Effects of Strong Magnetic Field on Texture and Grain Boundaries During Directional Solidification of Al-4.5Cu Alloy Postprint

Authors: Zhong Hua, Zhongming Ren, Li Chuanjun, Zhong Yunbo, Xuan Weidong, Wang Qiuliang

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Abstract

The formation behavior of textures and the distribution of grain boundary characteristics in solidification structures during directional solidification under a strong magnetic field were investigated for paramagnetic Al-4.5Cu alloy with Al-5Ti-1B refiner addition. The results indicate that at a temperature gradient of 27 K/cm, without magnetic field application, grain orientations are disordered after refinement. After applying a magnetic field, with increasing magnetic field strength, grain orientations change, and grains become aligned along the easy magnetization axis ⟨310⟩ of a-Al. With the formation of the ⟨310⟩ texture, the fraction of coincidence site lattice (CSL) grain boundaries in the grains increases. The rotation of magnetocrystalline anisotropic a-Al grains in the melt under magnetic torque in the magnetic field is the primary cause of texture formation. The effects of fluid flow under the magnetic field on texture formation and grain boundaries are also discussed.

Full Text

Preamble

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Texture Formation and Grain Boundary Characteristic of Al-4.5Cu Alloys Directionally Solidified Under High Magnetic Field

ZHONG Hua[1], REN Zhongming[1], LI Chuanjun[1], ZHONG Yunbo[1], XUAN Weidong[1], WANG Qiuliang[2]

[1]) State Key Laboratory of Advanced Special Steel, Shanghai University, Shanghai 200072

[2]) Institute of Electrical Engineering, Chinese Academy of Sciences, Beijing 100190

Correspondent: REN Zhongming, professor, Tel: (021)56331102, E-mail: renzm2201@163.com

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Abstract

Directional solidification of Al-4.5Cu alloy refined by adding Al-5Ti-1B has been carried out to investigate the texture formation and grain boundary characteristic of the paramagnetic crystal under a high magnetic field. OM and EBSD were applied to analyze the microstructures solidified at different temperature gradients (G) and magnetic field intensities (B). The results show that at the temperature gradient of 27 K/cm, the orientations of fcc α-Al grains without magnetic field are random. However, as a high magnetic field is imposed, the easy magnetization axes $\langle 310\rangle$ of the α-Al grains align parallel to the direction of the magnetic field, leading to $\langle 310\rangle$ texture. Meanwhile, the ratio of coincidence site lattice (CSL) grain boundaries increases with the increment of magnetic field intensity and reaches its maximum value at 4 T, but decreases as the magnetic field enhances further. On the other hand, when the temperature gradient is elevated, columnar dendrite morphology is exhibited without magnetic field; while a 6 T high magnetic field is introduced, the columnar dendrites are broken and equiaxed grains of random orientations are obtained. The alignment behavior of the free crystals in melt could be attributed to the magnetic crystalline anisotropy of α-Al. Moreover, the influence of fluid flow on the texture formation and CSL grain boundary development under magnetic field is discussed. The absence of convection is beneficial for grain reorientation and CSL boundary formation. The application of high static magnetic field will inhibit the macro-scale convection. However, the interaction between thermoelectric current and magnetic field will cause micro-scale fluid flow, i.e., thermoelectric magnetic convection (TEMC). The TEMC will give rise to perturbation near the solid-liquid interface, leading to the appearance of freckles as well as the decreasing of the ratio of CSL boundary. Moreover, it is proposed that the formation of CSL boundary is associated with the rotation of the free grains in melt along specific crystallographic axes by magnetic torque.

Keywords: high magnetic field, Al-4.5Cu alloy, Al-5Ti-1B refinement, directional solidification, orientation, grain boundary characteristic

Introduction

Texture has a significant influence on the mechanical and physical properties of polycrystalline materials, and obtaining specific textures through controlled processing has long been a research focus. During texturing, grain boundary characteristics often undergo concurrent changes. Garbacz and Grabski proposed that texturing strongly affects grain boundary characteristic distribution, with different texture types corresponding to different proportions of coincidence site lattice (CSL) boundaries. Studies have shown that increasing the proportion of CSL boundaries can improve material strength, ductility, and corrosion resistance.

