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## Pore behaviour during semi-solid alloy compression: Insights into defect creation under pressure

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The pre-existing porosity behaviour of globular semi-solid Al–Cu alloys during uniaxial compression is quantified during time-resolved synchrotron tomography at  $\sim 64\%$  and  $\sim 93\%$  solid. At  $\sim 64\%$  solid, both tortuous and round pores exist before deformation, respectively persisting and closing during compression, while at  $\sim 93\%$  solid, pre-existing tortuous pores open under compressive axial strain. It is demonstrated that pore behaviour reflects the immediate grain neighbourhood where the semi-solid alloy acts as a granular material, and that pore opening results from shear-induced dilation during uniaxial compression. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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A variety of casting processes apply pressure during solidification to control porosity. Examples include the intensification stage of high-pressure die casting (HPDC) [1-3] and squeeze casting [4,5], and semi-solid processing routes such as rheocasting and thixoforging [6-8]. While pressure can be very effective at reducing casting porosity (e.g. [4,9,10]), excessive pressure can cause casting defects such as cracks, localized porosity [11] and increased macrosegregation [12]. Previous rheology experiments on semi-solid Al and Mg alloys have shown that compressive and shear strains may lead to defects in the laboratory [13-15] that are analogous to those in industrial castings [13,16]. For example, dilatant shear banding and shear cracking develop in laboratory rheology experiments similar to defects in HPDC and semi-solid processing [16]. It has been suggested that defect formation under pressure is related to equiaxed semi-solid alloys behaving as a cohesionless granular material [13,16] whereby they undergo shear-induced dilation during deformation [16,17], i.e. the assembly of grains experiences an increase in volume as the discrete and independent grains push each other apart under compressive load, in ways reminiscent of particulate soils, slurries and rock [18–20]. In support of this, in situ radiography studies have directly measured grains pushing each other apart under compressive loading in semi-solid Al–Cu [21] and Fe–C alloys [17] and shear-induced dilation has recently been confirmed with time-resolved tomography [22]. However, many aspects of defect formation in pressurized castings remain poorly understood and, in particular, it is not clear how excessive pressure can increase local porosity.

The aim of this work is to image and quantify the evolution of pre-existing porosity during compression at different solid fractions in globular semi-solid alloys and to identify the conditions and mechanisms by which compressive strains can lead to porous defects.

In recent years, synchrotron tomography techniques have been developed to study semi-solid deformation in situ [23]. In this work, a bespoke tension–compression rig (P2R) fitted with a furnace [24,25] was used to conduct fast tomography at the JEEP (I12) beamline of the Diamond Light Source using a 53 keV beam. Both Al–15Cu and Al–8Cu (wt.%) alloys were used to provide two solid fractions at similar temperatures. A semi-solid microstructure was first engineered to optimize the spatiotemporal resolution of the experiment via a prior separate long-term heat treatment of approximately 5 °C

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above the eutectic temperature for 200 h, which coarsened the as-cast dendritic microstructure to large-scale globules. These globular microstructures were then partially remelted and deformed on the beamline. Each 5 mm diameter  $\times$  5 mm specimen was heated approximately 20 °C above the eutectic temperature, held for 5 min and then isothermally compressed while being rotated at 15°s<sup>-1</sup>. The Al-15Cu, 64% solid specimen was deformed at 5  $\mu$ m s<sup>-1</sup>. The Al–8Cu, 93% solid specimen was deformed at a lower strain rate of 1  $\mu$ m s<sup>-1</sup>, because damage formation in the 93% solid specimen is more rapid, with severe cracking. X-ray radiographs were acquired every 0.5° with an exposure time of 32 ms on a Phantom v. 7.3 camera (Vision Research) with 12.22 µm resolution. 3-D volumes were reconstructed using the filtered back-projection method [26,27].

Prior to deformation, the semi-solid specimens contained 64.3 vol.% (Al-15Cu) and 92.7 vol.% (Al-8Cu)  $\alpha$ -Al grains, respectively, as determined by image analysis. This is in good agreement with the calculated equilibrium volume fractions at the experimental temperatures of  $580.6 \pm 2.3$  and  $568.2 \pm 1.7$  °C, respectively, which correspond to  $62.2\pm1.3$ and  $94.5 \pm 0.03\%$  (estimated with the lever rule and a linear approximation to the liquidus, with the liquid and solid densities from ref [28]). Both specimens exhibit  $\alpha$ -Al globular grains with an average equivalent sphere diameter of  $401 \pm 12 \,\mu\text{m}$  at 64% solid and  $365 \pm 12 \,\mu\text{m}$  at 93% solid. All image processing used Avizo 7.0 (VisageImaging GmbH, Berlin, Germany) and MATLAB 7.1 (The Mathworks Inc., Natick, MA, 2000). All grains as well as pores over 10 pixels in volume ( $\sim 100 \ \mu m^3$ ) were thresholded using Otsu's method [29], tracked, and their shape and volume quantified during deformation.

The evolution of internal porosity during compression is shown qualitatively and quantitatively in Figure 1 for both 64 and 93% solid. At 64% solid, it can be seen in 2-D and 3-D (Fig. 1a,b) that pre-existing pores close during uniaxial compression, which is the expected behaviour. This is quantified in Figure 1c, where the volume of the pores decreases by an order of magnitude, from 0.064% before deformation to 0.007% at a true



Fig. 1. Internal porosity evolution during deformation. Transverse (xz) slices show the beginning, middle and end of the compression at (a) 64% and (d) 94% solid; 3-D rendering of the porosity for the same strains at (b) 64% and (e) 93% solid; change in volume of internal voids the bulk specimen with increasing strain for (c) 64% solid and (f) 93% solid.

strain of 26.1%. In contrast, at the higher solid fraction of 93%, the pores grow during uniaxial compression rather than shrink. In the more traditional 2-D image (Fig. 1d), this is not obvious as only a few small pores appear to have grown. However, the 3-D images and plot in Figure 1c,f, respectively, show that pores are growing quite significantly from 0.001% before deformation to 0.05% at a true strain of 2.44%. Compression can thus either decrease or increase porosity in globular semi-solid alloys depending on solid fraction. These phenomena were next studied by quantifying the microstructural response to load around pores.

Two types of pore morphologies were identified at 64% solid: tortuous and round, examples of which are rendered in Figure 2a and c, respectively. By tracking the evolution of pore volume, Figure 2a,b shows that the smaller tortuous pores maintain a near-constant volume during the entire deformation. In contrast, Figure 2c.d shows that all round pores decrease in diameter during uniaxial compression. Some disappear completely, such as pores A, B and C, while others decrease significantly in size, such as pores D and E. 3-D renderings of pores C and E illustrate this in Figure 2c. Classically, the evolution of porosity during casting solidification is due to a combination of dissolved gases (in Al alloys, hydrogen) [30,31] and inadequate feeding of the volumetric shrinkage [31,32]. The equation describing the pressure differential  $\Delta P$  between the gas pressure in the pore and the pressure in the solidifying liquid is [33]:



**Fig. 2.** Pore behaviour at 64% solid: (a) 3-D renderings of typical tortuous pores at strains indicated in plot (b); (b) change in volume of the tortuous pores with increasing strain; (c) 3-D renderings of typical round pores at strains indicated in plot (d); and (d) change in volume of the round pores with increasing strain.

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