Two- and three-dimensional studies of dendritic morphology in magnesium alloy by means of synchrotron X-ray microtomography and cellular automaton modelling

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Abstract. Magnesium is the lightest structural material. As one of the dominant microstructure features, dendritic pattern determines the mechanical behaviour and performance of magnesium alloys. Dendritic topological observation was carried out on Mg-based alloy using synchrotron X-ray micro-tomography and the microstructure pattern of \(\alpha\)-Mg dendrite was obtained. It was found that the \(\alpha\)-Mg dendrite grew with eighteen primary stems, of which six lay in the (0001) basal plane, and the other twelve in the (1\(\overline{1}\)0\(\overline{1}\)) plane. An according numerical model based on the cellular automata method was developed. By defining a specific capturing functional mechanism, simulation of \(\alpha\)-Mg dendrite in 3-D with eighteen branches was successfully achieved. The simulation results show that the model could reasonably describe the evolution of the dendritic microstructure and the subsequent dendrite morphology agrees well with that observed in the synchrotron X-ray tomography experiment.

1. Introduction

With demands for light-weighted, decreasing of fuel consumption and CO\(_2\) emission, conserving finite oil reserves, magnesium alloys have achieved increasing applications in automobile industry in recent years, such as steering wheels, gear boxes, instrument panels and air intake systems, etc. [1-3]. The mechanical properties of magnesium alloy castings are greatly affected by the microstructure of magnesium alloys, especially the dendritic structure. It’s of great importance to understand the dendritic morphology of magnesium alloys.

A great number of studies on the dendritic morphology of face-centered cubic (FCC) and body-centered cubic (BCC) alloy system have been carried out in two and three dimensions [4, 5]. It is widely accepted that the preferred growth orientation is typically along \(<100>\) in these alloys. The two-dimensional morphology of aluminum alloys exhibits four-fold symmetry, and the three-dimensional dendrite has six branches. For the hexagonal close-packed (HCP) alloy system, different growth orientations have been reported, such as \(<1\overline{1}0\overline{1}>\) [6, 7] or \(<0001>\) [6] for zinc, and \(<1\overline{1}0>\) for H\(_2\)O [4].

For magnesium alloys (HCP alloy system), very limited studies have been performed to investigate the 3-D morphology of the dendrite. Pettersen et al [8, 9] studied the AZ91 magnesium alloy under directional solidification using SEM and EBSD and found that dendrites have two different stem...
directions i.e. (2245) and (1120). Wang et al [10] obtained the three-dimensional morphology of directionally solidified Mg-9wt.%Al alloy by synchrotron X-ray tomography, and suggested that α-Mg(Al) grew along (1120) in the (0001) basal plane. The dendrites have plate-like morphology and exhibit six-fold symmetry in the (0001) basal plane. Wang et al [11] studied the three-dimensional equiaxed dendritic morphology of Mg-Zn alloys, which was similar to the (1120)-type stems in reference [8].

Numerical modeling and simulation is widely used to predict the microstructure evolution during solidification of magnesium alloy [12]. Böttger et al [13, 14] integrated a hexagonal anisotropy function into the phase-field model (PF), and simulated the three-dimensional dendritic growth of magnesium, which had six prism orientations in the (0001) basal plane and two basal orientations in the (0001) directions. Wu et al [15] simulated the dendrites growth of AM50 alloy by cellular automaton model (CA) and the results were consistent with [13, 14]. Eiken [16] investigated the growth texture evolution of magnesium by PF model. They found that grains grew faster in (1120) orientation and retarded in (0001) orientation. The grain arrangement is plate-like within the basal plane.

As seen, for the magnesium alloys the (1120)-type stems in the basal plane have been accepted as one type of the primary growth directions. However, the preferred growth directions in the non-basal plane have not been clearly determined, i.e. discrepancy exists between the experiment observed (2245) direction and the simulated (0001) direction.

In this paper, experiment methods including SEM, synchrotron X-ray micro-tomography, combining with a cellular automaton model were applied to study the 2-D and 3-D microstructure of Mg-30wt.%Gd alloy. The purpose was to shed some lights on determining and thus understanding the full structure of the magnesium alloy dendrites in 3-D.

2. Experimental methods

2.1. Materials
An Mg-30wt.%Gd alloy was selected because of the better absorption contrast. The eutectic temperature and liquidus temperatures of the alloy are 542°C and 587.2 °C, respectively. The alloy was prepared as follows. Firstly, magnesium (99.95 wt. %) and gadolinium (99.99 wt. %) were placed in a mild steel crucible and heated by an electric resistance furnace. The mixture in the crucible was melted and mixed at 800 °C under a protective gas of 99.7% N2 and 0.3% SF6. The melt was then poured into a permanent mould which was preheated to 300 °C and held for 30 min [17, 18]. A cylindrical specimen of 10 mm in diameter and 30 mm in length was machined from this object. Secondly, the specimen was sealed in a quartz tube with argon as protective atmosphere, then it was remelted and quenched between the liquidus and eutectic temperatures. Cylindrical specimens of 1.0 mm in diameter and 5 mm in length were machined from the specimen for tomography experiments.

