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In situ and real-time 3-D microtomography investigation of dendritic solidification in an Al–10 wt.% Cu alloy

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Abstract

The microstructural evolution of an Al–10 wt.% Cu alloy was investigated during solidification at constant cooling rate by in situ synchrotron X-ray microtomography with a resolution of 2.8 μm. Solidification of this alloy leads to a coarse dendritic microstructure which was fully characterized in terms of variation with temperature of the solid fraction, the specific surface area of the solid–liquid interface and the local curvatures of the solid phase. By analysing the evolution with solid fraction of individual dendrites, at least two coarsening mechanisms were clearly identified in addition to solidification growth. The first mechanism involves remelting of small secondary dendrite arms to the benefit of bigger adjacent arms. The second is the coalescence of adjacent secondary arms, with progressive filling of the inter-arm spacing and coalescence at the tips. Although this mechanism preferentially occurs at high solid fractions, these results show that the evolution of the dendritic microstructure during solidification is complex and involves the occurrence of various mechanisms operating concurrently. In situ X-ray tomography thus allows revisiting the various models which have been proposed to account for dendrite coarsening during solidification.
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1. Introduction

Solidification of metallic alloys in industrial casting processes involves the formation of solid dendrites, the characteristics of which depend on the cooling rate in particular. At high cooling rates, the dendrites are well defined, with primary, secondary and sometimes tertiary dendrite arms. At low cooling rates, conversely, the dendrites evolve strongly during cooling by diffusion of solute in the liquid so that the dendritic structure becomes degenerated. This evolution is even more pronounced when the alloy is held isothermally in the semi-solid range, sometimes leading to globular microstructures suitable for thixoforming.

Based on Kahlweit's observations of organic materials, used as transparent analogue systems for metallic alloys, Kattamis et al. proposed two models that can predict the evolution of microstructural parameters such as the specific surface area of the solid–liquid interface, S_v, during partial remelting [1] and during solidification [2–4]. "Model A", illustrated in Fig. 1a, describes the remelting of a small dendrite arm from its tip to its root to the benefit of bigger adjacent arms; the small arm is assumed to shrink at constant radius while the larger arms grow in radii at constant length. Diffusion of matter is supposed to occur through the liquid. Indeed, according to the Gibbs–Thomson

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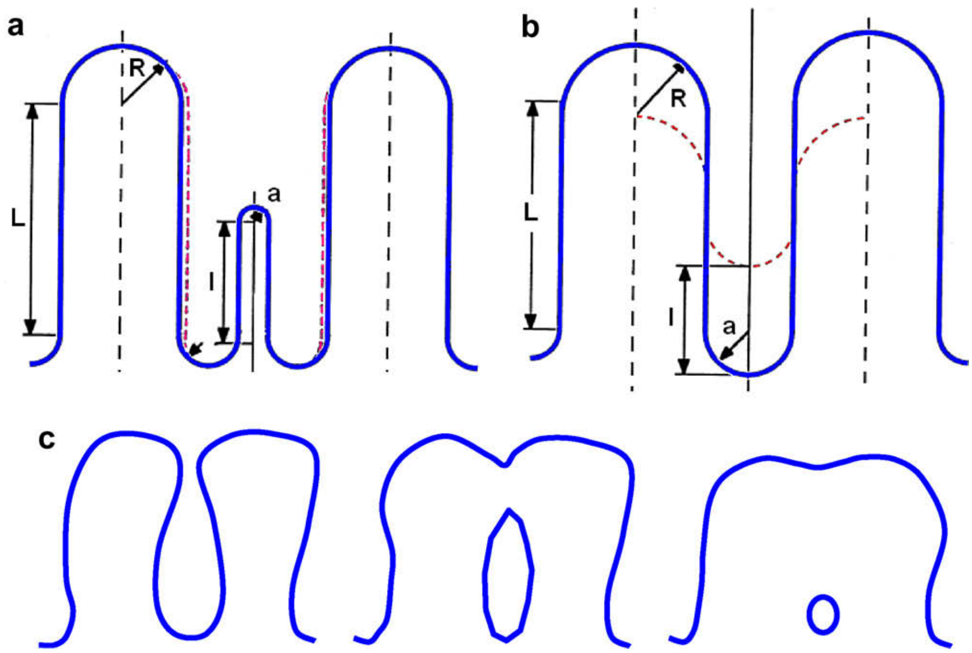

Fig. 1. Diagrams of (a) model A and (b) model B, reprinted from Ref. [4]; and of (c) the coalescence mechanism proposed by Mortensen [6].

equation, the solute concentration in the liquid at a liquid–solid interface is larger for regions with large curvature than for regions with small curvature. Thus, a flow of mass establishes from areas of large curvature towards those of small curvature. “Model B”, illustrated in Fig. 1b, describes the coalescence of adjacent arms with remelting of the tips and deposition at the root between them. These models were shown to describe the evolution of the specific surface area of the solid–liquid interface quite well when applied to post-quench experiments, particularly for short holding times. Young and Kirkwood [5], and later Mortensen [6], also proposed several models to describe coalescence of adjacent dendrite arms. A coalescence mechanism reported by Mortensen is illustrated in Fig. 1c: the dendrite arms have tear shapes, which may result from dissolution of the dendrite arm at the root and redeposition at the tip [5], and they coalesce at their tips with a liquid pool that eventually forms at the root.

Usually, the microstructures of metallic alloys are studied on metallographic sections obtained after complete solidification of the specimen or by quenching from the semi-solid state. Recently, the development of synchrotron techniques has allowed in situ observations of the evolution of microstructure during solidification. In particular, microradiography has been extensively used to study the solidification of alloys such as Al–Ni [7], Al–Cu [8] and Sn–Bi [9] in real-time. Radiography permits observation of the mechanisms at work at a time interval as low as 0.15 s [8]. This small time resolution allows visualization of the first stages of solidification inside samples thin enough (100–200 μm thick) for a single layer of dendrites to be observed. For example, Reinhart et al. [7] were able to observe and identify the interactions that occur between dendrites during the columnar to equiaxed transition (CET) using a large field of view, e.g. $15 \times 15\ \mathrm{mm}^2$, inside the sample. This allows them better understanding the factors, such as the pulling rate, that influence the CET [7]. However, a large field of view could only be obtained to the detriment of the spatial resolution, i.e. about 7–10 μm. Thus, the fineness of the microstructure, combined with a low absorption contrast between the solid and liquid phases at the beginning of solidification, make quantitative analysis on the scale of the dendrite arms difficult. Reducing the field of view to $1.5 \times 1.5\ \mathrm{mm}^2$ allows reaching a spatial resolution of 1.5 μm. For example, this has been used to obtain quantitative information about the solute concentration field in the liquid to understand how fragmentation of dendrites occurs [8,10]. In situ microradiography has mainly been applied to directional solidification conditions, i.e. with a temperature gradient, except for a study by Li et al. [9] in which the solidification conditions involved no temperature gradient. Real-time observations of dendritic semi-solid Sn–Bi using synchrotron microradiography permit qualitative observations of the mechanisms that operate on the dendritic microstructure, such as coalescence and dendrite arm remelting from tip towards root [9]. It was reported that coarsening by the remelting of the small dendrite arms prevails over coalescence, although both are observed at slow cooling rates, and that coarsening primarily affects the dendritic morphology during the early stages of solidification, i.e. 15–25% of the total solidification time [9].

The aim of the present study was to observe, in real-time and in three dimensions, the microstructural evolution of an Al–Cu alloy during solidification with no temperature gradient using fast X-ray microtomography in order to gain a better understanding of the dendrite arm interactions and to assess the validity of existing models.

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