



Shear mechanisms at 0–50% solid during equiaxed dendritic solidification of an AZ91 magnesium alloy

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Abstract

The rheology of equiaxed solidifying alloys containing 0–50% solid has been studied by vane rheometry. We focus on the incipient motion and subsequent flow mechanisms of quiescent solidifying microstructures. It is demonstrated that, once the growing crystals have impinged, the rheology can be interpreted within the framework of saturated compacted granular materials with little intercrystal cohesion. The material expands in response to shear and deforms primarily by Reynolds' dilatancy-enabled crystal rearrangement. Dilatancy leads to strain localisation in dilatant shear bands shortly after crystal impingement and is proposed to cause the initiation of cracks in the expanding regions between crystals at higher solid fraction.

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1. Introduction

Solidifying alloys are deformed in most casting processes due to natural sources such as convection, thermal contraction and solidification shrinkage. Moreover, many modern casting technologies intentionally deform the alloy throughout solidification in order to reduce processing steps and increase productivity. Examples include the continuous casting of steel, twin-roll casting of Al sheet and high-pressure die casting, in each of which solidification occurs concurrently with deformation. In addition to playing a key role in filling, feeding and deformation in such processes, mushy-zone rheology also directly leads to defects including deformation segregates in twin-roll cast sheet [1], segregation bands in centrifugal castings [2], and bands of porosity and macrosegregation in high-pressure die castings [3]. Past research has shown that similar defects can form when partially solid equiaxed alloys are deformed in rheology experiments [4–8]. This paper inves-

tigates the shear mechanisms operating when quiescent equiaxed microstructures containing 0–50% solid are sheared, and explores how deformation leads to casting defects.

During equiaxed solidification, it is common to define a crystal envelope as the surface connecting the extremities of the crystal [9–11]. In the case of dendritic growth, the envelope connects both the primary and secondary arm tips and can contain a significant liquid fraction [9]. In contrast, the envelope of a globular crystal is the solid–liquid interface and can contain no (entrapped) liquid [11]. It is therefore useful to split the total solid fraction, f_s , into the fraction of envelopes, f_g , and the solid fraction within envelopes, f_i , according to Eq. (1) [9,10]:

$$f_s = f_g \cdot f_i \quad (1)$$

Equiaxed growth causes crystal envelopes to impinge on one another at a solid fraction usually termed the dendrite coherency point, f_s^{Coh} [12], and, as crystal envelopes can be partially solid, f_s^{Coh} is strongly dependent on f_i . f_s^{Coh} has been experimentally found to vary from $f_s^{\text{Coh}} \approx 0.1$ for large, highly branched dendrites [12] to $f_s^{\text{Coh}} \approx 0.5$ for small, globular crystals [12,13].

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Studies on the deformation of quiescent Al and Sn alloys during equiaxed solidification have begun to develop a framework for low f_s equiaxed alloy rheology [4–7,14–17]. It is now well established that alloys possess no mechanical strength before crystal envelopes have impinged (when $f_s < f_s^{\text{Coh}}$) [4–6,16] and that f_s^{Coh} marks the onset of resistance to shear [5,6,16] and compressive forces [5]. The deformation mechanisms operating at $f_s > f_s^{\text{Coh}}$ have been partially deduced from examination of post-deformation microstructures, where it has been found that the dominant deformation mechanisms depend on the solid fraction relative to f_s^{Coh} [4,6,7,14,15]. At solid fractions shortly after f_s^{Coh} , there is rarely evidence of deformation of the solid and it has been proposed that the material changes shape by lubricated grain rearrangement by mechanisms including rolling and sliding [4–6,14]. Additionally, grain rearrangement is often localized at the shear plane [4–6,15] and there is usually evidence of flow of solute-enriched liquid to the shear plane [4,6,7,15].

As deformation occurs at solid fractions gradually higher than f_s^{Coh} , some deformation of the solid becomes apparent. This is most obvious for coarse dendritic microstructures where dendrite bending, dendrite fragmentation and the opening up of fissures between dendrites is reported [4]. When samples are sheared at higher solid fractions, a second transition in behavior occurs when open cracks develop in response to shear [4,6,15]. The solid fraction at the onset of shear cracking increases as the morphology becomes more globular and the severity of cracking increases with increasing shear rate and f_s above the cracking transition [4,6,15].

