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Time-resolved synchrotron tomographic quantification of deformation during indentation of an equiaxed semi-solid granular alloy

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ABSTRACT

Indentation is a well-established technique for measuring mechanical properties, such as hardness and creep, in solid materials at a continuum level. In this study, we performed indentation of a semi-solid granular alloy with an equiaxed dendritic microstructure. The resulting microstructural effects were quantified using a novel thermo-mechanical setup combined with 4D (three spatial dimensions plus time) synchrotron tomography and digital volume correlation. The experiments not only revealed the multitude of deformation mechanisms occurring at a microstructural level, (e.g. dilatancy, liquid flow, macrosegregation, shrinkage voids, and intra-granular deformation), but also allowed quantification of the evolution of the strain fields within the material. The resulting methodology is a powerful tool for assessing the evolution of localized deformation and hence material properties.

1. Introduction

Indentation is one of the standards for probing local plastic deformation and characterizing the hardness and other material properties of solid samples [1–6]. Indentation has also been employed to investigate high temperature properties such as creep [7–9] and to obtain the deformation response of granular ensembles [10,11]. Granular media (e.g. sands, magma, sugar) consist of solid grains and pores filled with gas or liquid [12]. The behaviour of a granular material under indentation by a rigid body is of vital importance to a wide range of applications, including soil-machine interaction [13], the deformation of volcanic edifices by viscous magma [14], and semi-solid metal fabrication [15,16].

The deformation zone generated by indentation is determined mostly from the final-deformation state, providing minimal information on the dynamic evolution. This dynamic process includes two important aspects: (1) the microstructural changes induced by localized deformation and (2) the distribution and evolution of the strain fields as deformation proceeds. A considerable challenge is presented when attempting to map the complicated time-dependent evolution of microstructures, material flow and stress/strain fields induced by the indenting rigid body due to sample opacity. Particle tracking methods based on optical images (which is inherently a 2D process) [17–19], confocal microscopy [11] and magnetic resonance techniques [12] have been developed to partially overcome these challenges; as such, they can provide near-surface material flow gradients and/or strain evolution but are limited in their ability to determine the dynamics on a microstructural level. Recently, the ability to resolve microstructures in optically-opaque materials combining X-ray tomography and digital volume correlation (DVC) to quantify displacement field has been shown to be a useful approach to investigate sub-indentation deformation processes [4,20], although time-dependent information (e.g. velocity field) has not yet been obtained.

The rheological behaviour of semi-solid alloys has attracted significant attention recently (e.g. Refs. [21–28]). The complex interactions of solid–solid and solid–liquid phases have displayed not only characteristic granular behaviour [21,23,28,29] but also intra-granular deformation [21,25,30]. Dilatancy, an inherent

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deformation response of granular materials, has been shown to occur in semi-solid alloys [21,26]. Meanwhile, strain localization during semi-solid deformation has also been observed in some recently studies using uniaxial compression [21] and a direct shear cell [31]. However, the relationship between localized deformation and dilatancy has not been directly quantified. Indentation loading is known to generate a highly localized deformation zone in the vicinity of the indenter [1,4,32]. Hence indentation may be an ideal approach to reveal the correlation between dilatancy and inhomogeneous deformation as long as we can quantitatively assess both simultaneously. In addition, indent loading is of practical relevance for advanced casting techniques, because mechanical forces are both imposed on the surface and also penetrate deep into the components [15,16]. Discrete finite element models [33] and discrete element models [22] are currently being developed to address micro mechanic of semi-solids, which crucially requires well-designed experiments with both microstructure responses and strain evolution for their validation.

Here we report the combined use of high speed synchrotron X-ray tomography, digital volume correlation (DVC), and a bespoke *in situ* indentation rig with a resistance furnace to resolve the formation of deformation zones during indentation loading in 4D, offering a new perspective for micro mechanical tests of granular materials. Secondly, by indenting a semi-solid equiaxed dendritic Al–Cu alloy with a solid fraction of 72%, this study demonstrates that dilatancy is a direct response of localized deformation, leading in turn to liquid migration, solute-segregation and solidification porosity. This study demonstrates the advantage of combining multiple complementary techniques to gain new insights into the mechanics of deforming semi-solid granular materials.

2. Materials and methods

A cylindrical sample (3 mm diameter and 3 mm height) of Al-15wt.%Cu was placed inside a boron nitride holder with inner diameter of 3 mm and wall thickness of 1 mm. A bespoke thermo-mechanical rig ("P2R") was used together with a split open resistance furnace (Fig. 1) [21,34]. The indenter was a 30° flat-tipped (~250 μm diameter) cone made of alumina.

