

# Evolution of microstructural length scales during solidification of magnesium alloys

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## Abstract

The microstructure of magnesium alloys usually presents a sixfold symmetry that is not amenable to the usual characterization of fourfold symmetric alloys. A more systematic and quantitative description can be achieved through Fourier analysis of the spatial distribution of the microstructure. Through this analysis, the microstructures obtained from phase-field simulations reveal self-affine regions with different scaling dimensions and crossover regions between them. The time evolution of these spatial scales and the analysis of local microstructural events reveal that isolated liquid pools are created through the merging of dendrite branches that follow the universal dynamics of merging predicted by Aagesen et al., and proceed to contract following the dynamics dictated by late stage cooling and back diffusion. Our results are consistent with new experiments that reveal a correlation between the dependence on cooling rate of the length scales associated with the spacing of main branches, and the distribution and average size of constituent phases that form during late stages of solidification.

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## 1. Introduction

Magnesium alloys, with their relatively high specific strength and stiffness, provide significant opportunities within the transportation industry to reduce mass, and thus improve fuel efficiency and reduce CO<sub>2</sub> emissions. Developing magnesium alloys with acceptable strength, formability and weldability requires establishing strong correlations between processing and final properties. Appropriate understanding of the morphology and length scales established during solidification is thus essential, especially in emerging technologies such as strip casting, where few thermo-mechanical post-processes are applied and thus the microstructure established at the time of solidification strongly influences the mechanical properties of the final product.

Magnesium alloys commonly form an underlying hexagonal close-packed (hcp) crystallographic lattice, which in the basal plane translates into a microstructure with sixfold symmetry. As a consequence, the resulting solidification structures cannot always be properly characterized by the notions of primary and secondary spacing that are commonly applied to microstructures with cubic symmetry. To date, there have been relatively few theoretical studies of solidification that are focused on microstructure selection in alloys with sixfold symmetry, and only a few numerical and experimental attempts to validate them [1–3].

In order to characterize the length scales inherent in complex microstructures, it is convenient to perform a Fourier analysis of their spatial distribution. For that purpose, an indicator-like function distinguishing between the solid and liquid phases is required. Such function can be obtained by image analysis of the microstructure formed during solidification or, in the case of phase-field simulations, from the phase-field variable. This characteristic function will include

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both regular and randomly fluctuating characteristics that can be extracted through multi-point correlation functions, the spatially averaged multi-point product of the characteristic function. The most physically relevant information can be extracted solely from the single point and autocorrelation functions. For perfectly self-similar objects, namely fractals, the power spectrum vs. wave vector magnitude will follow a power law whose exponent is the scaling index. In practice, complex morphologies may present several such power-law regions, with different scaling indices corresponding to different features or phenomena. Analyzing complex morphologies through Fourier analysis of the spatial distribution of the microstructure and its radial average is not new [4–6], and has been used mostly in image analysis. Here, we take a step further and analyze the power spectrum for insights into the morphologies that evolve during solidification. Our results on the formation and evolution of the liquid pools formed during solidification can provide valuable insight into the formation of secondary phases.

## 2. Methods

### 2.1. Phase field model

In this paper we model two-dimensional directional solidification of Mg–0.5 wt.% Al employing a modified version of the phase-field model described in detail in Refs. [7,8]. We replace the surface energy anisotropy function with the projection in the basal plane of the spherical harmonics representing the space group of the hcp crystal lattice [9,1,10]  $a(\hat{n}) \equiv a(\theta) = 1.008 + 0.002\cos(6(\theta - \theta_0))$ , where  $\theta$  is the angle between the normal to the interface and an underlying crystalline axis in the  $\langle 11\bar{2}0 \rangle$  direction (in the basal plane) and  $\theta_0$  is the angle between the direction of heat extraction and the crystalline axis. In this work we apply the maximum misorientation for hexagonal dendrites,  $\theta_0 = \pi/6$ . The anisotropy function  $a(\theta)$  makes the interface stiffness dependent on the orientation through the anisotropy strength and the misorientation angle from the crystal axis, imposing minimum stiffness at the main crystal axes directions. The material parameters employed [11] are presented in Table 1.

