heating effect were ignored. An error of 1 part in  $10^{-3}$  was recorded in the calculated value of the lattice spacing due to the heating effect of a  $3 \times 10^{-3}$  W electron beam on Swedish iron. This simple method eliminates a systematic error that always produces a larger value for the lattice parameter than the true value.

Values of  $\Delta\theta$  calculated from Equation 2 assuming the thermal expansion coefficient of iron to be  $12 \times 10^{-6}$  give temperature rises of 28, 52 and  $87^{\circ}$ C at the points on the graph in order of increasing beam wattage. These are the average temperature rises in the volume in which diffraction of the X-rays excited by the electron beam has occurred. Using these values in Equation 1 we calculate a value for r of  $7 \times 10^{-6}$ cm (i.e. 700 A). From this result it appears that most of the diffraction occurs within a small distance of the incident electron beam. Since there is a temperature gradient in this region there must be a variation in the lattice spacing. This effect must inevitably lead to a broadening of the diffracted X-ray peak and could account for the fact that the familiar white edge (absorption conic) is not seen on (220) Kossel curves from iron grains in back-reflection.

There has been much discussion in recent years about the volume of crystal required for the generation of Kossel patterns. The results of these experiments suggest that it should be possible to obtain Kossel patterns from volumes of less than  $1 \mu m$  in diameter but there will be some line broadening due to both particle size and temperature gradients. Difficulty arises becauses X-rays not satisfying the Bragg conditions for diffraction at planes in the crystal being bombarded by the electron beam pass on to adjacent crystals and may be diffracted by the lattice planes of these. The resulting pattern is

# **Directional coupled growth of a modified** *Inconel 713C alloy*

The morphology of MC carbide obtained by directional solidification of eutectic systems: X-MC where X is Fe, Ni, Co or simple alloys of these metals and M is Ta or Nb was previously investigated [1-4]. Walter and Cline [3] in their work on Co-TaC, Ni-TaC, Fe-TaC, NiCr-TaC, CoNiCr-TaC and FeCrA1-TaC eutectics used hypereutectic compositions in order to produce both primary faceted crystals and fibres of TaC within the same ingot. The study of these

difficult to interpret since it contains a large number of Kossel curves from crystals other than the one directly under examination.

Finally, the temperature rises calculated above are of considerable significance to those wishing to use electron-probe microanalysis for the study of trace elements in plastics or other materials of low melting point. It has often been assumed that coatings are required merely for the purpose of conducting away the incident electrons but in these investigations coatings must also supply good heat conduction.

### **Acknowledgements**

The author thanks Dr D. G. Fisher, Dr N. Swindells and Dr M. J. Bevis for useful discussions on the subject of this letter.

### **References**

- 1. B. H. HEISE, *J. Appl. Phys.* 33 (1962) 938.
- 2. R. E. HANNEMAN, R. E. OGILVIE and A. MODRZE-JEWSK1, *ibid* 33 (1962) 1429.
- 3. w. B. PEARSON, "A Handbook of Lattice Spacings and Structures of Metals and Alloys" (Pergamon Press, 1958).
- 4. K. J. H. MACKAY, Proceedings of the 4th International Congress on X-ray Optics (1965) p. 544.
- 5. R. CASTAINO, Thesis, University of Paris (1951).
- 6. D. G. FISHER and N. HARRIS, Proceedings of the 5th International Congress on X-ray Optics, (1968) p. 396.
- *7. Idem, J. AppL Cryst.* 3 (1970) 305.

*Received and accepted 11 March 1974* 

> N. HARRIS *Department of Metallurgy, and Materials Science, University of Liverpool, Liverpool, UK*

faceted TaC crystals enabled them to analyse the factors that determine fibre orientation and morphology, both of which were found to be significantly affected by addition of elements, such as Cr.

The main purpose of the present investigation was to extend these morphological studies to more complex systems and, more specifically, to. explore in a preliminary way the dependence of growth morphology of the carbide on growth conditions in a eutectic system consisting of a polynary matrix and a polynary carbide phase.

In a recent investigation [5] of the solidifica-



*Figure 1* Scanning electron micrographs of directionally solidified modified Inconel 713C alloy: (a) and (b) are longitudinal sections of a specimen grown at 1.7 cm h<sup>-1</sup> ( $\times$  900 and  $\times$  5000, respectively); (c) is a transverse section of a specimen grown at 2.2 cm h<sup>-1</sup> ( $\times$  900) and (d) is a transverse section of a specimen grown at 2.6 cm  $h^{-1}$  ( $\times$  1000).

tion behaviour of Inconel 713C alloy under various combinations of thermal gradient at the solid-liquid interface and growth rate, it was observed that in some cases of fast solidification a well-defined eutectic formed interdendritically.

This eutectic consisted of a nickel-rich  $\gamma$ -phase and a MC-carbide. The composition of this eutectic was determined by two independent procedures: (1) Inconel 713C was solidified very fast in a copper chill-mould and a very fine



*Figure 2 (a)* Photomicrograph of a transverse section of a specimen grown at 2.6 cm  $h^{-1}$  ( $\times$  1000); (b) and (c) are scanning electron micrographs of a transverse section of a specimen grown at 2.4 cm  $h^{-1}$  ( $\times$  600 and  $\times$  1500, respectively).

