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The effect of shape variations during growth on the morphology of *in-situ* eutectic composites

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An experimental investigation has been conducted of the influence on *in-situ* eutectic composite microstructures of both gradual and abrupt changes in cross-sectional area, both with and without simultaneous changes in growth direction, and of the effects of changes in growth direction at constant cross-section. Alloys of the binary (Al–Ni and ternary Al–Cu–Ni) eutectic were chosen to eliminate the effects of microsegregation and these were grown under a variety of conditions of growth rate and temperature gradient, consistent with those used for off-eutectic *in-situ* composites, and in apparatus generally used for that work and described previously. As well as the expected variation in lamellae or inter-rod spacing with changes of cross-section, abrupt decreases in cross-sectional growth area coupled with changes in growth direction produced a multigrain structure of various orientations. Potentially weak zones denuded of lamellae or rod growth were observed when the growth direction deviated beyond a limiting small angle at constant cross-section.

1. Introduction

The improved high temperature mechancial strength that can be obtained from aligned composite materials is well documented. Of the several alternative methods of manufacture, the *in-situ* composite, grown from a homogeneous melt offers inherent advantages over its competitors since problems of interfacial stress and chemical reaction are eliminated. With the publications of Flemings and co-workers [1–3] and others [4, 5], it is now well established that with suitable choice of the ratio, G_L/R, where G_L is the temperature gradient in the liquid ahead of the interface and R the growth rate, compositions far from the equilibrium eutectics can be grown as plane front *in-situ* composites. Moreover, Bibring [6] has demonstrated how further judicious doping with minor constituents can allow tailoring of the mechanical properties of such *in-situ* composites to meet particular demands.

However, whilst these fundamental metallurgical problems of *in-situ* composites are reasonably well understood, practical application requires that their morphological stability remain whilst complicated shapes, such as airfoils, are formed. To date, literature on this topic, with a few notable exceptions [7–9], has been scarce. Hunt *et al.* [7] have examined the behaviour of several binary eutectics when confronted with obstructions in the growth path, and Chadwick *et al.* [8] have employed experiments forcing the eutectic interface to traverse crucibles of various radii of curvature.

The present work is an exploratory study of the behaviour of binary and ternary eutectics when confronted with shape changes during growth. The shape changes considered are both gradual and abrupt changes in cross-section and angular deviations with constant cross-section. The choice of the eutectic compositions eliminates solute enrichment effects which are likely under these circumstances and which are expected to be significant for off-eutectic alloys.

2. Experimental procedure

Alloys of the binary (Al–6.1 wt % Ni) and ternary (Al–0.73 wt % Ni–32.94 wt % Cu) eutectic composition were made up and after chemical analysis were directionally solidifed using the apparatus and technique described by Rinaldi *et al.* [3]. Specimens were contained within a 4.8 mm i.d. by 170 mm long graphite (99.95%) crucible. Growth was accomplished by withdrawing at the required rate from a platinum resistance furnace into a water-cooled copper chill and was terminated by rapid quenching. Variations in cross-section up to 90% and deviations in the growth direction of between 6° and 40°, characterized by the angle θ, Fig. 1, were obtained with the aid of graphite inserts cemented in place prior to filling with alloy as described by Rinaldi [3]. Temperature gradients within the specimen were controlled by the furnace temperature and were measured with a chromel/alumel thermocouple of 0.1 mm diameter wire insulated in twin bore 0.92 mm diameter alumina and located just below the bottom insert. A list of specimens and associated growth conditions is given in Table I. After each run the outer crucible was broken off and the entire specimen/insert assembly was mounted and polished together.

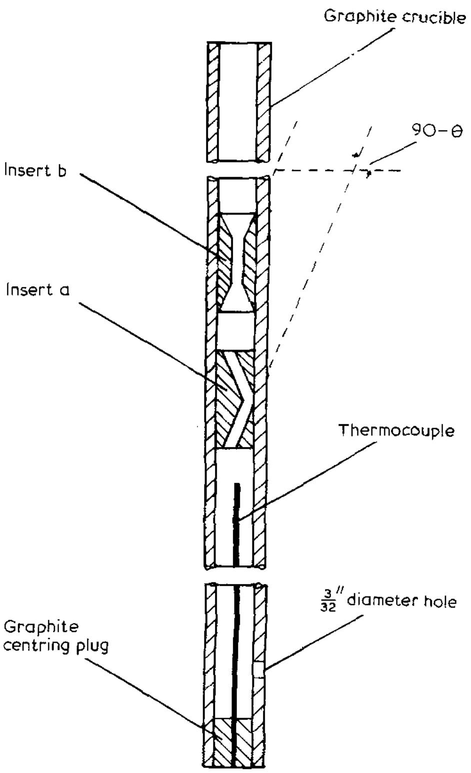

Figure 1 Cross-sectional view of graphite crucible and insert system.

