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Effect of different solute additions on dendrite morphology and orientation selection in cast binary magnesium alloys

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ABSTRACT

In solidification, dendritic morphology was observed to change accordingly if either the type or quantity of the additional element was modified. To gain insight into this phenomenon, the 3D dendrite morphology of different binary magnesium alloys, including MgAl, MgBa, MgCa and MgZn alloys was characterized using synchrotron X-ray tomography and electron backscattered diffraction. Results showed that for most Mg-based alloys, the dendrite exhibited a typical 18-branch morphology with preferred growth orientations along ⟨11$\bar{2}$0⟩ and ⟨11$\bar{2}$3⟩, whereas for MgZn alloys, the dendrite morphology would change from the 18-branch pattern to 12-branch if the Zn content increased, i.e. the so-called dendrite orientation transition (DOT) took place. This DOT behaviour of the Mg alloy dendrite was then successfully modelled using the 3D phase field method by changing the magnitude of related parameters in the specially formulated anisotropy function based on spherical harmonics.

1. Introduction

The dendrite morphology, observed during a wide range of solidification processes, stems from the instability of the solid-liquid interface during phase transition. The dendritic microstructure, along with secondary phases and segregation of impurities, have a profound influence on the mechanical properties of the structural material [1–5].

Nucleation and growth of the dendrite have been studied extensively for several decades, and major theoretical and computational advances have significantly improved one's understanding on the fundamental mechanism that drives such microstructure, especially for alloys with a *fcc* lattice structure. In addition to the commonly observed ⟨100⟩ growth direction [2,6], ⟨110⟩ [7], ⟨320⟩ [8] and ⟨210⟩ [9] were also observed as the preferred growth orientations under specific circumstances. For binary AlZn alloys, a phenomenon termed dendrite orientation transition (DOT) could take place [9–12]. A typical example was that the preferred growth direction/orientation of the dendrite shifted continuously from ⟨100⟩ to ⟨110⟩ if the zinc content in the AlZn alloys was increased. Besides, 3D phase field method was also developed and employed to study the DOT behaviour [13,14] and the subsequent dendrite growth and morphology change under circumstances of equiaxed and directional solidification [15–17].

Different from the aluminum alloys (or alloys with *fcc* lattice structure), very limited studies have been performed on the dendritic pattern formation of the magnesium alloys, i.e. with the *hcp* lattice structure [18,19]. Pettersen et al. [20,21] studied the AZ91 magnesium alloy (primary additional elements were Al and Zn) under directional solidification, and suggested that the dendrite stem grew either along ⟨11$\bar{2}$0⟩ directions with six secondary arms or along ⟨22$\bar{4}$5⟩ directions with three secondary arms. Wang et al. [19] found that the Mg-40 wt% Zn alloy dendrite grew along ⟨11$\bar{2}$0⟩ directions with six secondary arms stretching out. However, according to our previous studies [22,23], the dendrites of both MgSn and MgGd alloys had eighteen primary branches with six growing along ⟨11$\bar{2}$0⟩ directions on the basal plane and twelve others growing along ⟨11$\bar{2}$3⟩ directions on {10$\bar{1}$0} planes. These studies have raised the question that what is the exact influence of the solute element, including type and quantity, on the dendrite morphology and growth pattern of the magnesium alloys.

In this paper, the 3D dendrite morphology of magnesium alloys with different additional elements was studied using synchrotron X-ray tomography, accompanied with electron backscattered diffraction (EBSD). In particular, attention has been focused on the influence of the basic lattice structure, i.e. additional elements with

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fcc, *bcc*, *bct* and *hcp* were selected so their influence on the according dendrite morphology could be investigated. Surprisingly, the so-called DOT was also found for the MgZn alloy. To investigate such growth behaviour, a 3D phase field model was then employed to simulate the dendrite growth. It was found that the DOT of the MgZn alloy was largely determined by the magnitude of certain parameters in the anisotropy function. Detailed analysis was also performed on the growth pattern of the secondary arms for dendrite growing along different orientations.

2. Experiments and methods

2.1. Materials

Magnesium alloys including Mg-25 wt% Al, Mg-10 wt% Ba, Mg-15 wt% Ca and six MgZn alloys with different compositions ranging from 20 wt% to 45 wt% were prepared from 99.95 wt% purity base metals. The alloys were cast in a metal mould and then machined into rods of 10 mm in diameter and 30 mm in height. The rods were then sealed in a quartz tube filled with argon. These samples (alloy rods sealed inside the quartz tube) were then heated up until the rods were remelted and then the whole sample was quenched in water. It was shown that through the quenching, the dendrite could exhibit well-developed side-branching patterns and thus good for the 3-D characterisation using X-ray tomography. Cylindrical specimens of 1.0 mm in diameter and 5 mm in height were then machined for the subsequent synchrotron X-ray tomography experiments, and cubic samples of size $5 \times 5 \times 5$ mm^3 were machined for EBSD experiments.

2.2. Electron backscattered diffraction (EBSD)

For the EBSD analysis, the specimens were firstly grinded with SiC paper and then electropolished using a stainless steel cathode with a DC voltage of 2.5 V and a current of 0.2–0.3 A. A solution of 35 ml of phosphoric acid and 65 ml of ethyl alcohol was used as the electrolytic solution, and the electropolishing time was approximately 3–4 min at room temperature. The EBSD experiments were performed on a TESCAN MIRA3 LMH scanning electron microscope with HKL Channel 5 system.