2.2. Synchrotron X-ray micro-tomography
Tomography experiments were carried out at the BL13W1 beam line of the Shanghai Synchrotron Radiation Facility (SSRF). A high-speed CCD camera was used to record the transmitted intensity of a nearly monochromatic X-ray beam when the sample was continuously rotated over 180° [19]. An X-ray energy of ~21.5 keV was used to penetrate the specimens, and 900 projections were taken between 0° and 180° using an exposure time of 4s per projection. Dark field images without X-rays, and flat field images with X-rays but without the sample were also recorded for further image-processing [19, 20]. The distance between the specimen and the camera was 20 cm. The reconstructions resulted in volumes of 2048³ voxel with a voxel size of (0.74 μm)³.
2.3. Image processing

A software package PITRE [21] was used to convert the images from raw data to reconstructed tomographic slices and from 32 bit to 8 bit. The dendritic microstructure was segmented from sub-volumes of about 400×400×400 voxels (296μm³) after filtering and thresholding (figure 1).

![Figure 1](image)

**Figure 1.** Key steps of data processing including (a) cropping (b) 3D median filter (c) thresholding, and (d) segmentation.

3. Results and discussion

3.1. The 2-D microstructure

Based on the two-dimensional characterization, some interesting dendritic microstructure can be observed. Figure 2 shows the optical micrographs (OM) and simulated dendrites of magnesium alloy. The model was based on our previous work [12]. By using a special neighborhood configuration with the square CA grid, and the capturing rules proposed by BELTRAN-SANCHEZ and STEFANESCU, modeling of dendritic growth was achieved [12]. As observed in figures 2a-c designated by “1”, the primary dendrite exhibits a typical six-fold symmetric shape with six branches growing out from the nuclei. Figure 2d indicates the simulated equiaxed dendrites under cooling rate of 80 K/s which is roughly equal to that in the quenched condition. According to figure 2d, the simulated dendrites exhibit six-fold symmetric shape as designated by “1” (figure 2a-c). It is noted that not all the dendrites of magnesium alloy are fully simulated in figure 2d. With the six-fold symmetric morphology, six separated particles with five branches (designated by “2”) can be found in figure 2a and 2c. Instead of six-fold symmetric structure, most dendrites develop a low symmetrical or even disordered morphology as four-branch dendrites designated by “4”, tortoise shell-like dendrites designated by “5” and five-branch dendrites of separated particles designated by “3”.

3.2. Three-dimensional microstructure

3.2.1. Synchrotron tomography. Figure 3a shows the 3D morphology of one of the reconstructed magnesium dendrites from synchrotron X-ray micro-tomography. Some planes and directions are used to cut the dendrite to help understanding the dendritic microstructure. Figure 3b indicates the morphology on S₀, which has six branches i.e. d₁~d₆ and the angles between the dᵢ-type directions are 60°. For the two-dimensional projections on S₁ (figure 3c), which contain the three planes across g₁,
and \( g_4 \), \( g_2 \) and \( g_5 \), or \( g_3 \) and \( g_6 \), the dendritic microstructure have two branches on \( S_0 \) and other four branches next to them (in figure 3d). It is noted that the \( S_1 \)-type planes are perpendicular to plane \( S_0 \) and magnesium dendrites have eighteen branches. To achieve a better understanding of the dendritic morphology, two other planes i.e. \( S_2 \) and \( S_3 \) (figure 3e and 3g) are added in the dendrite. For the projections on \( S_2 \) and \( S_3 \), the dendrites have six and four branches respectively, as observed in figure 3f and 3h.

**Figure 2.** Optical micrographs (a-c) and simulated results (d) of dendritic morphology of magnesium alloy.

**Figure 3.** Three-dimensional dendritic microstructure of magnesium alloy (a) and cross profiles with different sections: (a-b) section \( S_0 \); (c-d) section \( S_1 \); (e-f) section \( S_2 \); (g-h) section \( S_3 \).
As demonstrated in figure 3, the dendrites exhibit six branches on planes S₀, S₁, and S₂. Only the dendrites on S₀ are symmetric in six-fold, and the plane is designated as the basal plane. It is equivalent to \{0001\} crystallographic plane. The \(d_{\alpha}\)-type direction is equivalent to \(\langle 11\bar{2}0 \rangle\) crystallographic orientation. The dendrites on S₁ or S₂ are not symmetric in six-fold, and the angles between the branched on the basal plane and the non-basal plane are also different from each other. Based on orientation measurements of thirty dendrites, the angle between \(g_3\) and \(d_3\), namely \(\theta\), is determined to be \(54°±3°\). Then the \(g_{\gamma}\)-type direction is equivalent to \(\langle 1\bar{1}\bar{2}3 \rangle\) crystallographic orientation, which is close to the reported \(\langle 22\bar{4}5 \rangle\) direction in literature [8]. The dendrites on S₃, which have four branches, are consistent with the “4” dendrite in figure 2b. In this way, the 3D dendritic morphology of magnesium alloy grows along \(\langle 1\bar{1}\bar{2}0 \rangle\) and \(\langle 1\bar{1}\bar{2}3 \rangle\) orientations, and these dendritic morphologies and patterns in figure 2 can be explained and understood.