3. Experimental results and discussion

In regions removed from the inserts, both the binary and the ternary eutectic alloys showed no change in morphologies with growth conditions except for variations in inter-rod, (in the case of the binary eutectic) or interlamellar spacings with

TABLE I Operational parameters of runs 0002–0021

Sample number	Alloy	Angle θ	Rate ($cm\ h^{-1}$)	Gradient ($^\circ C\ cm^{-1}$)	G/R ($10^{-5}\ ^\circ C\ sec\ cm^{-2}$)
0002	Al–6.1% Ni	14°	–	–	–
0003	Al–6.1% Ni	14°	0.75	235	11.3
0004	Al–6.1% Ni	14°	1.83	278	5.47
0005	Al–6.1% Ni	14°	2.73	310	4.08
0006	Al–6.1% Ni	18°	1.85	270	5.46
0007	Al–6.1% Ni	18°	2.02	–	–
0008	Al–6.1% Ni	18°	1.15	255	8.0
0009	Al–.73% Ni–32.94% Cu	18°	1.20	342	10.3
0010	Al–.73% Ni–32.94% Cu	16°	1.20	273	8.2
0011	Al–.73% Ni–32.94% Cu	20°	1.20	276	8.27
0012	Al–.73% Ni–32.94% Cu	20°	2.22	276	4.5
0013	Al–.73% Ni–32.94% Cu	14°	1.2	400	12.0
0014	Al–.73% Ni–32.94% Cu	10°	2.0	350	6.3
0015	Al–.73% Ni–32.94% Cu	6°	1.2	375	11.0
0016	Al–.73% Ni–32.94% Cu	14°	2.2	375	6.0
0017	Al–.73% Ni–32.94% Cu	6°	1.8	330	6.6
0018	Al–.73% Ni–32.94% Cu	8°	1.2	342	10.3
0019	Al–.73% Ni–32.94% Cu	12°	1.2	342	10.0
0020	Al–.73% Ni–32.94% Cu	12°	2.0	334	6.0
0021	Al–.73% Ni–32.94% Cu	8°	2.2	230	3.76

growth rate. Thus, assuming solute effects to be negligible for eutectic compositions, the following observations within the inserts can be attributed to the shape and angular deviations they imposed upon the composites.

For simplicity, the results may be sub-divided into three groups: (1) gradual changes in specimen cross-sectional area; (2) abrupt contraction of cross-sectional area with simultaneous change in growth direction; (3) abrupt changes in growth direction with constant cross-section.

3.1. Gradual changes in specimen cross-section

These observations relate to insert (b) of Fig. 1 in which it can be seen exist regions of both converging and diverging cross-section, together with a short length of constant reduced cross-section. From experiments in which the specimens were quenched with the liquid/solid interface within the lower, converging, and middle, constant, cross-section regions of insert (b), it can be stated that within these regions the interface remained flat and perpendicular to the crucible axis.

In the mouth of the insert in which the cross-section is continuously converging, the lamellae and rods, consistent with previous work [9], continued to grow parallel to the crucible axis regardless of the value of θ and this persisted through the region of constant reduced cross-section also (Fig. 2). Contrary to the results of Farag [9], however, only a few specimens showed a slight tendency for the rods or lamellae to coarsen at the upper specimen/insert interface (Fig. 3). For the binary eutectic alloy, a transition of rod to place morphology was observed for the Al_3Ni phase which was

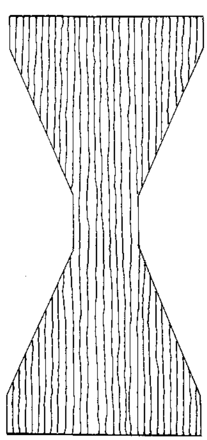

Figure 2 Schematic view of insert type b, $\theta > 8°$, showing the structure orientated in the original growth direction.

