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In situ X-ray observation of semi-solid deformation and failure in Al–Cu alloys

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Abstract

Semi-solid deformation has been directly observed in an Al–12 wt.% Cu alloy through the combination of real-time synchrotron X-ray radiography and a bespoke high-temperature tensile tester over a range of fraction solid from 0.35 to 0.98. During deformation at low and moderate fraction solids, the X-ray radiographs indicate that there is significant feeding of interdendritic liquid in the region of strain localization prior to crack formation. Furthermore, the measured load required to initiate localized tensile deformation was found to be similar over the range of fraction solid 0.35 to 0.66. At higher fraction solids, the radiographic observations are consistent with classical hot tearing behaviour: limited liquid flow due to low permeability; void nucleation and coalescence; and final failure. Based on these results, a three-stage mechanism for semi-solid failure is proposed which includes the effects of liquid flow and micro-neck formation.

Keywords: Semi-solid behaviour; Al–Cu alloys; X-ray radiography; Deformation; Fracture

1. Introduction

The study of deformation in semi-solids is important for a great number of phenomena, from the solidification of metallic alloys to the flow of granular materials such as magma flow and pastes [1–6]. While low tensile strain of the semi-solid is accommodated by liquid flow, grain rearrangement and/or plastic deformation, the combination of increasing strain above ~2% and high fraction solid results in void formation [7,8]. During solidification of alloys, these voids may coalesce and form macroscopic cracks, defects known as hot tears, in the semi-solid.

Direct observation of semi-solid tensile deformation is challenging because of the high temperatures and the similarity in opacity of both the solid and liquid phases. In spite of these challenges, in situ observation of this deformation has been attempted by a number of techniques. Pellini [9] used X-ray radiography to observe hot tear formation in Al–Cu alloys at low resolution. Farup et al. [10] combined an optical microscope and hot stage to study hot tear formation in succinonitrile–acetone. Both Davidson et al. [11] and Mitchell et al. [12] recorded the formation of surface hot tears in an Al–Cu alloy using a closed-circuit television camera. The results presented by the above researchers suggest that semi-solid deformation is not homogeneous at the scale of the microstructure, but is accompanied by a process of strain localization due to local variations in grain size and shape. Furthermore, although the observations were of shear behaviour and not tensile deformation, the results obtained by Metz and Flemings [13] and more recently by Gourlay et al. [14–16] have been quite revealing with respect to the fundamentals of semi-solid deformation mechanisms.

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Unfortunately, the majority of the above studies lacked the combination of high resolution with a sufficiently large field of view required to see how the microstructure and interdendritic flow influence strain localization.

One alternative potential method for observing semi-solid tensile deformation is a laboratory or synchrotron X-ray source, which will provide images with good phase contrast, high resolution and fast acquisition times. Recently, Terzi et al. [17] performed such an experiment using a synchrotron-based three-dimensional X-ray microtomography setup to investigate semi-solid deformation in an Al–8 wt.% Cu alloy. This experiment corroborated the earlier findings of non-homogeneous semi-solid deformation while observing the accumulation of liquid and the formation of voids. However, since the results of only one specimen were presented, the effects of temperature and fraction solid could not be characterized. In the present study, the deformation behaviour of a semi-solid Al–12 wt.% Cu alloy has been examined over a range of temperatures within the semi-solid by combining synchrotron-based X-ray radiography with a bespoke semi-solid deformation apparatus. This combination allowed for measurement of the various load–displacement relationships while concurrently observing the evolution of solidification microstructures and the formation of a hot tear.

2. Experimental

2.1. Material

To perform the experiments, an Al–12 wt.% Cu alloy was cast using a directional solidification apparatus [18,19]. The resulting microstructure was equiaxed with a grain size of 120 μm. Flat tensile specimens 100 mm (length) × 5 mm (height) × 1 mm (thickness) were then machined from this master alloy.

2.2. Semi-solid deformation apparatus

The semi-solid deformation apparatus consisted of a tensile/compression platform with an integrated infrared furnace, as shown in Fig. 1. Axial loads were measured using a 250 N load cell with a resolution of 0.1 N. Cross-head displacement was obtained using a two-shaft cylindrical aerostatic journal bearing with a cable and pulley system. The displacement was controlled via a rotary encoder with a resolution of 0.4 μm, and a linear encoder with a resolution of 0.2 μm. An annular Heraeus Noblelight Omega infrared heater was used to heat the specimen. This annular shape allowed the X-rays to pass unhindered from the X-ray source through the sample to the detector. Heat was focused onto the gauge length using a selective gold coating on the infrared furnace, creating a parabolic temperature profile with a hot spot at the centre of the gauge region. The temperature of the specimen was monitored using a single K-type thermocouple placed at the hot spot. Although the thermocouple may separate from the specimen during the test, the intention is to perform isothermal deformation experiments. In this case, furnace control is adequate to maintain the isothermal condition. The error in the temperature reading is estimated to be in the range of 5 °C due to variations in contact pressure between the thermocouple and the tensile specimens.

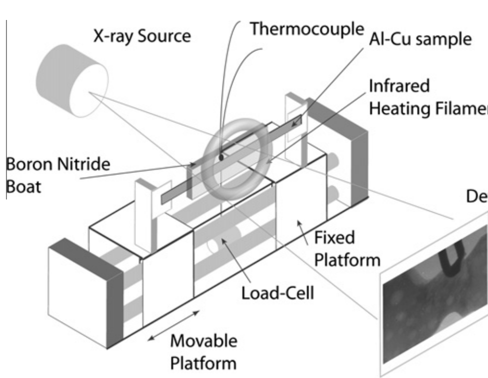

Fig. 1. Schematic of the semi-solid deformation apparatus.

To contain the molten metal, the specimen was placed within a boron nitride (BN) container, which was open at both ends and therefore free-floating, i.e. supported only by the solid outside the gauge region. A BN container was used due to its unique combination of properties: non-wettable by most molten metals including Al alloys, high thermal conductivity, and relatively transparent to X-rays. These unique property combinations also ensure that there is very little friction between the molten metal and the container during deformation. The inner cross-section of this container was also 5 × 1 mm, i.e. only slightly larger than the cross-section of the test specimen, in order to maintain specimen shape during deformation. The combination of a tight-fitting container and the layer of aluminum oxide on the specimen's surface meant that the initial specimen cross-section geometry of 5 × 1 mm was maintained during heating and melting, reducing in size only once significant strain was imparted. The ability of the aluminum oxide skin to contain molten metal has been well documented in the literature [20].

2.3. X-ray radiographic observations

Real-time observations of semi-solid deformation were performed using X-ray radiography. The Diamond B16 beam-line (Diamond Light Source, Didcot, UK) provided the X-ray source, while the images were acquired at an image capture rate of 1.25 Hz and a spatial resolution of ~3.0 μm using a 4008 × 2672 pixel detector. The specimen gauge thickness in the X-ray beam direction was 1 mm.

2.4. Test methodology

Testing consisted of applying tensile deformation at various stages of solidification. In total, four tests were conducted. First, the test specimens were heated until the