In recent years, with advances in superconducting technology, high magnetic fields have been widely applied in materials processing. Both solid-solid and solid-liquid phase transformations in magnetic fields can induce texture formation. Ban et al. observed that during bulk solidification of Al-2.89Fe alloy in a 12 T magnetic field, primary Al_3Fe phases aligned along the easy magnetization direction [121]. Zuo et al. investigated the bulk solidification behavior of Fe-49Sn alloy under high magnetic fields and found that with increasing magnetic field intensity, the alignment of Fe-rich phases parallel to the magnetic field direction increased, and the X-ray diffraction intensity of α-Fe (110) planes was enhanced. Shen et al. studied the effect of longitudinal high magnetic fields on the directional solidification microstructure of Al-40Cu hypereutectic alloy, reporting that primary Al_2Cu phases aligned along the magnetic field direction. Li et al. examined the orientation behavior of primary phases in Al-Ni and Bi-Mn systems during directional solidification under high magnetic fields, demonstrating that Al_3Ni and BiMn primary phases aligned along the magnetic field direction. Watanabe et al. observed that during annealing of Fe-Co alloys under magnetic fields, the proportions of low-angle and CSL boundaries increased with magnetic field intensity, and these low-energy special boundaries were proportional to texture intensity in the material. Sun et al. found that a certain proportion of CSL boundaries appeared in fine-grained pure Al obtained through electromagnetic oscillation under high magnetic fields. Previous studies have primarily focused on ferromagnetic solid solution alloys or intermetallic compounds with lower crystal symmetry such as hexagonal and tetragonal structures, with less research on paramagnetic fcc solid solution alloys.

When metals undergo directional solidification under high magnetic fields, thermoelectric effects exist in addition to magnetic orientation. Thermoelectric effects can drive fluid flow in the liquid phase and induce stresses in the solid phase, making microstructure evolution during solidification more complex. Li et al. found that during directional solidification of Al-4.5Cu alloy under high magnetic fields, fine-grained structures could be obtained while the proportions of low-angle and $\Sigma 3$ boundaries increased, which was attributed to the effect of thermoelectric magnetic forces acting on columnar dendrites. In this study, by adding Al-5Ti-1B refiner to Al-4.5Cu alloy to suppress columnar grain formation and ensure freely mobile grains exist in the mushy zone, we investigate texture formation and grain boundary characteristic distribution in paramagnetic fcc α-Al solid solution alloys during directional solidification under high magnetic fields without the influence of thermoelectric magnetic forces.

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1 Experimental Methods

High-purity metal raw materials (99.99% Al and 99.99% Cu) were weighed according to the Al-4.5Cu alloy composition and placed in an alumina crucible. The charge was heated to 720 °C in a resistance furnace, held for 20 minutes after melting, and stirred uniformly with a graphite rod. Following degassing (with high-purity Ar gas) and slag removal, Al-5Ti-1B refiner was added at 0.02 wt% Ti relative to the total melt weight. After holding for 5 minutes, the melt was suction-cast into quartz tubes with 3 mm inner diameter and cooled in air. The resulting rods were ground with sandpaper, ultrasonically cleaned in acetone, and sealed in alumina tubes (3 mm inner diameter, 5 mm outer diameter, 200 mm length) for directional solidification experiments.

The experimental apparatus is described in reference [20]. The longitudinal high static magnetic field was generated by a 10 T superconducting magnet. The directional solidification furnace was a Bridgman-type furnace with temperature control accuracy of $±$0.1 °C, enabling different temperature gradients in the samples by adjusting furnace temperature. Using the method described in reference [21], temperature gradients were measured at different furnace temperatures: 27, 65, and 101 K/cm at furnace temperatures of 750, 850, and 950 °C, respectively. Before withdrawal, the sample was held in the furnace for 30 minutes to achieve partial melting, after which the stepper motor was activated to pull the sample downward. In these experiments, the withdrawal rate was constant at 10 mm/s. After stable growth over a certain distance (50-60 mm), the sample was quenched into a water-cooled chamber containing Ga-In-Sn liquid metal at a rate of 15000 mm/s. The solid-liquid interface position was predetermined for different temperature gradients, and the furnace height was adjusted to ensure the solid-liquid interface remained within the uniform magnetic field region during solidification.

The quenched solid-liquid interface region was sectioned for metallographic observation. Longitudinal sections parallel to the magnetic field direction were prepared by grinding and polishing, then etched with 0.5% HF solution (by volume) to reveal the microstructure, which was examined using a Leica DM6000 optical microscope (OM). The steady-state growth portion was sectioned for orientation and grain boundary analysis. Transverse sections perpendicular to the magnetic field direction were prepared by electropolishing and analyzed using electron backscatter diffraction (EBSD) in an Applo300 scanning electron microscope (SEM) equipped with an Oxford Instruments Nordlys EBSD detector. The acquired data were processed using Channel 5 software from HKL Technology.

