



## Review

# Characterization of microstructures and growth orientation deviating of Al<sub>2</sub>Cu phase dendrite at different directional solidification rates



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## ABSTRACT

At different directional solidification rates (10, 20 and 100 μm/s), microstructures and growth orientation variations of Al<sub>2</sub>Cu dendrite in Al-40%Cu alloy were characterized. When solidification rates were ranged from 10 to 100 μm/s, three-dimensional microstructure of Al<sub>2</sub>Cu dendrite changed from faceted L-shaped patterns to non-faceted complex dendrite morphology in transverse section. By the macro and micro orientation analysis characterize methods, [001] growth direction of Al<sub>2</sub>Cu dendrites with different morphologies was determined. The deviation angle between [001] direction and the heat flow direction was increased with solidified rate increasing. The experimental results showed that the regular solidified microstructure and growth orientation along the heat flow direction could be well controlled under lower directional solidification rate.

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## 1. Introduction

In the process of dendrite growth, the primary dendrite can form various morphologies and microstructure distributions under directional solidification [1–5], which could result that the toughening methods mainly including the fine grain toughening and the

second phase toughening occurs. This seriously leads to the crack propagation path deflecting, which improves the fracture toughness and affects mechanical property of the alloys. Thus, the study on dendrite growth is a significant practical importance [6,7]. So far more works indicated that dendrite microstructure evolution was strongly related on the directional solidification rate and the alloy composition, especially the former effect factor. The effect of solidification rate on dendrite growth morphology should be concerned. Li et al. [8] observed various Si dendrite microstructures and the columnar to equiaxed dendrite transition in Al-Si alloy

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under different directional solidification rates. Wang et al. [9] studied the microstructure evolution of Al-Cu alloy through changing the directional solidification rate. They found that the planar-to-cellular transformation of Al dendrite morphology occurred with the solidification rates increasing. Besides, Liu et al. [10] investigated the interface morphologies of single crystal super alloy CMSX-2 and found that the evolution of interface morphologies of dendrite had been changed with directional solidification rate increasing. The above results indicated that the solidification rate played a vital role in dendrite growth.

On the other hand, when dendrite morphologies evolution occurred at different directional solidification rates, its growth orientation variations were also accompanied [6]. Then, dendrite would grow along its growth orientation in growth process. So it is very important to investigate the growth orientation variations of dendrite. Until now, more previous studies ascribed that there had been three main specific directions for dendrite growth under directional solidification: the dendrite growth orientation, the heat flow direction and the preferred crystallographic orientation [11]. And then the relationship among them was transformed with the solidification rate changing. Ardakani et al. [12] showed that the growth orientation of nickel-base single crystal super alloys was [001] direction, which was between the heat flow direction and the preferred orientation during directional solidification. Further, Esaka and Shionzuka et al. [13] found that the dendrite growth orientation gradually turned toward its preferred orientation with the directional solidification rate increasing. However, until recently, the deviation degree between them were not given and discussed, which would lead to the dendrite growth direction not controlled well. Therefore, it is necessary to provide effective analysis methods to characterize dendrite growth orientation deviating during directional solidification.

Thus, in this work, the two-dimensional (2D), three-dimensional (3D) microstructure evolution and growth orientation of Al<sub>2</sub>Cu dendrite were investigated during directional solidification. Moreover, the deviation relationship between the dendrite growth orientation and the heat flow direction was characterized and analysed by the macro orientation (the rotating orientation X-ray diffraction) and the micro orientation (the electron back-scattered diffraction) analysis methods.

## 2. Experimental procedures

### 2.1. Materials

Al-40 wt.% Cu alloy used in this study was prepared in a vacuum induction melting furnace with purity aluminium (99.9 wt.%) and copper (99.99 wt.%). The composition of the ingot measured by chemical titration is Al-39.2 wt.% Cu. The cast sample were enveloped in high purity Al<sub>2</sub>O<sub>3</sub> tube with a inner diameter of 4 mm and length 100 mm. Directional solidification experiments were carried out using a Bridgman vertical vacuum furnace described elsewhere [14]. In this study the thermal gradient was measured using a  $\Phi$ 0.5 mm NiCr–NiSi thermocouple and its value was about 250 K/cm at 10  $\mu$ m/s. In directional solidification process, the sample was heated by a graphite heater at 750 °C and then held isothermal for 20 min. Subsequently, the sample was moved downwards at 10, 20, and 100  $\mu$ m/s, respectively. In order to keep the S/L interface, when the directional solidification distance reached 50 mm, the sample was quenched into a liquid Ga–In–Sn pool.