2.3. The synchrotron X-ray tomography

The synchrotron X-ray tomography experiments were performed at the BL13W1 beamline of the Shanghai Synchrotron Radiation Facility (SSRF). An X-ray with energy of 22–28 keV was used to penetrate the cylindrical specimen, and 900 image projections were collected using an exposure time of ~1 s. The distance between the specimen and camera was 20 cm. The reconstruction was performed in volumes of 2048^3 voxels with a voxel size of approximately 0.65 μm.

3. Experiment results

3.1. The α-Mg dendrite

Taking Mg-30 wt% Sn alloy for instance, a typical 2D image slice from the X-ray tomography experiment was shown in Fig. 1a. The dendrite morphology in the image slice was then extracted using image processing techniques including 3D median filter (see Fig. 1b), thresholding (Fig. 1c) and segmentation (see Fig. 1d), i.e. same as those applied in our previous work [20]. The reconstructed 3D morphology of the dendrite was shown in Fig. 1e–g.

Fig. 1f and g show that a typical α-Mg dendrite had eighteen primary branches, six of which (designated as A_1–A_6 in Fig. 1f) grew on the basal plane in a six-fold symmetry while the rest twelve (four of which were designated as B_1–B_4) also grew symmetrically but on different planes. According to EBSD analysis, the six preferred growth directions on the basal plane were along $\langle 11\bar{2}0\rangle$ while the other twelve were along $\langle 11\bar{2}3\rangle$.

The reconstructed 3D morphology of Mg-30 wt% Sn, Mg-25 wt% Al, Mg-10 wt% Ba and Mg-20 wt% Y alloys were shown in Fig. 2. To achieve a better illustration, dendrite morphologies viewed from $\langle 0001\rangle$ and $\langle 10\bar{1}0\rangle$ directions were shown in detail in the second and third column in Fig. 2, respectively. The lattice structure for element Sn is *bct*, while it is *fcc*, *bcc* and *hcp* for Al, Ba and Y, respectively. As shown in Fig. 2, the dendrites all exhibited a typical and similar 18-primary-branch morphology in 3D, and EBSD analysis also revealed that the two preferred growth directions were $\langle 11\bar{2}0\rangle$ and $\langle 11\bar{2}3\rangle$, which was the same as that reported in our previous work [22]. However, morphology difference still existed for dendrites of different alloys. The first difference was the angle magnitude i.e. θ between $\langle 11\bar{2}0\rangle$ and $\langle 11\bar{2}3\rangle$. Here, only the acute angle was considered, i.e. the magnitude was $180^\circ-\theta$ when θ was higher than 90°. Comparison of this angle was shown in detail in Table 1, as well as other key parameters of the additional elements investigated. It can be seen from Table 1 that, elements Gd and Y had the same lattice structure as Mg, i.e. *hcp* and the measured θ was about $54 \pm 3^\circ$ and $52 \pm 3^\circ$ respectively, which were slightly larger than those of other alloys.

The second difference was on the length of the two primary branches, i.e. $\langle 11\bar{2}0\rangle$ and $\langle 11\bar{2}3\rangle$. For MgGd and MgY alloys, according to the reconstructed morphology for multiple dendrites, the length of the $\langle 11\bar{2}3\rangle$ primary branch was shown to be always longer than that of the $\langle 11\bar{2}0\rangle$ ones, whereas for other alloys, the length of both $\langle 11\bar{2}0\rangle$ and $\langle 11\bar{2}3\rangle$ primary branches was nearly the same. Consequently, the envelop of the α-Mg(Gd/Y) dendrite always took a form of regular hexagonal prism, but for α-Mg(Sn/Al/Ba/Ca) dendrites it exhibited a truncated hexagonal bi-prism [26]. The length difference between the two primary branches partially indicated the difference of the growth velocity along $\langle 11\bar{2}0\rangle$ and $\langle 11\bar{2}3\rangle$ directions.

Another difference was on the detailed feature of the dendrite morphology. For the 18-branch morphology, the α-Mg dendrites for both MgGd and MgY alloys were nearly the same and very difficult to distinguish from one another. The α-Mg(Ca) dendrite was similar to that of MgSn alloy. The $\langle 11\bar{2}0\rangle$ branch for α-Mg(Ba) dendrite tended to split on the $\{10\bar{1}0\}$ plane (see Fig. 2i). These differences in morphology could be caused by the change or modification of surface energy during dendrite growth because of the difference in lattice structure, solid solubility and solute concentration.

As mentioned, based on EBSD analysis and under directional solidification, Pettersen et al. [20,21] found that the non-basal direction was $\langle 22\bar{4}5\rangle$, which slightly deviated from the $\langle 11\bar{2}3\rangle$ direction found in this study (the difference was only 4°–5° taken $c/a = 1.624$). However, it should be mentioned that the addition of solute element in the magnesium would distort the lattice structure and consequently alter the magnitude of the c/a ratio, but this effect was not considered in Pettersen et al. [20,21]. Besides, the accuracy of the EBSD analysis was highly dependent on how well the selected surface (i.e. 2D slice image) was aligned with the reference crystalline plane and the magnitude of the c/a ratio as an input value. The imperfect alignment of the target surface with the reference plane and the alternation of the c/a ratio due to addition of solute elements would cause inevitable error in identifying the growth directions of the dendrite. In terms of angle, this error could be at least several degrees, which possibly led to the same uncertainty on $\langle 22\bar{4}5\rangle$ and $\langle 11\bar{2}3\rangle$. This doesn't even account for the fact that error could also be induced during the measurement procedure, e.g. either using 2D slice images to render the 3D