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Time-resolved synchrotron tomographic quantification of deformation-induced flow in a semi-solid equiaxed dendritic Al–Cu alloy

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The rheology of semi-solid alloys has been studied by a novel *in situ* tomographic technique. Via extruding an equiaxed Al–15 wt.%Cu alloy, the inhomogeneous coherent compression of the α-Al grains was quantified, including the interdendritic channel closure and formation of a liquid extrudate. This investigation not only provides important insights into the microstructural changes occurring during semi-solid deformation, but also offers a validation benchmark for segregation and rheological models.

Keywords: Semi-solid deformation; Segregation; Liquid flow; X-ray tomography

Understanding liquid migration through a deforming semi-solid medium is critical for a wide range of processes from metal casting [1,2] to volcanology [3–5]: during casting, deformation of semi-solid alloys can influence liquid flow, resulting in macrosegregation, which can degrade the mechanical properties of the final product [2,6–10]; semi-solid magma is deformed by convection and/or tectonic plate spreading, inducing melt migration and segregation [3,4,11].

In solidification processing, deformation in the semi-solid can induce a range of defects, including extrusion segregation in squeeze-casting [1] and surface exudation in direct-chill casting [7]. Although several prior investigations have identified deformation-driven melt flow as a possible mechanism of such defects [1,7,12], the influence of stress on a semi-solid alloy and the melt flow through the equiaxed microstructure are not clearly understood. Many models have been developed to predict the formation of those defects, based on the proposition of the mushy zone as a sponge saturated with liquid [2,7,13,14]. However, currently there are no direct validation techniques that capture the kinetics incorporated in this hypothesis; *in situ* synchrotron tomography is one possible solution.

Recently, high speed X-ray tomography has been utilized to perform four dimensional imaging (4D, i.e. 3D plus time) of the pore-scale fluid flow [15], solidification [16–18], and the influence of deformation on semi-sold alloys [19–21]. Tensile and uniaxial compression tests have been used previously with the help of 4D imaging to study semi-solid deformation; these were mainly focused on the formation of damage (hot tearing) as a result of the granular response of the mushy zone [19,21,22]. In this paper, we describe the application of an indirect extrusion cell to study the rheological behavior of the mushy zone and the mechanisms responsible for the liquid migration induced by deformation. Such an indirect extrusion cell can also be used to study how extrusion segregation and exudation form, since it mimics their forming conditions.

The sample was semi-solid, equiaxed dendritic Al–15 wt.%Cu; a cylindrical specimen 2.9 mm in diameter by 2.9 mm long was prepared using wire electro-discharge machining, and then inserted in a boron nitride holder with an inner diameter (ID) of 3 mm and outer diameter (OD) of 5 mm. An alumina tube (1.5 mm ID and 3 mm OD) was placed on top of the specimen forming an indirect extrusion cell (Fig. 1). The entire extrusion set-up was enclosed within a resistive furnace [21], mounted on a bespoke mechanical testing rig with inbuilt rotation (P2R [20,21]).

The experiment was conducted using 53 keV monochromatic X-rays on the I12 beamline at Diamond Light Source. A high speed X-ray imaging system was used,

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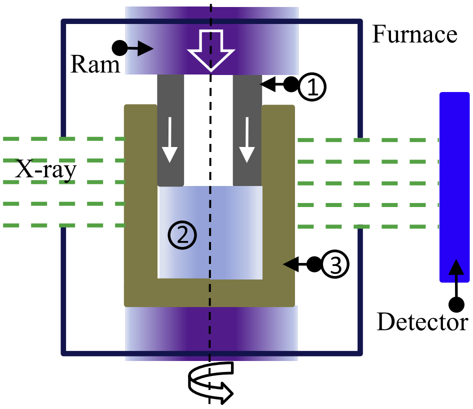

Figure 1. Schematic of the extrusion cell and *in situ* resistance furnace: 1 – alumina tube; 2 – specimen; 3 – boron nitride holder.

consisting of the beamline's custom-built imaging modules coupled to a CMOS camera (Miro 310M, Vision Research, USA). The imaging system provided a field of view (FOV) of 5.12 × 3.2 mm and 4 μm pixel size. The sample was positioned so that the top half of the billet and extrudate was in the FOV. The sample was heated to 560 ± 2 °C (27 ± 3% liquid fraction) in 15 min, and then held for 10 min for thermal homogenization. Subsequently, the top ram was moved down at 1 μm/s, forcing the alumina tube downwards while measuring loads.