2 Experimental Results

[Figure 1: see original paper] shows the microstructures near the solid-liquid interface (indicated by dashed lines) quenched under different magnetic field intensities at a temperature gradient of 27 K/cm. Uniformly distributed tiny grains can be observed in the liquid phase ahead of the solid-liquid interface, indicating that the heterogeneous nucleation substrate particles obtained after adding Al-5Ti-1B refiner were uniformly distributed in the liquid phase. After directional solidification began, the liquid phase ahead of the solid-liquid interface became undercooled, and α-Al nucleated and grew on these substrates. As solidification proceeded, grains in the mushy zone continuously settled, resulting in a fine-grained structure. Without magnetic field (Fig. 1a), the solid-liquid interface exhibited significant undulations, indicating fluid flow in the melt. When a 2 T magnetic field was applied (Fig. 1b), the interface undulations decreased and "freckle-like" structures appeared in the solid phase, which are generally considered to be associated with fluid flow. At 4 T magnetic field intensity (Fig. 1c), the solid-liquid interface became relatively flat, the freckle-like structures disappeared, and the grains were relatively dense. When the magnetic field intensity increased to 6 T (Fig. 1d), the solid-liquid interface became inclined and new freckle-like structures appeared at the sample edge, indicating new flow in the mushy zone. These results demonstrate that at constant temperature gradient and withdrawal rate, fluid flow in the melt was first suppressed as magnetic field intensity increased (0-4 T), but new flow was generated when the magnetic field increased further (6 T).

[Figure 2: see original paper] presents the solidification microstructures near the quenched solid-liquid interface under different temperature gradients with and without magnetic field. At temperature gradients of 65 and 101 K/cm without magnetic field, the solidification structures exhibited columnar grain morphology (Figs. 2a and c). Under a 6 T magnetic field, columnar grains were broken (Figs. 2b and d) and equiaxed grain morphology appeared (Fig. 2d).

EBSD analysis was performed on the transverse sections of the steady-state growth portion to obtain grain reconstruction maps, as shown in [Figure 3: see original paper] and [Figure 4: see original paper], where grain colors correspond to different orientations. Statistical analysis revealed that at a temperature gradient of 27 K/cm, the average grain diameter was 143 m without magnetic field, and increased to 211, 165, and 215 m under magnetic fields of 2, 4, and 6 T, respectively. When the temperature gradient increased to 65 K/cm, the average grain diameter was 269 m without magnetic field and 236 m under 6 T magnetic field. At 101 K/cm temperature gradient, the average grain diameter was 397 m without magnetic field and 285 m under 6 T magnetic field.

[Figure 5: see original paper] shows inverse pole figures (IPFs) of grains in the steady-state growth portion under different magnetic field intensities at a temperature gradient of 27 K/cm. Different colored spots in the IPFs represent grains with different orientations. Without magnetic field and under 2 T magnetic field, grains were randomly distributed (Figs. 5a and b). When the magnetic field intensity increased to 4 T, the orientation distribution began to change (Fig. 5c). Under 6 T magnetic field, most grains clustered near the ⟨310⟩ orientation (within the dashed semicircle in Fig. 5d).

[Figure 6: see original paper] presents IPFs of grain orientations in the steady-state growth portion under different temperature gradients with and without magnetic field. Without magnetic field at 65 K/cm temperature gradient, grain orientations began to converge toward the ⟨001⟩ pole in the IPF due to partial columnar grain formation (Fig. 6a), with fewer grains near the ⟨310⟩ orientation (within the dashed semicircle in Fig. 6b). When the temperature gradient increased to 101 K/cm, the solidification structure consisted mainly of columnar grains with orientations concentrated near the ⟨001⟩ pole (Fig. 6c), representing the preferred growth direction of α-Al. After applying a 6 T magnetic field, columnar-to-equiaxed transition was induced by the magnetic field at temperature gradients of 65 and 101 K/cm, resulting in equiaxed grain structures but with random orientations (Figs. 6b and d).

Using EBSD grain boundary characteristic maps ([Figure 7: see original paper]), the proportion of CSL boundaries in the steady-state growth portion was statistically analyzed at a temperature gradient of 27 K/cm under different magnetic field intensities. The results show that the proportions of CSL boundaries were 12.3%, 12.7%, 15.9%, and 14.8% at magnetic field intensities of 0, 2, 4, and 6 T, respectively. The proportion of CSL boundaries first increased with magnetic field intensity, reaching a maximum at 4 T, but decreased when the magnetic field increased further. Combined with grain orientation distribution analysis (Fig. 5), these results indicate that the proportion of CSL boundaries in the solidification structure is closely related to texture formation.