### 2.2. Characterization

The directionally solidified samples obtained in the experiments were then sectioned horizontally and vertically, respectively. And

the specimens polished and etched using solvent Kroll of H<sub>2</sub>O (46 mL) + HNO<sub>3</sub> (3 mL) + HF (1 mL) for about 15 s. The scanning electron microscopy (SEM, JSM-6390A) were employed to photograph the specimens microstructures. The materialise's interactive medical image control system (Mimics) software was applied to reconstruct the three-dimensional (3D) microstructures images of the primary Al<sub>2</sub>Cu phase. The growth orientations were investigated by means of the X-ray powder diffraction (XRD, D/max-3) and the electron back-scattered diffraction (EBSD) in scanning electron microscopy (SEM, Zeiss Supra 55) equipped with the Channel 5 EBSD system (HKL Technology-Oxford instrument). In addition, the deviation angle in directional solidified alloy was measured by the rotating orientation X-ray diffraction (RO-XRD) method in X-ray diffractometer (XRD, Rigaku's D/max2400).

## 3. Results and discussions

### 3.1. Microstructure evolution (2D and 3D)

It is well known that the solidified microstructures of Al-40%Cu alloy consist of primary  $\theta$ -Al<sub>2</sub>Cu phase and eutectic (Al/Al<sub>2</sub>Cu) phase based on the Al-Cu phase diagram shown in Fig. 1. In directional solidification process, when the melt temperature was cooling down, the  $\theta$ -Al<sub>2</sub>Cu phase would be precipitated firstly as the primary phase from the liquid metals and grew along its growth directions to form dendrite morphology. With a large number of Al<sub>2</sub>Cu dendrite precipitating and growing, the composition of the remaining liquid melt was gradually reduced to the eutectic composition. Then the remaining liquid occurred the eutectic reaction and developed in the form of the coupled eutectic (Al/Al<sub>2</sub>Cu). When at directional solidification rate of 10  $\mu$ m/s, the primary Al<sub>2</sub>Cu dendrite could be clearly distinguished from the eutectic in Fig. 2(a). The dendrite displayed regular faceted L-shaped patterns [15] in transverse section and the eutectic exhibited regular lamellar structure morphology. When solidification rate increased to 20  $\mu$ m/s, Al<sub>2</sub>Cu dendrite displayed irregular non-faceted L-shaped patterns and I-shaped patterns shown in Fig. 2(b). The surface of the dendrite began roughening, which resulted in its edges and corners gradually disappeared. Moreover, the dendrite size was smaller than alloy at 10  $\mu$ m/s. While the primary Al<sub>2</sub>Cu dendrites surface roughened further with the solidification rate increasing. Then at 100  $\mu$ m/s the dendrite displayed irregular complex three lobately morphology with non-faceted characteristics in Fig. 2(c). The dendrite edges and corners were completely smooth and its size was smallest, which

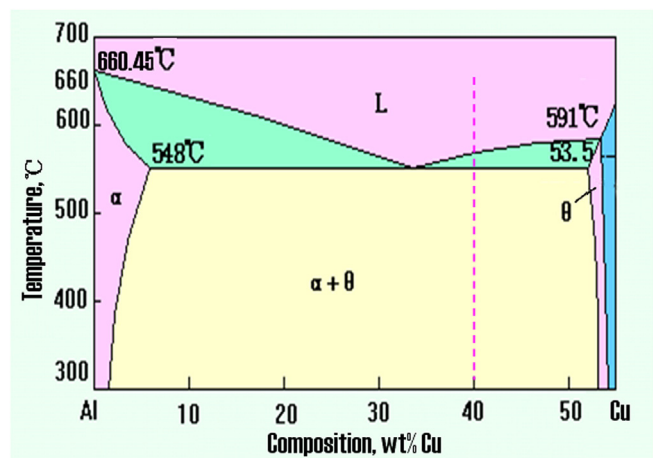


Fig. 1. Phase diagram of Al-Cu alloy.

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