Seven tomograms were captured, each comprising 900 radiographs, collected within 9 s at 45 s intervals. A filtered back projection algorithm was used to reconstruct the data to generate a tomography (unsigned 16-bit integral) [23]. Noise reduction was performed using a 3D median filter, followed by an anisotropic diffusion filter [24] using Avizo 8 (FEI VSG, France). Liquid phases were segmented by the Otsu method [25] using MATLAB 2012b (The Mathworks Inc., USA); errors were evaluated by varying the threshold value (24108) by ±50.

Figure 2a–c displays the resulting 2D longitudinal slices of the specimen under extrusion at the displacements of 0, 162 and 324 μm, respectively. The dark gray dendrites are the α-Al grains, while the Cu-enriched liquid is light gray. The corresponding 3D volume-rendered image is shown in the Supplementary information. A small amount of liquid segregated into the tube on top of the sample is notable (Fig. 2a at $d = 0$ μm); this extrudate is due to the stress caused by thermal expansion during heating. The subsequent response of the mush to the applied deformation is shown in Fig. 2b (162 μm) and Fig. 2c (324 μm). As deformation progressed, more melt flowed into the alumina tube from the semi-solid specimen. The liquid channels under the wall of the extrusion tube closed in response to the deformation (zone D in Fig. 2b and c). The evolution of the extruded liquid (Fig. 2e–g) displayed the characteristic profile of laminar flow in a pipe. We can also observe the closure of pre-existing porosity (Fig. 2e–i) due to the compressive strain.

In addition to making the above qualitative observations, we performed a detailed, time-resolved quantification of the extrusion. From $d = 0$ to 324 μm, the volume of the expelled liquid in the tube increased from ≈0.2 to ≈2 mm^3 at an almost constant rate of ≈0.0055 mm^3 per μm displacement. The extruded liquid volume increased at the same rate as the volumetric displacement (≈0.0053 mm^3/μm) of the alumina tube. The liquid fraction in the billet (lower part of the specimen) decreased from 26.7 ± 2.8% to 15.1 ± 2.1%, indicating densification of the mush (Fig. 3b). The extraction of the liquid by compression of the solid skeleton can be understood by considering the mush to be a saturated sponge, consisting of two phases (the solid grains and the liquid phase). This observation is contrary to the shear-induced dilation that is observed during direct shearing [26] and uniaxial semi-solid compression of equiaxed dendrites [21] and globular grains [22], where the liquid channels locally open rather than close. This suggests that different stress states can alter the fluid flow via different mechanisms (sponge or granular). The experiment reveals that constrained compressive stress densifies the solid skeleton and expels liquid from the mush (spongy-like behavior); shear stress is known to cause dilation, drawing liquid from the surrounding neighborhood into the dilated spaces between the grains (granular behavior) [21]. Therefore, when modeling semi-solid deformation, the effect of the stress states on the modulation of liquid flow needs be accounted for.

Along with liquid, a small amount of the solid phase was ejected into the die cavity (Fig. 2d–f). The peak height of extruded solid increased gradually (Fig. 3a). A magnified view of the extruded grains is shown in Figure 2j and k. Those grains located near the extruder inlet were free to move and appear to be sheared by the grains below, leading to dilatant translation and rotation (e.g. the grain A moved

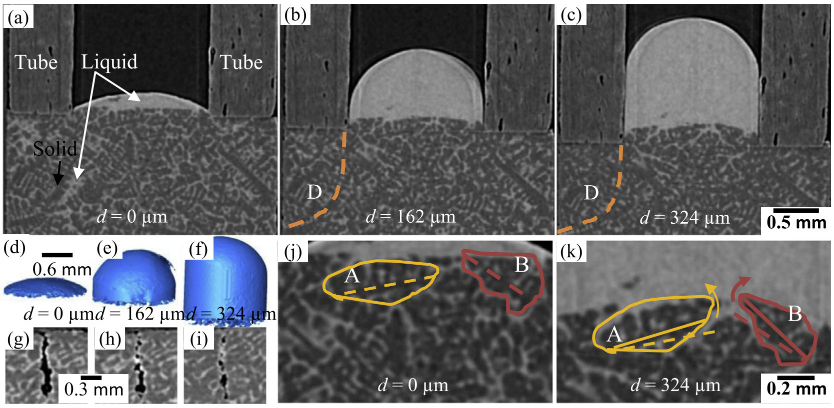

Figure 2. (a–c) 2D longitude slices of Al15Cu extruded at increasing vertical displacement; (d–f) 3D profile of the extrudate; (g–i) pore closure during extrusion; (j and k) magnified view showing the grain movements near the extruder inlet.