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Study of columnar–equiaxed transition and anaxial columnar dendrites growth of hypoeutectic alloy with synchrotron radiation

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Abstract. Among solidification processes, the columnar to equiaxed transition (CET) is critical component for formation either of microstructure or macrostructure. Directional solidification of Al-Cu alloy was performed at the BL13W beamline of Shanghai Synchrotron Radiation Facility (SSRF). The imaging results reveal that CET was provoked by external thermal disturbance. The fragment of dendrite tips were come from solute-rich-zone. And they floated to the mushy zone to form the equiaxed dendrites. As the conditions are suitable, a new dendrite morphology sprout and grow up, in which dendrites grow along <110> directions in a binary Al-Cu alloy. These dendrites have no obvious primary arms and were named anaxial columnar dendrites.

1. Introduction
During directional solidification, the thermal flux direction and melt flow are considered that play an important role[1] in the evolution of liquid-solid interface morphology. Among the interface morphology evolutions, the columnar-to-equiaxed transition (CET) is an important part which is still indistinct at some points [2]. Detachment, drift and growth of the fragments of dendrite during solidification, has been widely accepted as the inducement of CET [3-6]. But the internal trig mechanism is still the basic to clarify solidification process.

Based on the imaging observations, the behaviors of fragment in the front of S/L interface were analyzed and discussed. At the same time, the new growth style of dendrite was found. These dendrites have no obvious primary arms and were named anaxial columnar dendrites[7]. They were formed at low thermal gradient (G,T=4.0 K/mm) and solidification speed (v=0.067 mm/s) and are hard to be identified in a traditional metallographic test.

2. Experimental procedure
The solidification was carried out inside a resistance furnace with two opposite windows. 15wt% Al-Cu alloy were prepared by 5N purity Al and Cu in a graphite crucible. Rectangular slices with 60×15×0.7
mm³ in size were cut and sandwiched between two 120-μm-thick Al₂O₃ plates. The edge of the samples was sealed by high temperature adhesive. Four thermocouples were embedded into the sample with a interval of 8 mm along the edge to measure the temperature gradient during directional solidification.

The synchrotron imaging experiments were performed at beam line BL13W of SSRF. The sample was fixed upright in the holder of the furnace and heated to about 40 K higher than the melting point of the alloy. The cooling rate was kept at R=0.028 K/s and the temperature gradient over the region to be observed was \( G_T = 5.0 \text{ K/mm} \). An incident monochromatic X-ray energy of 20 keV was employed. A detector dead time was 0.2 s, together with an exposure time of 2.5 s. The full image field of view corresponded to \( 6.8 \times 3.4 \text{ mm}^2 \).

3. Results and discuss

3.1. Behavior characteristics of fragment

According to the phase diagram of binary Al-Cu system, the alloy with a nominal composition Al-15 wt%Cu, has a typical hypoeutectic structure. As the eutectic temperature (821.4 K) of Al-Cu alloys is much lower than that (~893 K) of the liquidus, a wider mushy zone would exist during directional solidification. The main dendrite growth and its morphology evolvement happen in the melt in front of the mushy zone (~1-2mm in thickness here), which corresponds to the constitutional supercooling zone.

The whole process of CET during the directional solidification of the alloy was shown in Fig 1. The moment right before CET occurs was set at \( t=0.0 \text{ s} \) (Fig 1a). A slight indication of interface instability was observed at the tips of the dendrites. Meanwhile, a dendrite fragment (named A) detached from the dendritic network was found in a liquid pocket at the right side of the observed area. The interface instability of the columnar dendrites became obvious in Fig 1b. The growth of the columnar dendrites was inhibited and the equiaxed characteristics appeared from the newborn dendrites. The protuberances at the columnar dendrite tips grew up rapidly in different directions (Fig 1b).

