapour. The supersaturation $(P_M - P_T)/P_T$, where P_T, P_M are equilibrium vapour pressures of pure iron at the growth temperature, T, and at the maximum temperature, 1250° C, varied from 0 to 2500; over this range, whisker diameters increase from ~1 to ~50 μm, and lengths decrease from ~1000 to ~5 μm. The micrographs refer to a supersaturation of 10 to 200, whisker diameter ~10 μm and length ~150 μm. These dimensions are similar to those of whiskers grown on pure iron at a similar temperature and vacuum [3].

There is no direct evidence as to the role of impurities in the growth of these whiskers. The fact that they mostly develop near (but not *at*) grain boundaries, where impurities are apt to segregate, suggests that impurities may play a part, and certainly the extreme sensitivity of the metal surface to contamination (as shown by the difficulty of securing reproducible surface-controlled secondary recrystallisation) supports the view that the same contamination may help whisker growth. A recent study of whisker growth on pure iron during high temperature oxidation [4] established clearly that surface impurities determined where whiskers grow, which may be a pointer to interpreting the present results. In that study, it was found that whisker growth varied from one grain to another, which was interpreted in terms of orientation-dependent impurity adsorption. Figs. 1 and 2 show a similar variation of whisker density in the present experiment.

Acknowledgements

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References

1. J. C. WALTER and E. G. DUNN, *Acta Met.* **7** (1959) 424.
2. P. R. THORNTON, Science Journal **1** (1965) No. 9, 66.
3. C. R. MORELOCK, referred to in "The Art and Science of Growing Crystals", edited by J. J. Gilman (Wiley, 1963) p. 37.
4. R. F. W. PEASE and R. A. PLOC, *Trans. Amer. Inst. Min. Metall. Engrs.* **233** (1965) 1949.

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Formation of Zinc Sulphide Polytypes by Spiral Growth around Dislocation Clusters

Crystals of the ZnS type have two basic modifications, namely the cubic sphalerite structure and the hexagonal wurtzite structure (3C and 2H, respectively, in Ramsdell's notation [1]). However, ZnS crystals rarely appear in these extreme forms, but usually display a large variety of polytypes, which are intermediate between the basic cubic and the hexagonal structures. Although a large amount of work on polytypism