Letters

Experimental observations of dendritic duplex crystals grown in complex Ni base alloys

In the course of investigations carried out at Ispra (Materials Division) on the production of Ni-base fibre-composites by unidirectional solidification [1, 2], some dendritic morphologies were obtained (Fig. 1) under various experimental conditions in a complex Ni-Ta-Cr-Mn system [3], based on the Ni-Ni_3Ta eutectic. Generally, the addition to binary eutectics of one or more additional elements emphasizes the growth instability at the solidification front with the destruction of the regular structure [4-6]. Consequently, the regular eutectic structure is first replaced by a cellular structure and then, when the amount of the other alloying elements exceeds the impurity level, primary crystals of one eutectic phase with a dendritic morphology appear. In order to study their topography by electron scanning microscopy and their structure by X-rays, the dendrites were isolated from the matrix by a natural decanting technique, i.e., by a technique in which the solidification front is separated from the liquid front by the force of gravity [7]. Dendritic structures have been obtained by liquid decanting by different researchers [8-10] during various conditions of steady-state growth in order to observe the topography of the dendrites and to study the growth mechanisms.

Fig. 2 shows a scanning electron microscope image of the network of dendritic crystals obtained by this decanting technique. The crystals are aligned in the same direction along the axis of the solidification ingot (ϕ, 12 mm) and the periodicity of the network is regular over the whole section of the ingot. The phenomenon is easily reproduced: more than twenty dendrite arrays were prepared in order to obtain the necessary experimental material. The crystals present primary and secondary arms and in some cases, also ternary branches, as can be seen in Fig. 3, which shows some dendritic crystals at higher magnification than

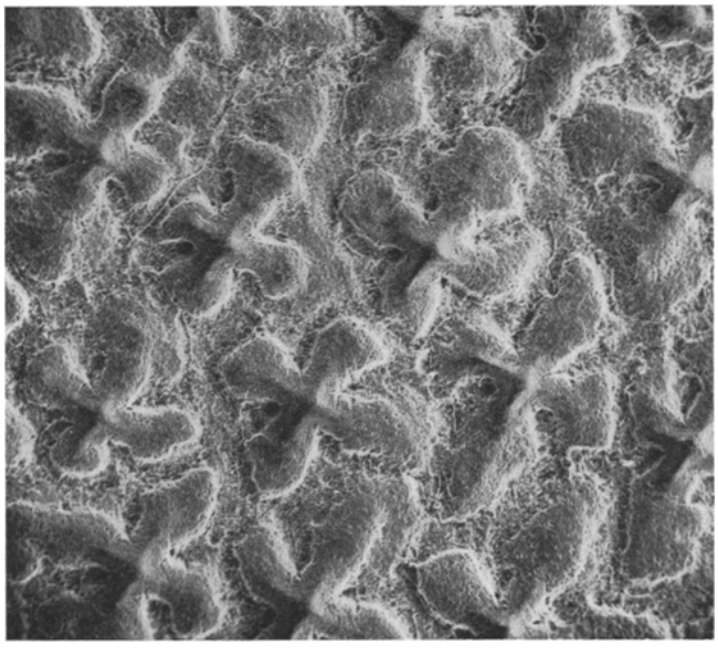

Figure 1 A scanning electron microscope image showing the dendrite structure on a complex Ni-Ta-Cr-Mn system (approximate composition Ni 57, Ta 21, Cr 15 and Mn 7 wt %) unidirectionally solidified in the following condition: thermal gradient 80°C cm^{-1}, solidification rate 9.5 cm h^{-1}. The image is of a transversal section of an ingot (ϕ, 12 mm) strongly attacked by electrolytic etching (× 100).