The experiments were performed at Diamond Light Source beamline I12 [35] with 53 keV monochromatic X-ray beam and coupled to a Vision Research Miro 310M camera via an imaging chain. The pixel size was 4 μm. The sample was heated to 560 ± 2 °C and held for 10 min, creating a semi-solid sample with a solid volume fraction (f_s) of 72%. The deformation was then started at a speed of 5 μm/s. High speed tomographic scans were taken in intervals of 9 s, acquiring one 3D dataset with 900 projections in 9 s over a sweep of 180°. Nine datasets were recorded in total, during which the indenter travelled a distance of 720 μm. Another single dataset was acquired at the end of the deformation process, after a total penetration of 1500 μm. Then the sample was cooled with the furnace turned off. A final dataset was recorded when the sample reached room temperature.

3D reconstructions were performed using the *Savu* system [36,37]. A 3D median filter followed by a non-local diffusion filter, using Avizo 8 (FEI VSG, France), was used to reduce noise. The liquid and pore phases were segmented from the solid using the Otsu threshold method [38]. The thickness of the liquid channels was measured using BoneJ [39]. The movement of individual grains (rotation angle and translation magnitude) at a representative vertical plane was determined via an image registration scheme (Avizo 8, FEI VSG, France). Due to the complexity of the dendritic structure and limited image contrast, automatic isolation of the individual dendrites could not be completed. Instead, a careful manual separation using visual observations was performed. A

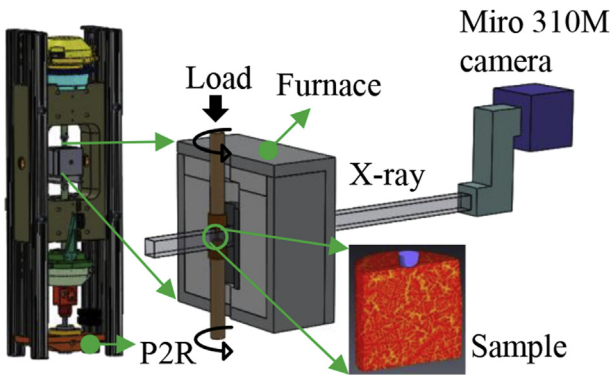

Fig. 1. Schematic of the experimental apparatus.

registration algorithm with affine transformation and an iterative optimization (Avizo 8, FEI VSG, France) was then applied to track the movement (translation and rotation) of the same dendrite from an indentation depth of $I = 0$–720 μm at the representative vertical plane.

DaVis Strain Master Version 8.1 was used to measure the 3D displacement fields. The DVC procedure is described in Ref. [21]. Here, a subset of 64 × 64 × 64 pixels with 50% overlap was used. The resulting spatial resolution of the displacement field is 32 pixels, corresponding to 128 μm. The displacement fields between successive tomographic datasets were integrated through the data series to calculate the accumulated displacement fields ($\mathbf{u}_i, i = x, y, z$). The velocity was obtained using $\mathbf{V}_i = \frac{\mathbf{u}_i}{\Delta t}$, where u_x, u_y, u_z denote the displacement field at x, y, z direction, respectively, and V_x, V_y, V_z represent the velocity component at x, y, z direction.

The accumulated displacement fields were also used to calculate the strain tensor ($\boldsymbol{\varepsilon}_{\mathbf{ij}}$, $i, j = x, y, z$) by a finite difference method. The strain tensor was further decomposed to octahedral normal strain (ε_n) and shear strain (ε_s), respectively.

3. Results and discussion

The influence of deformation zone formation on the microstructure is presented both qualitatively and quantitatively. The real-time tomographic imaging of indentation processes at high temperature and semi-solid state allowed us to quantify grain motion, the degree of dilatancy and associated liquid flow, together with strain evolution.

3.1. Grain motion

Fig. 2 shows a typical sequence of 2D, longitudinally-sectioned (y-z) tomographic slices recorded during the indentation of the semi-solid Al–Cu sample. Four different penetration depths ($I = 0$, 90, 360 and 720 μm) are shown in Fig. 2a to d, respectively. Supplementary movie S1 shows the full indentation process. The Al-rich dendrites are darker grey and the Cu-rich liquid is white, due to the difference in X-ray attenuation of the two phases (shown in reverse contrast, where lighter colour indicates denser materials). The initial equiaxed dendritic microstructure is clearly distinguishable (Fig. 2a). After 90 μm of indentation, Fig. 2b, the grains directly below the indenter were slightly displaced, causing the surrounding grains to move as well; this was buffered by the thin layers of intergranular liquid. With further deformation, the movement of grains continued (Fig. 2c and d, and more clearly shown in the Supplementary movie S1). Such movement indicates that a force chain was built up through the contact points of grains.