3 Analysis and Discussion

When a crystal with magnetic crystalline anisotropy is placed in a static magnetic field, it tends to align its easy magnetization axis parallel to the magnetic field direction to minimize system energy, provided it can rotate freely. The magnetic torque T acting on a crystal in a uniform magnetic field can be expressed as:

$$T = \frac{\Delta\chi V B^2}{2\mu_0} \sin 2\theta$$

where Δ is the difference in magnetic susceptibility between two crystal directions, $_0$ is the vacuum permeability, B is the magnetic field intensity, V is the crystal volume, and is the angle between the magnetic field and the crystal direction with maximum magnetic susceptibility.

The grain refinement mechanism of Al-5Ti-1B refiner in aluminum alloys is as follows: When the refiner is added to the melt, suspended Al_3Ti or TiB_2 nuclei form in the melt, and subsequent α-Al heterogeneous nucleation and growth occur on these substrate phases. Al_3Ti has a body-centered tetragonal structure, while TiB_2 has a hexagonal structure. Both have potentially more significant magnetic anisotropy than fcc α-Al crystals due to their crystal symmetry. These substrate phases are suspended in the liquid phase and can rotate under magnetic field, thereby affecting solidification structure orientation. However, equation (1) indicates that magnetic torque is proportional to crystal volume. Literature reports that rod-like or plate-like $TiAl_3$ particles observed in refined grains have average diameters of 20-40 m, while TiB_2 particles are generally smaller than 2 m. In this study, the obtained α-Al grain diameters were all above 150 m (Fig. 3). Therefore, the magnetic torque acting on α-Al phases is much greater than that on substrate particles, and the final grain orientation should be determined by the magnetic anisotropy of α-Al.

Zhu measured the magnetic susceptibility of different crystal planes in single-crystal pure Al, demonstrating that the magnetic crystalline anisotropy of single-crystal Al is significant, with the (310) crystal plane exhibiting maximum magnetic susceptibility. According to equation (1), when a magnetic field is applied, Al grains will rotate to align their ⟨310⟩ crystal direction parallel to the magnetic field direction, with the orientation effect becoming more pronounced at higher magnetic field intensities (Fig. 8).

When the melt temperature is too high, Al-5Ti-1B refiner degrades. Studies have shown that when melt temperature exceeds 800 °C, the refining effect is lost within 10 minutes after addition. In this experiment, the sample was held for a certain period (30 minutes) before withdrawal to establish a stable temperature gradient. At higher temperature gradients (furnace temperatures of 850 and 950 °C), the refiner in the liquid phase was partially or completely ineffective, resulting in columnar grain structures without magnetic field. Columnar grains cannot rotate freely to align their ⟨310⟩ easy magnetization direction parallel to the magnetic field (Figs. 4a and c). After magnetic field application, columnar grains were broken by thermoelectric magnetic forces to obtain equiaxed grains, but the fine-grained structure exhibited random orientations due to small intergranular spacing and lack of free rotation space (Figs. 4b and d), consistent with literature reports. Additionally, as temperature gradient increased, the mushy zone length decreased. According to the Al-Cu phase diagram, the temperature interval between solidus and liquidus for Al-4.5Cu alloy is approximately 74 K. The mushy zone length L can be estimated by:

$$L = \frac{\Delta T}{G}$$

where ΔT is the temperature difference between solidus and liquidus for a given composition, and G is the temperature gradient. From equation (2), mushy zone lengths were 27, 11, and 7.3 mm at temperature gradients of 27, 65, and 101 K/cm, respectively. Thus, the mushy zone is longer at lower temperature gradients, providing more time for free grains to rotate and facilitating orientation. This explains why grain orientation was pronounced at lower temperature gradients (Fig. 5d) but random at higher temperature gradients (Figs. 6b and d) under the same magnetic field intensity (6 T).