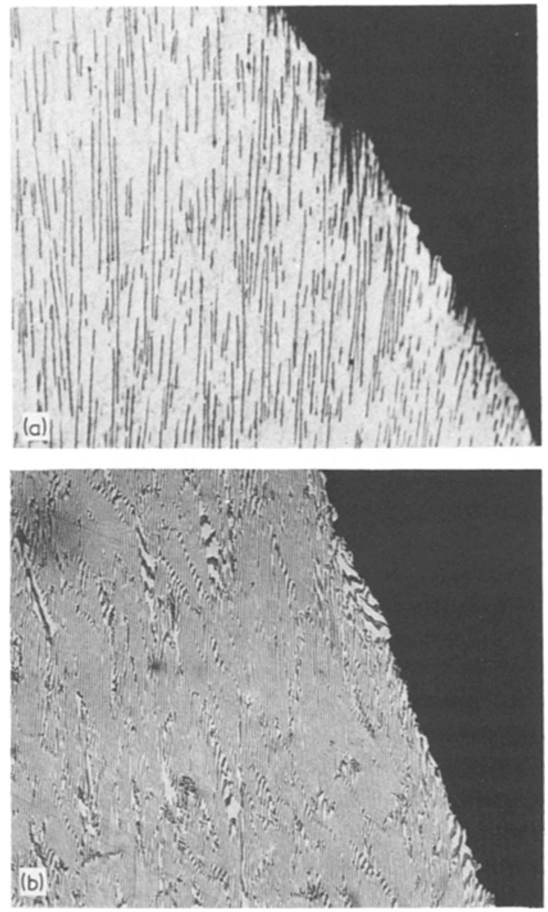

Figure 3 Insert b, lower portion, structure aligned in the original growth direction, longitudinal view, $\times 60$. (a) Sample no. 0008, binary Al–Ni eutectic, $\theta = 18°$. (b) Sample no. 0014, ternary Al–Ni–Cu eutectic, $\theta = 10°$, corresponding schematic Fig. 2.

reversed as the cross-section diverged again. Such observations have been reported previously [9–11] and attributed to various causes. The present results, discussed further below, support Hertzberg's [11] suggestion of a growth rate dependent transition. In all, it is considered that a converging solidification cross-section would not seriously impair the mechanical strength of these composites, although for compositions removed from the eutectic, solute accumulation effects may prove harmful.

At the existing end of the insert in which the specimen cross-section is continuously diverging, the structures depended on the value of θ. For values of θ greater than about 8°, all the rods or lamellae continued to grow parallel to the crucible axis such that their nucleation occurred on the upward facing insert wall. For values of $\theta < 8°$,

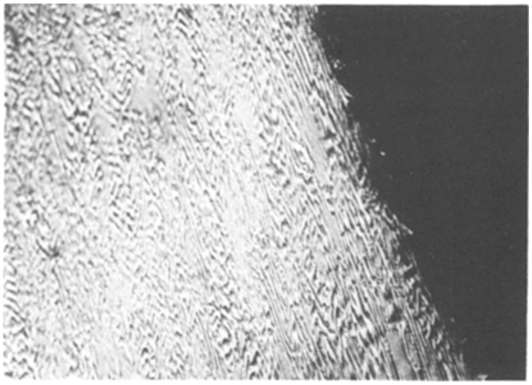

Figure 4 Sample no. 0017, insert type b, ternary Al–Ni–Cu eutectic, upper portion, structure follows the contour of the insert, $\theta = 6°$, longitudinal view, $\times 50$, corresponding schematic Fig. 5.

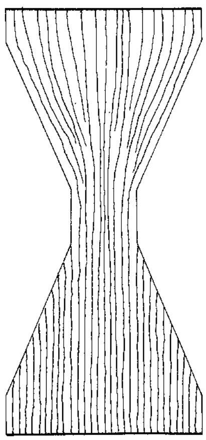

Figure 5 Schematic view of insert type b, $\theta < 8°$, showing the structure following the contour of the insert at the upper portion.

