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In situ synchrotron tomographic quantification of granular and intragranular deformation during semi-solid compression of an equiaxed dendritic Al–Cu alloy

B. Cai [a,b], S. Karagadde [a,b], L. Yuan [c], T.J. Marrow [d], T. Connolley [e], P.D. Lee [a,b,*]

[a] *School of Materials, University of Manchester, Manchester, UK*
[b] *Research Complex at Harwell, Rutherford Appleton Laboratory, Harwell, Oxfordshire, UK*
[c] *GE Global Research Center, Niskayuna, NY, USA*
[d] *Department of Materials, University of Oxford, Oxford, UK*
[e] *Diamond Light Source Ltd., Harwell Science and Innovation Campus, Didcot, UK*

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Abstract

Semi-solid deformation mechanisms are important in a range of manufacturing and natural phenomena, which range from squeeze casting to magma flows. Using fast synchrotron X-ray tomography and a bespoke precision thermomechanical rig, we performed a four-dimensional (3-D plus time) quantitative investigation of the granular behaviour of equiaxed dendritic three-phase materials. This methodology produced new insights into the formation of damage during the isothermal semi-solid compression (~30% liquid fraction) of an Al–15 wt.%Cu alloy at both a macroscopic and microscopic level. Grain rearrangements, such as translation and rotation, were observed and lead to local dilatancy. The resulting flow of Cu-rich intergranular liquid into the dilated interstices gave rise to a local increase in liquid fraction, followed by rapid void growth above a critical axial strain of −6.4%. The local normal and shear strain distributions were quantified using digital volume correlation, identifying dilatant shear bands. At a microstructural level, the individual grains were also seen to undergo intragranular deformation, leading to bending and fragmentation of dendrites as grains interlock.

Keywords: Semi-solid deformation; Granular mechanics; Microstructural response; Dilatancy; Hot tearing

1. Introduction

The microstructural and mechanical response of semi-solid mixtures in the range of 20–50% liquid fraction during deformation is termed semi-solid mechanics. This range of liquid fraction is important in both materials processing (e.g. metallic component fabrication [1,2]) and many natural phenomena (e.g. magma flows [3,4]). For example, during the casting of aerospace or automotive metallic components, the thermal contraction and/or imposed deformation during solidification can influence the microstructure and defects formed (e.g. grain size [5], porosity [6], segregation [7] and hot tearing [1,8]). In many industrial processes where deformation is imposed, such as semi-solid processing and twin roll casting, the effect is particularly strong [5,9]. Therefore, an improved understanding of the response of a solidifying structure to deformation is important when designing a range of manufacturing processes.

Semi-solid systems are conventionally treated as homogeneous media, described using governing laws based on a continuum approach [10,11]. However, this treatment

* Corresponding author at: School of Materials, University of Manchester, Manchester, UK.
E-mail addresses: peter.lee@manchester.ac.uk, p.d.lee@ic.ac.uk (P.D. Lee).

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cannot account for localized phenomena, such as strain localization, which leads to defects such as shear banding or void formation [12]. A granular mechanics approach has been proposed by some authors to link the microstructural evolution to the semi-solid responses due to deformation, by treating solid grains as particulate suspensions [5,12–15]. For example, Spencer et al. [13] discovered that a semi-solid metallic alloy's viscosity depends on the solid fraction and decreases with increasing shear rate (shear-thinning), analogous to colloidal suspensions. Tzimas and Zavaliangos [16] discussed the occurrence of dilatancy in semi-solid equiaxed alloys during compression, and more recently Gourlayet al. [2] reported that partially solidified alloys exhibit Reynolds dilatancy under shear leading to strain localization. Dilation is also an important feature of saturated granular materials. The previous studies suggest that the size and morphology of solid particles and the liquid fraction influence the occurrence of granular phenomena in semi-solid alloys [17,18]. Although granular mechanics can explain some features observed during the deformation of three-phase solids, many other behaviours have been observed in such systems that are not currently explained by these models, such as grain-to-grain interactions and liquid flow, and the resulting localized phenomena at a microstructural scale, such as flow of solid particles [19,20] agglomeration/deagglomeration [9,21], viscoplastic deformation of grains [1,22,23] and damage formation [24–26]. Whether such behaviour can be explained by granular mechanics needs to be validated through experiments.