Grain orientation behavior during solidification is also affected by melt flow, as flow-induced disturbances are detrimental to grain alignment. Inclined solid-liquid interfaces and freckle-like structures appear when flow exists in the mushy zone. Without magnetic field, density differences in the melt during directional solidification cause convection, leading to inclined solid-liquid interfaces (Fig. 1a). The influence of high static magnetic fields on flow during directional solidification involves two main aspects: magnetic damping effect and thermoelectric magnetic convection (TEMC) effect. The magnetic damping effect refers to the generation of Lorentz forces opposite to the flow direction when conductive fluid moves in a magnetic field, which suppresses flow. This effect can be characterized by the dimensionless Hartmann number (Ha), defined as the ratio of magnetic damping force to viscous force:

$$Ha = BL\sqrt{\frac{\sigma}{\rho\nu}}$$

where σ is the electrical conductivity of the metal melt, is the melt density, is the melt kinematic viscosity, and L is the characteristic length scale. The Ha number increases monotonically with magnetic field intensity B and is constrained by the length scale L, showing significant suppression of crucible-scale flows (>1 mm) but weaker suppression of micro-scale flows (<1 mm). According to equation (3), magnetic damping effect strengthens with increasing magnetic field intensity at constant sample dimensions, suppressing macro-scale flow in the melt, reducing freckle-like structures, and flattening the solid-liquid interface (Fig. 1c).

The TEMC effect arises because temperature gradients exist ahead of the solid-liquid interface during directional solidification, and the Seebeck coefficients of solid and liquid phases differ. According to thermoelectric principles, thermoelectric currents are generated ahead of the solid-liquid interface. When a magnetic field is applied, interaction between thermoelectric currents and the magnetic field produces Lorentz forces that drive flow, generating both crucible-scale and micro-scale convection. The TEMC flow velocity u_{TEMC} can be expressed as:

$$u_{TEMC} = \frac{\sigma S \Delta T B L}{\rho}$$

where S is the difference in Seebeck coefficients between solid and liquid phases. Equation (4) shows that TEMC intensity increases with magnetic field and is also affected by the length scale L. At crucible scales (>1 mm), magnetic damping and TEMC effects constrain each other. At weak magnetic field intensity (2 T), TEMC causes freckle-like structures in the solidification structure (Fig. 1b), resulting in random grain orientations. However, as magnetic field intensity increases (4 T), enhanced magnetic damping weakens TEMC, eliminating freckle-like structures and facilitating grain orientation (Fig. 1c). Under 6 T magnetic field, crucible-scale flow is suppressed by magnetic damping, but grain-scale TEMC (200-500 m) persists, causing freckle-like structures to reappear (Fig. 1d) and disturbing grain boundary formation, leading to decreased proportions of special boundaries. Nevertheless, magnetic torque also reaches its maximum at this field strength, causing orientation of numerous grains (Fig. 5d).

Grain boundary formation in solidification structures differs from that in recrystallization annealing. During recrystallization annealing, grain boundary changes occur primarily through migration in the solid phase. During solidification, growing grains contact each other, and the remaining liquid film between them transforms to solid, forming grain boundaries. According to the five degrees of freedom definition of grain boundaries, boundary properties are determined by the orientations of the two adjacent grains. As analyzed above, magnetic fields can cause suspended α-Al grains in the mushy zone to rotate and orient, thereby changing grain boundary characteristic distribution in the solidification structure. According to equation (1), larger differences in magnetic susceptibility between crystal directions result in stronger magnetic torque on grains. Literature indicates that crystal directions with larger magnetic susceptibility in α-Al are generally high-index directions, while low-index directions have relatively smaller magnetic susceptibility. CSL boundaries can be defined as one grain rotated relative to another around a specific crystal direction by a particular angle, with this direction generally being a low-index crystal axis. When a sufficiently large magnetic field is applied and flow disturbance in the melt is relatively small, the probability of crystal rotation around low-index crystal axes increases, leading to higher proportions of CSL boundaries in the solidification structure.

Conclusions

(1) During directional solidification of Al-4.5Cu alloy refined with Al-5Ti-1B under high magnetic fields, refined grains align along the easy magnetization direction $\langle 310 \rangle$ due to the magnetic crystalline anisotropy of α-Al. At a temperature gradient of 27 K/cm, the degree of orientation increases with magnetic field intensity.

(2) High temperature gradients narrow the mushy zone, reducing space for free

grain rotation and being unfavorable for grain orientation. The magnetic damping effect suppresses fluid flow in the melt, facilitating grain orientation, while thermoelectric magnetic convection increases disturbances and weakens the orientation effect of the magnetic field.

(3) The proportion of CSL boundaries in solidification structures is related to texture type. Applying magnetic fields can increase the proportion of CSL boundaries in refined solidification structures, with magnetic field-induced grain orientation being the cause of increased CSL boundary proportion. Additionally, the proportion of CSL boundaries is affected by fluid flow, with flow in the melt being detrimental to CSL boundary formation.

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Note: Figure translations are in progress. See original paper for figures.