Recent experimental research has demonstrated that, when solidifying alloys are sheared shortly after f_s^{Coh} , the material behaves as a saturated compacted granular material and exhibits Reynolds' dilatancy [8]. It was shown that crystal rearrangement within a crystal network causes the packing density of the solid to decrease and the assembly of solid to expand. It was also shown that the granular behavior of partially solid alloys causes strain to localize into dilatant shear bands 7–18 mean grains thick, similar to those that form in saturated granular materials such as soils [8]. The present paper has three aims: (i) to present further evidence for dilatant granular behavior in solidifying alloys containing a solid network; (ii) to explore how this behavior fits into the general rheological framework for low f_s equiaxed alloys developed in previous studies [4,6,7,16,17]; and (iii) to investigate how the dilatant granular behavior leads to rheology-related casting defects.

2. Experimental methods

Magnesium alloy AZ91, with composition given in Table 1, was used throughout the research. This alloy solidifies in an equiaxed mode over a wide range of solidification conditions and, in the range 0–50% solid, consists of a mixture of liquid and primary solid, L + (Mg). Two techniques were used to characterize the rheology: (i) the continuous-

Table 1

Composition of AZ91 as determined by ICP-AES (wt.%)

Mg	Al	Zn	Mn	Si	Fe	Ni	Cu
Bal.	8.97	0.74	0.15	0.01	0.002	0.003	0.002

torque dendrite coherency method [12] was used to determine the point of crystal impingement; and (ii) vane rheometry [18,19] was used to measure the rheological response of microstructures containing 0–50% solid.

In both sets of experiments, samples were prepared by casting 90 g of AZ91 into boron nitride (BN) coated mild steel cylindrical cups and allowing them to solidify. The cups had dimensions height = 55 mm, ID = 40 mm and wall thickness = 2 mm, and contained four ribs to prevent outer wall-slip. The cups were later fastened into a test rig which incorporates a Physica DSR150 rheometer and the alloy was completely remelted using a furnace on the test rig. A mixture of CO₂ and tetrafluoroethane was used as a cover gas to prevent excessive oxidation of the melt. Once the alloy was liquid, a BN-coated 304 stainless steel four-bladed vane and a K-type thermocouple (calibrated against 99.98 wt.% Al) were lowered into the melt and the system was held at 700 °C (~100 °C superheat) for 20 min. At this point the experimental set-up appeared as shown in Fig. 1a and b. All experiments were started by removing the furnace and allowing the sample to cool naturally in air. The cooling rate in the liquid prior to nucleation of the first solid was ~1 °C s⁻¹ and, at 10–50% solid during solidification, the cooling rate varied from 0.30 to 0.45 °C s⁻¹. During both sets of experiments, temperature, torque and rotation angle were recorded at 20 datapoints s⁻¹.

Dendrite coherency experiments employed a similar method to the rheological technique developed by Arnberg et al. [12]. The blade was rotated at 0.05 rpm from 650 °C (fully liquid) and rotation continued as the alloy solidified. This low rotation rate was selected to continuously measure the resistance to flow during solidification without disturbing the developing microstructure.

In vane rheometry experiments, vane rotation was only initiated once the alloy had reached a desired temperature within the freezing range. The vane was then rotated at 5 rpm (0.52 rad s⁻¹) for one vane rotation and was then stopped. At the end of deformation, samples were either quenched in an ice + water slurry or allowed to continue solidifying naturally. In quenched samples, the peak cooling rate in the early stages of quenching was ~9 °C s⁻¹, approximately 20 times the cooling rate just prior to the quench. Fig. 1c summarizes the sequence of events in vane rheometry experiments. Note that deformation was carried out during continuous cooling (Fig. 1c) to ensure that the crystal morphology and intercrystal cohesion were representative of natural solidification and therefore deformation was not isothermal. The experiment temperature is defined here as the temperature at peak torque (M^{peak}) as shown in the insert in Fig. 1c. The peak shear stress was then calculated from the peak torque by assuming that

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