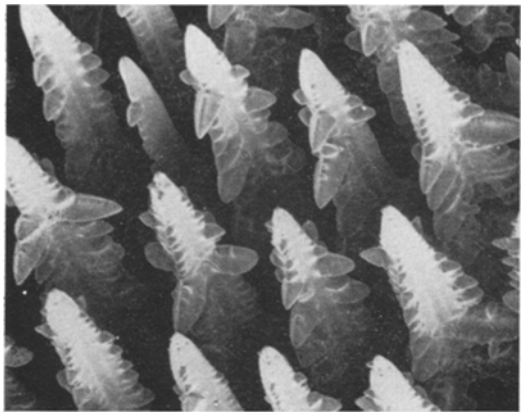

Figure 2 A scanning electron microscope image showing the dendrite structure network of the type shown in Fig.1, isolated from the matrix by natural decanting technique. The electron beam is not parallel to the growth direction as in the case of Fig. 1 (× 70).

in Fig. 2. The analysis at still higher magnification (Fig. 4) shows the surface of a single dendrite crystal. There is a certain roughness produced by an array of small precipitates of Widmanstätten type, as was confirmed *a posteriori* by an X-ray examination. The morphology of the dendrites clearly shows a cubic symmetry, also confirmed by X-rays.

The following technique has been used to isolate single dendrites without deforming them:

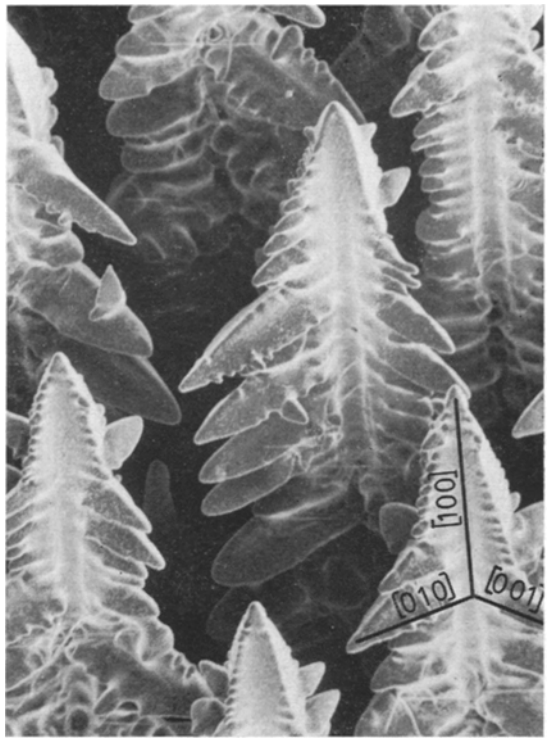

Figure 3 A view of some dendrite crystals with ternary arms nucleated on the secondary arms (× 200).

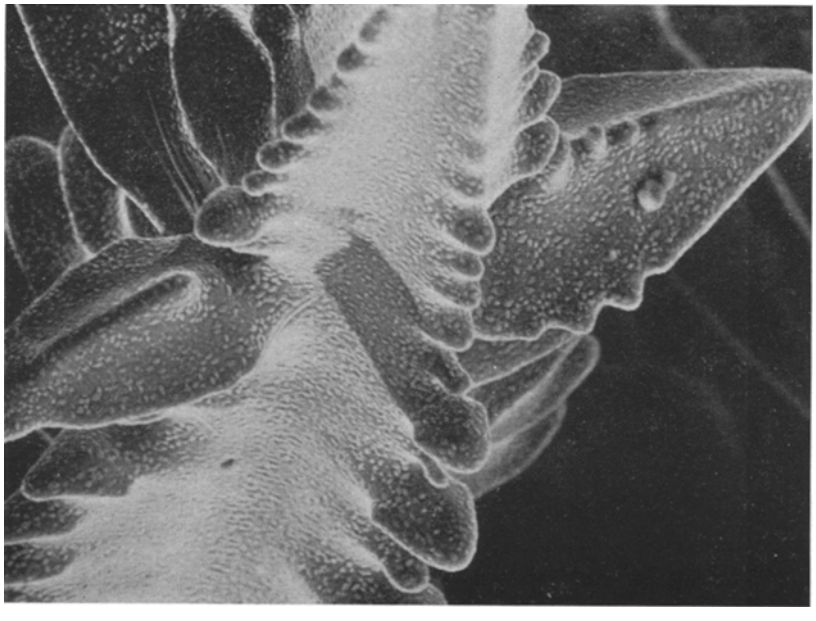

Figure 4 Dendrite crystal with Widmanstätten precipitates visible on the surface, showing the two-phase structure of the dendrites (× 750).