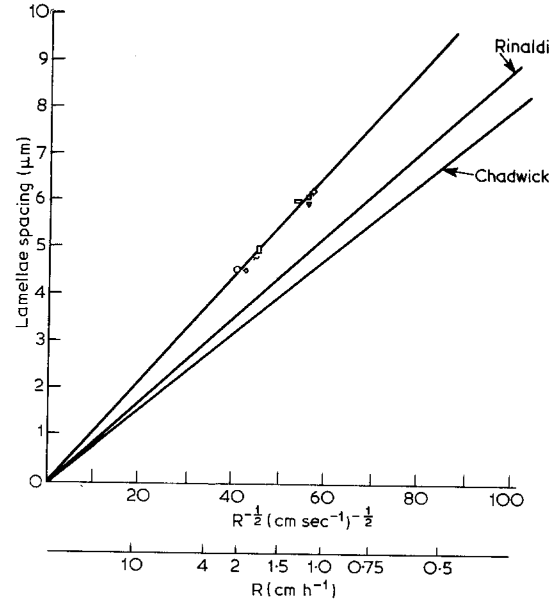

Figure 6 Lamellae spacing as a function of growth rate for ternary Al–Ni–Cu eutectic, Rinaldi's data [3] for compositions containing from 17.5 to 12.3 at. % Cu and from 0.0 to 1.32 at. % Ni, Chadwick's [10] data for the Al–Cu binary eutectic.

regions close to the insert wall exhibited rods or lamellae growing parallel to it, (Figs. 4 and 5) while the central region of the specimen continued to show no deviation in rod or lamellar direction from the crucible axis. Similar results were observed by Farag [9], although he reported a transition in growth structure at around 20° and also by Chadwick [12] who has shown essentially the same effect for diverging cross-section with a small or large radius of curvature. It would suggest that in such regions, the liquid/solid interface becomes curved; although it is unlikely that this would significantly weaken the structure since the rods and lamellae remain essentially aligned.

From an analysis of the rod or lamellar spacings, λ, in regions removed from the inserts, we have obtained for both alloys the expected linear relation of λ versus $R^{-1/2}$ shown in Fig. 6 for the ternary eutectic alloy. Using a serial polishing technique, which yielded results typically shown in Fig. 7, we have, therefore, been able to translate lamellar or rod spacings within the insert into approximate growth rates. This yielded curves for both alloys as shown in Fig. 8, which shows clearly how the growth rate drops with converging cross-section and vice versa. The two scatter bands in this figure result from the grouping of various specimen withdrawal rates employed. Fluctuations within each band can be attributed to minor variations in the withdrawal rate and no correlation with θ was obtained. The measured change in growth rate agrees well with Hertzberg's [11] explanation for the change from rod to plate morphology of the binary Al–Ni eutectic alloy.

3.2. Abrupt contraction of cross-section with simultaneous change of growth direction

These observations relate to the lower regions of insert (a), Fig. 1, and can be sub-divided with respect to the value of θ. In regions to either side of the orifice of insert (a), regardless of θ, the rods or lamellae, simply grew into and terminated at the insert bottom face without deviation or morphology change. For $\theta > 8°$ in the region of the orifice, the matrix developed a multi-grain struc-

