



# Mechanism selection for spontaneous grain refinement in undercooled metallic melts

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## Abstract

In a previous paper (Castle et al., 2014) [17], we employed a melt fluxing technique to study the velocity–undercooling relationship and microstructural development of a Cu–8.9 wt.% Ni alloy in order to investigate the fundamental mechanism behind the rapid solidification phenomenon of “spontaneous grain refinement”. A number of growth transitions were identified with increasing undercooling, including an extended dendrite orientation transition through: fully  $\langle 100 \rangle$ -oriented  $\rightarrow$  multiply twinned, mixed  $\langle 100 \rangle / \langle 111 \rangle$ -oriented  $\rightarrow$  fully  $\langle 111 \rangle$ -oriented. Here, we present the results of an identical study of a Cu–Ni alloy of lower Ni content and observe a similar set of growth transitions, with some notable differences. In particular, three distinct grain refinement mechanisms have been observed between the two alloys, including: recrystallization, dendrite fragmentation and dendritic seaweed fragmentation. It appears that the mechanism selected depends strongly upon the original growth structure present, which we suggest is dictated by the balance between the capillary and kinetic anisotropies; with the addition of Ni to Cu either increasing the strength of the kinetic anisotropy, decreasing the strength of the capillary anisotropy, or a combination of both. The unambiguous identification of three different spontaneous grain refinement mechanisms in two closely related alloys is significant as it may help to resolve some of the debate surrounding the “true” grain refinement mechanism.

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## 1. Introduction

“Spontaneous grain refinement” is a rapid solidification phenomenon observed in metals, in which abrupt transitions between coarse columnar and fine equiaxed grain structures are observed at well-defined values of undercooling,  $\Delta T^*$ . In general, for pure metals (e.g. [1–3]), one grain refinement transition is observed at high  $\Delta T$ ; whilst for alloys (e.g. [4–7]), two grain refinement transitions occur, with an additional transition at low  $\Delta T$  and columnar

growth stabilized at intermediate  $\Delta T$ . First reported in 1959 by Walker [8] for bulk undercooled Ni, spontaneous grain refinement has received a large amount of interest, since it demonstrates that grain refinement can be achieved in a way which is intrinsic to the solidification process itself. This therefore provides an attractive alternative to the conventional chemical or mechanical means of producing grain-refined metals, offering significant commercial potential. Furthermore, since the onset of spontaneous grain refinement alters or removes the original growth structure, continuous measurements of the original dendrite length scale cannot be made as a function of undercooling (and, by extension, growth velocity). This therefore presents problems for the modelling of rapid dendrite growth, since there is a subsequent lack of data on which

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to base and test mathematical models. As such, many studies have been undertaken in order to elucidate the fundamental mechanism behind spontaneous grain refinement. Theories have ranged from: cavitation in the melt, leading to copious nucleation ahead of the solid–liquid interface [8]; partial remelting of secondary and tertiary dendrite branches [9]; recrystallization [2,10,11]; dendrite fragmentation [12–14]; surface-energy-driven remelting and fragmentation of dendrites [15]; and a transition to dendritic seaweed growth followed by rapid remelting at the split dendrite junctions during recalescence [16]. However, the contrasting and conflicting evidence produced by different studies has resulted in a large amount of debate on the matter, with no individual model able to provide a complete explanation for the behaviour observed.

In a previous paper [17], we employed a containerless melt-fluxing technique to undercool and rapidly solidify a Cu–8.9 wt.% Ni alloy. A careful study of the evolution of microstructure and texture with increasing undercooling/solidification velocity was then undertaken in order to further elucidate the grain refinement mechanism. An extended transition in growth orientation was observed, with normal,  $\langle 100 \rangle$ -oriented growth dominating at low  $\Delta T$  and anomalous,  $\langle 111 \rangle$ -oriented growth dominating at high  $\Delta T$ . Within the intermediate undercooling range, the competing anisotropies of the two growth directions were observed to give rise to mixed-orientation dendrites, dendritic seaweed “branches” and twinning, with several samples exhibiting multiply twinned growth [17]. Further analysis of these twinning relationships will be presented in a separate paper in due course. Upon the observed transition to fully  $\langle 111 \rangle$ -oriented growth, a transition to a coarse dendritic grain structure was observed. This was coincident with a positive break in the gradient of the growth velocity vs. undercooling curve, indicating that the  $\langle 111 \rangle$ -oriented growth was faster than the  $\langle 100 \rangle$  growth it replaced. Microstructural analysis indicated that grain refinement at low undercooling had occurred via a recrystallization mechanism, whilst at high undercooling, a partially grain refined sample was obtained, in which the substructure of the non-grain refined portion consisted of dendritic seaweed, suggesting that seaweed is the precursor to grain refinement at high undercooling in this alloy.