It was also noted that the detached dendrite tended to drift upwards away from its original location. Besides, Dendrite A rotated when it floated up in the melt. The rotation angle of the dendrite was measured and shown in Fig 1c. Its axis rotated clockwise 32.2° during 10 seconds floating, indicating that there is a micro melt flow paralleled to the growth interface of the dendrites. Tiny needlelike dendrite fragmentations, named B-D respectively, were observed in Fig 1(c, d, e, g). Their movement and growth processes could be tracked during the observation clearly. According to the initial shape and the size of the fragmentations, it could be inferred that they evolved from the broken dendrite arms, especially the secondary and tertiary arms detached from the dendritic network. From Fig 1f, Fragmentation B had drifted next to the upper edge of the observed area and became a big dendrite. When its movement stopped, the growth space for the dendrites next to it was limited and the solute in the melt between them enriched gradually. A fracture process of a big arm (named G) was recorded by Fig 1g. The detached dendritic arm had two indistinct images to which the arrows pointed at the same figure because of the long exposure time. The result indicated that the fracture of the dendrite was very sudden. The arm moved upwards quickly after fracture and then stopped because of the obstruction of Dendrites B and D, which stood above it, as shown in Fig 1h. From Fig 1h, the growth front of the mushy zone had proceeded out of view. Although the dendrite tips in the view field almost stopped growing, the coarsening of dendrites and the solute enrichment were still found by further observations.

3.2. Anaxial columnar dendrites growth

Anaxial columnar dendrites have special growth characteristics that are completely different from regular dendrites. There is no axis (primary arm) as the trunk of a dendrite. It is composed of a pair of laterally growing dendrites which are split by a linear liquid zone. In this synchrotron imaging work, it is very
coincidental to capture the images of the anaxial columnar dendrites at the time of early, as shown in Fig 1(b-d). The two buds of anaxial columnar dendrite, marked 1 and 2, grew up from the stem of the fragment A shown in Fig 2(a,b), in which their growth angles were measured and noted. According to the growth process of fragment A, it can be found that: anaxial columnar dendrite 1 born at the moment while fragment A started to rotate. At that time, the tip of anaxial columnar dendrite 1 just pointed to the opposite direction of heat flow (see Fig 1a). It is not coincidence. With the rotation of fragment A, the anaxial columnar dendrite 2 was born and pointed to the opposite direction of heat flow, too (see Fig 2b). Further, the relative position of anaxial columnar dendrite 2 was similar to dendrite 1 which nucleated at the verge of the solute boundary layer ahead of the growth interface. From these facts, it can be inferred that there is a lot of chances for anaxial columnar dendrite nucleation under suitable thermal gradient and solute concentration.

It is well known that the preferred growth direction of aluminum solid solution which is the primary phase of hypoeutectic alloy is [100][8]. Among the growth direction system, only [110] is angled 45 degrees from [100] direction, as noted in Fig 2. The dendrite fragment A in Fig 2 inherited the favorite growth direction of the original columnar dendrite. Therefore, the anaxial columnar dendrites head to [110].

![Fig 1. Columnar-to-equiaxed transition during directional solidification of an Al-15 wt.% Cu sample (R=0.028 K/s, Gτ =5.0 K/mm). g – direction of gravity, 1 and 2 – anaxial columnar dendrite. (a) columnar dendrites, (b) interface instability, (c) rotation of dendrites, (d) fragmentations, (e) drift and growth of fragmentations, (f) dendrite growth is blocked, (g) fracture of dendritic arm and (h) stagnation of dendrite growth.](image-url)
4. Conclusion
The nature convection, caused by external thermal disturbance, leads to growth interface instability and triggers CET. The well-developed columnar dendrites of hypoeutectic alloy can provide the open solute enriched zone. This zone is the only area in which the high order arms of dendrite could detach from matrix.

A new anaxial columnar dendrite morphology was found to appear in directional solidification of the Al-15wt.% Cu alloy. Its growth process was observed using the real-time X-ray imaging technique. The anaxial columnar dendrite is composed of a pair of stems, which are divided by a narrow liquid zone located in its center. This work also provides experimental evidences for the crystalline orientation relationship between the regular and anaxial columnar dendrite. The regular dendrite grows along <100> directions, while the columnar dendrite grows along <110> directions.

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