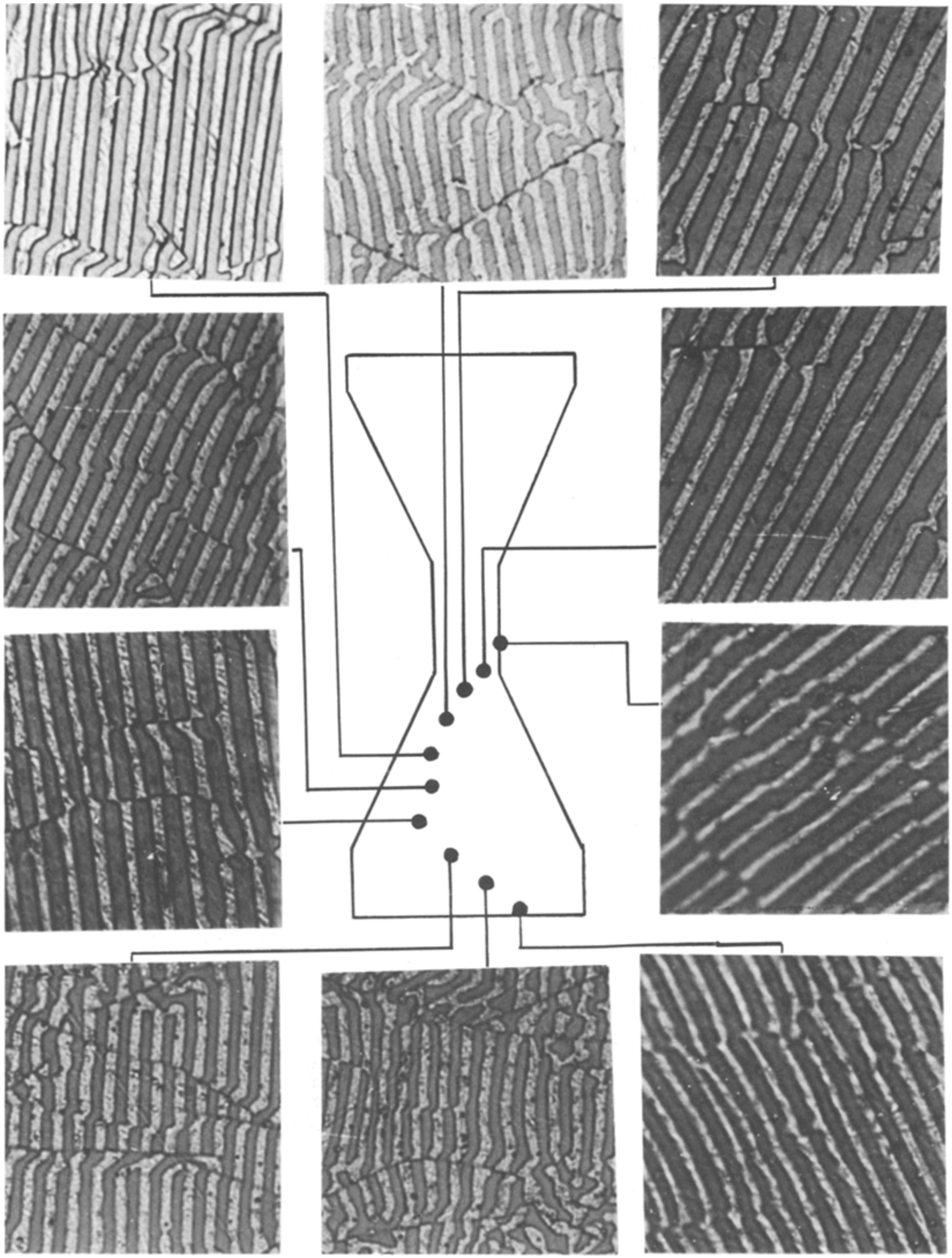

Figure 7 Sample no. 0014, lower half of insert type b, ternary Al–Ni–Cu eutectic, transverse view at various locations, illustrating the change in lamellae spacing × 760.

ture shown in Fig. 9 with the aid of polarized light. The rods or lamellae of the grain near the upper insert wall grew parallel to the insert wall and continued to grow parallel to the crucible axis, and it was this grain which eventually dominated the growth, outgrowing the other two. The rods or lamellae of the central grain adopted an orientation between these extremes as shown in the schematic of Fig. 10. These results suggest that in this region (assuming it can be delineated