Such an extended transition in growth orientation had not previously been reported as a function of undercooling. Abrupt, growth velocity/undercooling-mediated switches in preferred growth orientation have been reported in transparent metal analogue systems [18,19], in strongly faceting Ge–Fe [1] and in Cu–Sn [20]; and an extended “dendrite orientation transition” (DOT) has been experimentally and computationally observed by Dantzig et al. [21] as a function of increasing Zn content in Al. Here,  $\langle 100 \rangle$ -oriented growth is observed for low Zn content and  $\langle 110 \rangle$ -oriented growth is observed for high Zn content. At intermediate Zn concentrations, the close competition between the differently directed anisotropies is observed to give rise to textured, seaweed-like structures. It is subsequently suggested

that the weak anisotropy of the solid–liquid interfacial energy of Al is altered by the addition of Zn, which has a relatively high interfacial energy anisotropy. This reinforced the findings of Haxhimali et al. [22], who had previously reported that changes in the composition-dependent interfacial anisotropy parameters could lead to changes in preferred growth orientation, accounting for the atypical dendrite growth directions observed in some face-centred cubic (fcc) alloys [23–26]. Within this work, an orientation selection map was produced, exhibiting a region of  $\langle 100 \rangle$ -oriented growth, a region of  $\langle 110 \rangle$ -oriented growth and a region of parameter space in between the two orientations in which “hyperbranched” structures were observed. When the corresponding data of several pure fcc metals (from Ref. [27]) were plotted onto this orientation selection map, it was shown that several of these metals—including Cu and Ni—lie close to the  $\langle 100 \rangle$ -hyperbranched boundary. This might therefore suggest that the growth orientation transition observed in our previously investigated Cu–Ni alloy may, at least in part, be due to the alteration of the interfacial energy anisotropy of Cu by the addition of Ni.

Alternatively, the orientation transition may be due to the increasing influence of the kinetic anisotropy over the solidification process as the growth velocity is increased. If sufficient influence is gained, the kinetic anisotropy may compete with, and then override, the capillary anisotropy, perhaps accounting for the observation of a mixed-orientation regime, followed by the transition to fully  $\langle 111 \rangle$ -oriented growth. At the highest velocities recorded, the subsequent, strongly dominant kinetic anisotropy may then account for the final observed transition to seaweed growth. Bragard et al. [28] have shown that a sufficiently high kinetic anisotropy can lead to a transition to dendritic seaweed growth in the vicinity of the  $\Delta T^*$  transition; however, it is noted that this is only when set to unrealistically high values. As such, our observation of seaweed growth suggests that either the model is in some way flawed, or that other factors may be contributing to the development of these growth instabilities. Since it has been shown that the effective crystal anisotropy of the  $\langle 111 \rangle$  orientation is weak [29], it may be that the transition to this orientation, in combination with the increased kinetic influence, is enough to induce a transition to seaweed growth. Alternatively, it may be that a combination of increased kinetic effect and an altered interfacial energy anisotropy (as discussed above with regards to the work of Haxhimali [22] and Dantzig et al. [21]) is giving rise to the growth orientation transition and seaweed growth observed in our previous work.

In order to investigate our previous findings further, the present report details the results of an identical study into a Cu–Ni alloy of lower Ni content. A comparison of the results of the two studies is then given, in order to examine the effect of Ni concentration on the observed growth transitions, and a discussion is subsequently provided in consideration of the possible contributing factors to the behaviour observed.

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