eutectic microconstituent was obtained interdendritically. Electron microprobe analysis was conducted at random locations within these eutectic regions, using a defocused electron beam, and average concentrations were measured for all elements for which it was possible; (2) the molecular composition of the carbide was established by electron microprobe analysis conducted on isolated coarse carbide particles outside the eutectic regions. This composition was:  $(Nb_{0.63}Ti_{0.31}Mo_{0.06})C$ , with traces of Zr, Ni and Cr. The weight percentage composition of the  $\gamma$ -phase was also determined by the same technique near the primary dendrite arm

boundaries. It was assumed that the composition of the carbide in the eutectic regions was approximately similar to that of isolated carbide particles. The volume fraction of carbide was measured within the eutectic regions, using quantitative metallographic techniques [6, 7] and found to be equal to 0.09. Thus, the composition of the eutectic liquid was deduced. Results of these two calculations were reasonably close and were averaged. The adopted wt  $\frac{\%}{\%}$ composition of the eutectic liquid was:  $Cr =$ 12.07, A1 = 5.57, Ti = 1.84, Mo = 4.62,  $(Nb + Ta) = 6.64$ ,  $B = 0.01$ ,  $Zr = 0.1$ ,  $C =$ 1.055,  $Co = 0.53$ ,  $Ni = balance$ . An alloy of this

composition was prepared by modifying an as-received Inconel 713C alloy with a masteralloy containing 68.5 wt  $\%$  (Nb + Ta), 17.2 wt  $\%$ Ti and 14.3 wt% C, elements, whose concentration was higher in the eutectic liquid. The alloy thus prepared was cast in copper chill moulds in the form of rods (0.190 in. diameter  $\times$  8 in. long). These rods were zone-melted and resolidified in an MRC EBZ-93 electron-beam zone-melting furnace under a gradient  $G = 120^{\circ}$ C cm<sup>-1</sup> and at growth rates varying from about 0.25 to 2.6  $cm h^{-1}$ . It was anticipated that melt composition and growth conditions would affect substantially the selective absorption of impurities and molecular attachment rates on different crystallographic planes, with tendency of the faster growing surfaces to disappear leaving the crystal finally bounded by slow growth planes.

The specimens were sectioned transversely and longitudinally, polished, deeply etched with Marble's reagent and examined metallographically and by scanning electron microscopy.

The carbide phase appeared to consist of a mixture of trifoils [1-4] and individual fibres, with the relative number of trifoils decreasing with increasing growth rate and finally vanishing for a growth rate  $R \simeq 2$  cm h<sup>-1</sup>. Genetic explanation of carbide trifoils was reported previously [3, 4]. Examination of longitudinal sections indicated that the faces of the blades in the trifoils bore growth marks and serrations (Fig. la and b) analogous to those observed by Walter and Cline [3]. According to these authors the growth marks would indicate that the apex of the trifoil grows ahead of the side phases with their leading edges at an angle to the transverse plane of the ingot. They would also indicate that the liquid-matrix-MC carbide junction is not flat but concave upward.

For R higher than about 2.0 cm  $h^{-1}$  the carbide is in the form of individual fibres of square cross-sections (Fig. 1c) with axis  $\langle 100 \rangle$  corresponding to the rapid growth direction determined by Walter and Cline [3], in primary TaC particles by X-ray diffraction. These faceted fibres have four {111} facet planes and their external surface is bounded by four {110} planes. The fibre morphology differs from grain to grain. When the orientation of fibres and matrix are the same or close, fibres are smooth or with minor serrations, otherwise they are corrugated (Fig. 2b and c). These corrugations have regular spacings. In some cases the rods are rectangular and exhibit a tendency for preferential growth sideways along  $\langle 100 \rangle$  directions (Figs. ld and 2a). Fast solidification eliminates grains with axes oriented more than  $10^{\circ}$  off the  $\langle 100 \rangle$  direction, and fibres are not corrugated. The fixed fibre orientation indicates that the ends of fibres lead the solid-liquid interface, but the presence of corrugated fibres in grains of orientation different from that of the fibre would imply an interaction of the two phases at the MC carbide-matrix liquid junction [3 ]. These surface perturbations in the  $\langle 100 \rangle$  direction, normal to the edges of the rods, can be attributed to either another new set of  $\{111\}$  facets along the  $\{110\}$ sides of the fibres, as explained by Walter and Cline [3 ] or, more probably, to surface instability with perturbations parallel to the fibre axis (Z perturbations) or sometimes perturbations in circular shape ( $\phi$  perturbations) or even combinations of both (Fig. 2b and c). These observations are analogous to those of Coriell *et al.* [8] for surface instability of ice-water.

Within the growth rate range investigated here, the fibre spacing was found to depend on growth rate according to the relationship:  $\lambda^2 R = 11.75$ , where  $\lambda$  is the spacing in um and R is the growth rate in cm  $h^{-1}$ .

## **Acknowledgement**

This work was supported by the University of Connecticut Research Foundation.

#### **References**

- **1. H. BIBRING, G.** SEIBEL and **M.** RABINOVITCH, *Compt. Rend. Acad. Sci. Paris* 271 (1970) 1521.
- 2. H. BIBRING, Proceedings of the Conference on *In Situ* Composites, Lakeville, Connecticut 2 (1973) p. 1.
- 3. J. L. WALTER and H. E. CLINE, *ibid*, 1 (1973) p. 61.
- 4. v. M. PATARINI and T. Z. KATTAMIS, *ibid,* 2 (1973) p. 187.
- 5. A. K. BHAMBRI and T. Z. KATTAMIS, *Met. Trans.*  to be published.
- 6. E. E. UNDERWOOD, "Quantitative Stereology" (Adison-Wesley, Reading, Massachusetts, 1970).
- 7. J. E. HILLIARD and J. w. COHN, *Trans. Met. Soc. AIME* 221 (1961) 344.
- 8. S. R. CORIELL, S. C. HARDY and R. F. SEKERKA, *d. Crystal Growth* 11 (1971) 53.

*Received 11 March* 

*and accepted 18 March 1974* A. K. BHAMBRI T. Z. KATTAMIS

*Department of Metallurgy, Institute of Materials Science, University of Connecticut, Storm, Connecticut, USA*