Commonly, constitutive equations have been used to describe the mechanical behaviour of semi-solid alloys via continuum analysis of a range of tests, including: tensile loading [26–29], compression [30–32], direct shear [33], rheometry [13,17,34] and indentation [35]. It is worth noting here that compression has been extensively used due to its ease of implementation and close resemblance to many key industrial processes. Additionally, properties such as the yield stress and viscosity of a semi-solid mush can be directly measured [30–32]. However, in most of these studies, the effects of deformation on microstructure were quantified only using post mortem analysis, limiting our understanding of any time-dependent kinetics. To understand and quantify the underlying kinetics or microstructure-dependent interactions, simultaneous measurement of the mechanical properties of semi-solid alloys and direct quantification of microstructural evolution with time is necessary.

A few recent studies have reported direct observation of granular shear deformation in semi-solid Al–15 wt.%Cu alloys [19] and low-carbon steel [36] using X-ray radiography, but did not measure the macroscopic mechanical behaviour. However, these 2-D studies did provide the first direct evidence of local dilatancy, induced by rearrangement of grains under shear deformation in metallic systems [19,37,38]. Unfortunately, these radiographic observations require a very thin sample thickness, and may not represent the 3-D bulk behaviour due to restricted out-of-plane motion and the friction of particles along the sample container wall.

Ultra-fast X-ray tomography can now overcome many of the limitations of radiography, resolving real-time 4-D information [39–42]. This technique has been used by several authors to quantify microstructure and defect formation during solidification [43–45] and under tension [8,46,47]. In this study, we present the first in situ 4-D (3-D plus time) quantitation of semi-solid compression of equiaxed dendritic grains. Using a bespoke thermomechanical rig designed for X-ray tomography, both the macroscopic mechanical behaviour and the evolution of microstructure and damage are simultaneously measured. The microstructural dynamics can then be correlated with the true stress and strain measurements, providing fundamental understanding of the responses of partially solidified alloys to the imposed loading. We demonstrate that this methodology can provide unique advantages when developing and validating semi-solid constitutive models, elucidating the behaviour of granular semi-solid systems and the nature of underlying granular deformation mechanisms.

2. Experimental methods

2.1. Materials

An Al–15 wt.%Cu alloy was selected for two key reasons: firstly to achieve a solid fraction typical of widely used commercial alloys (e.g. A356 [48]); and secondly for its X-ray attenuation variation between the primary phase and interdendritic liquid. The latter is due to the higher electron density of copper and its low partition coefficient, resulting in preferential segregation into the interdendritic liquid. Cylinders 3 mm diameter and 4 mm high were wire electrodischarge machined from 2 kg cast cylinders. Their microstructure was equiaxed dendritic, with a grain size of ~600 μm.

2.2. Testing apparatus and procedures

Semi-solid compression tests were performed using the bespoke P2R mechanical test rig [8] designed for in situ X-ray tomographic experiments, with air-bearing continuous rotation built into the load train. This allows simultaneous tension, compression and/or torsion during tomography, with 100 nm motion and 0.1 N load measurement precision. A bespoke PID-controlled resistance furnace with an X-ray-transparent window [8,41,49] was mounted on the mechanical rig, and the entire thermomechanical setup was integrated into the I12 beamline at Diamond Light Source (Fig. 1a).

The experimental setup is shown in the insert of Fig. 1a. The specimen was placed at the centre of a boron nitride holder (inner diameter 7 mm, wall thickness 1 mm) to ensure that the sample was secure and the deformation was unconstrained. A pre-load of 3 N was applied to