Our model includes thermal noise to promote side branching. Details on the inclusion of thermal noise in the model can be found in Refs. [12,13]. Our model also includes non-vanishing diffusivity in the solid, which, although significantly smaller than that of the liquid phase, is responsible for coarsening. The model is implemented on

the adaptive mesh refinement (AMR) algorithm developed by Provatas and co-workers [14,15,8].

### 2.2. Experimental procedure

As-received Mg–0.5 wt.% Al was melted inside an electrical resistance furnace, (Lindberg/Blue M) at 730 °C and poured into a cylindrical stainless steel crucible, as described in detail in Ref. [9,12]. As the coolant, water is splashed from below, promoting transient upward directional solidification. The crucible and surrounding insulation were designed such that the heat flow is unidirectional in a large region at the center of the cylindrical crucible, meaning that the temperature varies only in the longitudinal direction and is practically uniform in the transverse direction. The entire procedure was carried out in a protective environment of  $\text{CO}_2\text{SF}_6 - 0.5\%$  gas. To measure the temperature at different distances from the chill wall, K-type nickel–chromium-base sheathed thermocouples were aligned longitudinally at the center of the crucible 1 mm apart from each other, starting from 1 mm above the chilling surface. The interface velocity and temperature gradient were calculated from the time evolution of the temperature readings registered by the thermocouples. Depending on the coolant type, the pressure of the fluid and the front's relative position from the chill wall, the growth velocity range attained was 0.43–3.7  $\text{mm s}^{-1}$ , while the temperature gradient ranged from 0.6 to 4.45  $\text{K mm}^{-1}$ . The samples were cut and polished down to 0.05  $\mu\text{m}$  and etched in a solution of 20 ml of water, 20 ml of acetic acid, 60 ml of ethylene glycol and 1 ml of  $\text{HNO}_3$  [2] for about 5 min. The microstructure was then studied under a light microscope.

### 2.3. Microstructure analysis

It is convenient to consider  $p(\mathbf{r}) = (1 - \phi(\mathbf{r}))/2$  as a characteristic function, since the phase field ( $\phi$ ) varies between  $-1$  (liquid) and  $1$  (solid). Setting the threshold of  $p(\mathbf{r})$  to the color map range of an image creates an equivalent characteristic function to analyze our experiments. Our data are analyzed using the single point and autocorrelation functions defined by:

$$C^{(1)} = \frac{1}{A} \int p(\mathbf{r}) d^2r \quad (1)$$

$$C^{(2)}(\mathbf{r}) = \frac{1}{A} \int p(\mathbf{r}') p(\mathbf{r}' + \mathbf{r}) d^2r' \quad (2)$$

where the spatial average is over the domain of area  $A$ . The single-point correlation function  $C^{(1)}$  is just the solid fraction. The autocorrelation function  $C^{(2)}$  is related to the probability of two randomly chosen points in the domain of  $p(\mathbf{r})$  to lie within the same phase. Moreover, the Fourier transform of the autocorrelation function is equal to the power spectrum of the characteristic function:

$$C_F^{(2)}(\mathbf{k}) = \frac{1}{A} \int C^{(2)}(\mathbf{r}) e^{-i\mathbf{k}\cdot\mathbf{r}} d^2r = |\sigma(\mathbf{k})|^2 \quad (3)$$

Table 1

Material parameters defining the MgAl system [11].  $m$  is the liquidus slope,  $c_0$  the alloy composition,  $\kappa$  the partition coefficient,  $D$  the solute diffusivity in the liquid,  $\varepsilon$  the ratio of solid to liquid solute diffusivity and  $\Gamma$  the Gibbs–Thomson coefficient.

$ m $ (K/wt.%)	$c_0$ (wt.%)	$\kappa$	$D$ ( $\mu\text{m}^2/\text{s}$ )	$\varepsilon$	$\Gamma$ (K $\mu\text{m}$ )
5.5	0.5	0.4	1800	$10^{-5}$	0.62

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