the whole specimen was embedded in a normal plexiglas metallographic resin, the bulk base was ground off, and the resin was dissolved in order to liberate the dendrites. The study was then made using single-crystal X-ray techniques, i.e., Laue, oscillating crystal and Weissenberg patterns. Each dendrite was found to be a single crystal with fcc crystal structure (a_0 = 3.62 Å) which corresponds to the nickel lattice with unit cell parameter increased by elements in solid solution. The growth direction is parallel to a ⟨100⟩ row of the cubic lattice, the secondary branches are also parallel to these ⟨100⟩ directions. The presence of a second phase is clearly indicated by Weissenberg films (Fig. 5) which can be interpreted as the superposition of four single-crystal patterns. This second phase is tetragonal with lattice parameters a = 3.62 Å, c = 7.46 Å, c/a = 2.06, which corresponds to a Ni_3Ta-β type of structure [11]. Relative intensities of the spots: strong when h, k, $l/2$ have the same parity and weak when h, k, $l/2$ have different parities, also confirm this type of structure. The relative orientation of the precipitates on the matrix may be deduced from the Weissenberg pattern; there are three families of precipitates with the three possible cubic orientations:

$$(001)_{\text{prec.}} \parallel \{100\}_{\text{matrix}}$$
$$[010]_{\text{prec.}} \parallel \langle 100\rangle_{\text{matrix}} .$$

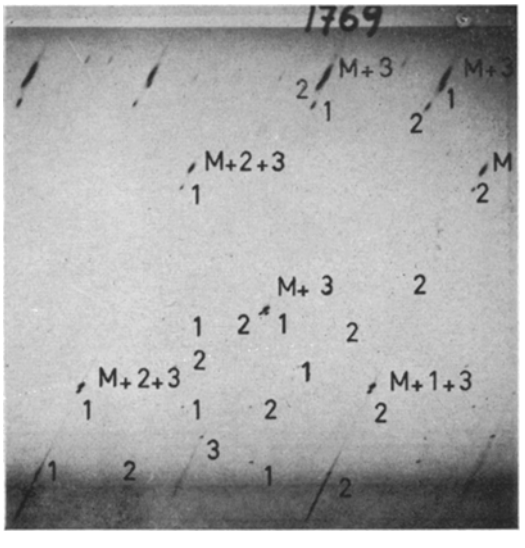

Figure 5 Detail of Weissenberg film which shows diffraction spots of: M, matrix; 1, first family of precipitates; 2, second family of precipitates; 3, third family of precipitates.

This mutual orientation is perfect so that each family of precipitates diffracts X-rays as a single crystal. The correspondence of lattices is perfect (within experimental error) in the $(001)_{\text{prec.}}$ plane and the mismatch is only 3% along the c-axis in the $(010)_{\text{prec.}}$ plane.

One may suppose that the dendrites in the complex system investigated (Ni-Ta-Cr-Mn (Ni corner)) grow as monocrystalline Ni-rich solution with Ta, Cr and Mn, and are transformed into duplex crystals by Widmanstätten precipitation of a second phase (Ni_3Ta β) owing to the large variation with temperature of solubility of Ta in Ni [12]. The dendritic growth mechanism is under investigation by the authors in a Ni-Ta-Cr-Mn system of different compositions and by different solidification parameters (thermal gradient, solidification rate etc.).

Acknowledgement

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