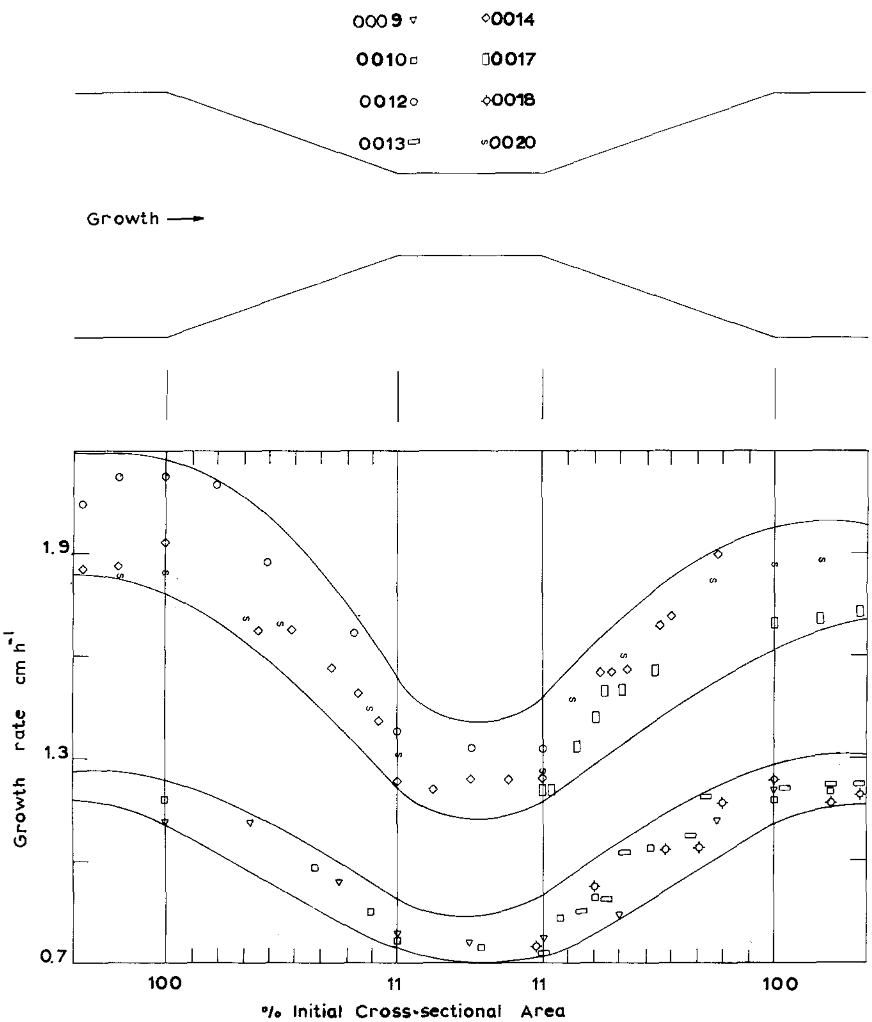

Figure 8 Solidification rate at various percentages of initial cross-sectional area into the portions of insert type b, ternary Al–Ni–Cu eutectic.

Figure 9 Sample no. 0020, insert type a, ternary Al–Ni–Cu eutectic, lower portion, viewed under polarized light, $\theta = 20°$, longitudinal view, $\times 65$.

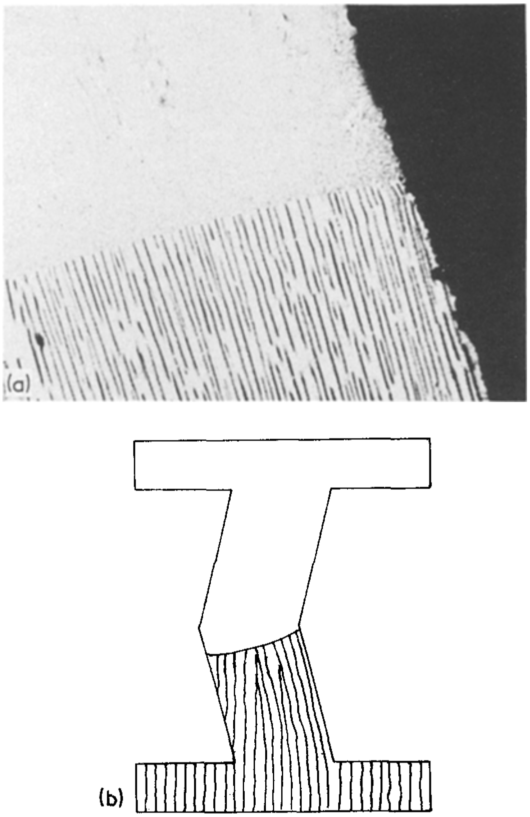

Figure 10 (a) Sample no. 0003, insert type a, binary Al–Ni eutectic, lower portion, location of liquid/solid interface, $\theta = 14°$, longitudinal view, $\times 65$. (b) Schematic.

by constructing perpendiculars to the lamellae), the liquid/solid interface adopts a concave downwards shape. For $\theta < 8°$, no multigrain structure developed and the rods and lamellae tended to align themselves parallel to the insert wall, such that the liquid/solid interface was perpendicular to it.

3.3. Abrupt changes in growth direction with constant cross-section

These observations relate to the central portion of insert (a) in Fig. 1. From experiments in which growth was terminated within the upper portion of the insert, the liquid/solid interface in this region remained planar and perpendicular to the insert wall regardless of the value of θ. These results are confirmed by the observation that also in this region, in all specimens, (and for $\theta < 8°$ for all regions), the rods or lamellae grew parallel to the insert wall consistent with the suggestion of Chadwick that they grow parallel to the local heat flow path. As discussed earlier, for $\theta > 8°$ the