3.2.2. Three-dimensional simulated results. Based on the reconstructed three-dimensional dendritic morphology of magnesium alloy, a model describing eighteen-branch growth of magnesium alloy is constructed. As indicated in figure 4, it has six prime orientations along \(\langle 1\bar{1}\bar{2}0 \rangle\) in the basal plane and twelve orientation along \(\langle 1\bar{1}\bar{2}3 \rangle\) in the non-basal plane. A corresponding neighbourhood configuration was defined based on this schematic growth model and our previous work [15].

![Figure 4. Schematic diagram of three-dimensional growth model of magnesium alloy.](image)

Table 1. Parameters and physical properties of Mg-30wt.%Gd alloy used in the present simulation [12, 15]

| Property                                    | Mg-30wt.%Gd |
|---------------------------------------------|-------------|
| Initial concentration of Gd (wt. pct)       | 30.0        |
| Solute partition coefficient                | 0.6         |
| Liquidus slope (K/wt pct)                   | -2.1        |
| Gibbs-Thamson coefficient (m·K)             | \(6.2×10^{-7}\) |
| Liquidus temperature (K)                    | \(5.87×10^{2}\) |
| Solidus temperature (K)                     | \(5.42×10^{2}\) |
| Solute diffusion coefficient in liquid (m²/s)| \(1.8×10^{9}\) |
| Solute diffusion coefficient in solid (m²/s)| \(1.0×10^{12}\) |

The dendritic growth of Mg-30wt.%Gd alloy was simulated to verify the established three-dimensional Cellular Automaton model. A single nucleus was planted at the center of a calculation
domain, i.e. 200×200×200 grids with a grid size of 2 µm. The temperature field was uniform with a cooling rate of 80 K/s. The parameters and physical properties of Mg-30wt.%Gd alloy used in this simulation are listed in Table 1 [12, 15]. Figure 5a and 5b demonstrate the simulated three-dimensional dendritic morphology and solute map of Mg-30wt.%Gd alloy. It can be observed that there is a strong dominance of eighteen orientation in the simulated 3D dendrite. For the xy-plane shown in figure 5c, the primary dendrite arms grow along six prism orientation with the angle of 60° between arms, the same as the six-fold symmetric morphology cut by S0 indicated in figure 3b. For the xz-plane (figure 5d), the dendrite arms grow along six arms i.e. two arms in the xy-plane and four arms next to them as the six branches morphology cut by S1 (figure 3d). Compared with the reconstructed three-dimensional dendrite of magnesium alloy in figure 3, the simulated results with eighteen arms dendrite agree well with the experimental results.

The effect of the cooling rates on the three-dimensional dendritic morphology of magnesium alloy were also studied. A single nucleus was planted at the center of a calculation domain i.e. 200×200×200 grids with a grid size of 1 µm, three different cooling rates of 30, 50 and 80 K/s. Figure 6 shows the simulated equiaxed dendritic growth of Mg-30wt.%Gd alloy under different cooling rate. It can be observed that with the increase of the cooling rate, the dendrite grew into a more well-developed morphology and the branches of dendrite arms became enhanced. For the primary arms on the non-basal plane, only a few secondary arms branch under the cooling rate of 30 K/s (figure 6d), while secondary arms develop sufficiently under the cooling rate of 80 K/s (figure 6f). An increasing cooling rate results in a higher growth velocity, and then lead to a higher accumulation of the solute at the solidification front (figure 6d-6f). Hence, the primary dendrite arms grow faster with an increasing cooling rate [15].

Figure 5. Simulated dendritic growth of Mg-30wt.%Gd with eighteen-branch structure.
4. Conclusions
(1) The 2-D and 3-D dendritic microstructure of magnesium alloy was studied using SEM and the synchrotron X-ray micro-tomography. It was found that the α-Mg dendrite has eighteen branches including six growing along the $\langle 1\bar{1}20 \rangle$ direction in the basal plane and the other twelve growing along the $\langle 1\bar{1}23 \rangle$ in the $\langle 10\bar{1}0 \rangle$ plane.
(2) Based on the observed dendritic morphology, a three-dimensional cellular automaton model was developed to simulate the dendritic growth of magnesium alloy. Simulated dendrites grow into eighteen branches which are agree with the experimental results.

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