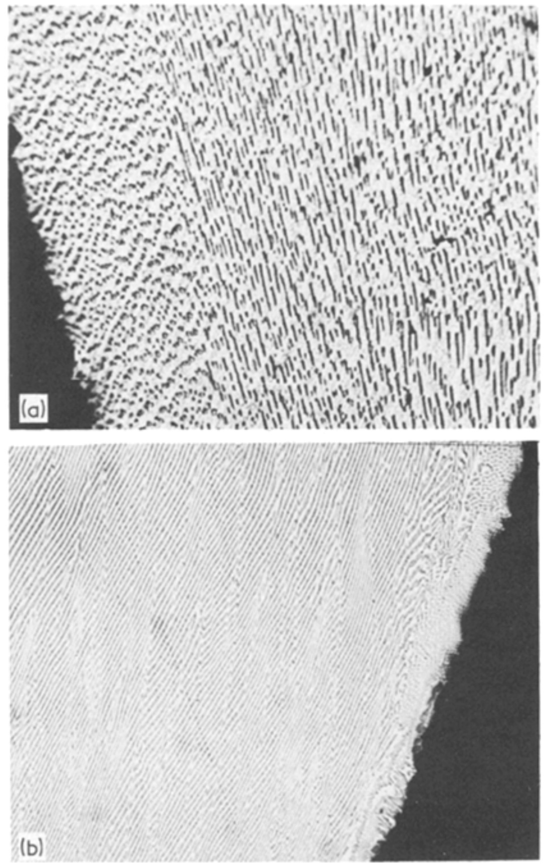

Figure 11. Insert type a, middle portion after the bend, downward face, depleted zone, longitudinal view, $\times 65$. (a) Sample no. 0004, binary Al–Ni, $\theta = 14°$. (b) Sample no. 0013, ternary Al–Ni–Cu, $\theta = 14°$, corresponding schematic Fig. 12.

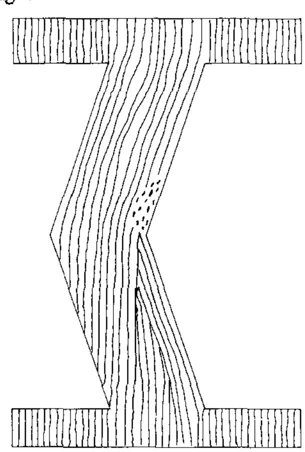

Figure 12 Schematic view of insert type a, $\theta > 8°$, showing the different grain orientations developed.

liquid/solid interface shape would appear to be concave downward in the lower portion of insert (a).

The behaviour of the structure at the bend was dependent upon θ. For angles of θ larger than about 8°, a misorientated zone was observed on the down face in the region just beyond the acute angled corner projection, Figs. 11 and 12. This region corresponds to that in which the local interface movement tends to zero. It also, however, corresponds to the region in which one of three matrix grains completely outgrows its two competitors as discussed in Section 2 and is, therefore, a region in which fresh nucleation must take place. On the upward face of the same specimens, that is, in the region of the oblique angled corners continuous rod or lamellar growth was observed. This was also true for the entire region in specimens with $\theta < 8^\circ$ in which, at all locations, the rods or lamellae grow parallel to the insert wall.

4. Conclusions

(1) Gradual convergence of the solidifying cross-section results in a decrease in the rate of solidification, that is the rate of movement of the composite interface with an associated increase in inter-rod or inter-lamellar spacings. Gradual divergence of the solidifying cross-section causes an equal and opposite increase in the solidification rate. For mild section variations this is not expected to seriously impair mechanical properties.

(2) Gradual divergence of the solidifying cross-section at angles less than 8° cause the surface rods or lamellae to grow parallel to the diverging contour rather than the main growth direction and inter-rod or lamellar spacing decreases.

(3) Gradual divergence of the solidifying cross-section at angles between 8 and 20° does not alter the orientation of the rods or lamellae from their initial alignment.

(4) Abrupt contraction of cross-section, when accompanied by changes in growth direction $> 8^\circ$ and up to 20° result in the formation of new matrix grains with lamellae or rods orientated either parallel to the original growth direction, parallel to the new growth direction or midway between these extremes, the grain or grains with lamellae or rods growing in the original direction eventually dominates.

(5) Abrupt contraction in cross-section accompanied by changes in growth direction $< 8^\circ$ does not produce the multiple grain effect and the rods or lamellae grow parallel to the new growth direction.

(6) Abrupt changes in growth direction of large angle ($> 8^\circ$) at constant cross-section give rise to lamellar or rod misorientations which can be potential areas of weakness. Deviations less than 8° result in continuous lamellar or rod structures parallel to the growth direction.

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