In Situ Environmental Scanning Electron Microscopy (ESEM) of Semi-Solid Samples

A.J. Smith[a], H.V. Atkinson[b], S.V. Hainsworth[c], H.B. Dong[d] and R. Haghayeghi[e]

Mechanics of Materials Group, Department of Engineering, University of Leicester, University Rd., Leicester, LE1 7RH, UK.

[a]ajs55@le.ac.uk, [b]hva2@le.ac.uk, [c]svh2@le.ac.uk, [d]hd38@le.ac.uk, [e]rh126@le.ac.uk

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Abstract. The ESEM (Environmental Scanning Electron Microscope) is an instrument that circumvents a limitation of conventional SEM, in that samples can be examined in a gaseous atmosphere rather than a vacuum. With a heating stage, dynamic processes can be observed *in situ* at high temperature. In this study, A201 aluminium alloy samples with globular structures have been examined in the semisolid region. In addition, a manipulator has been installed onto the heating stage to allow the probing of semi-solid surfaces. The paper shows the potential for manipulating semi-solid materials in order to better understand thixotropic phenomena.

Introduction

An Environmental Scanning Electron Microscope (ESEM) is a modification of a traditional Scanning Electron Microscope (SEM) in that it allows the sample to be imaged under a low pressure atmosphere of the operator's choice [1]. Atmospheres regularly used include water vapour, nitrogen and argon. The electron beam ionises the gas to give positive ions and these, in turn, reduce the charge build-up that occurs on the material being sampled, which charges negatively due to bombardment with electrons. The ionised gas also acts to amplify the image signal when secondary electrons are used to image the surface. The ESEM can be used with a heating stage. The sample is placed in a small ceramic cup. Above 400˚C, a heat shield, with a small hole to allow imaging, must be placed above the ceramic cup to protect the detector. The Gaseous Secondary Electron Detector (GSED) is placed directly above the sample, around the pole piece, and is used to acquire images when the heating stage is in use; other detectors are sensitive to the light emitted by hot samples. Energy Dispersive X-ray analysis (EDX) cannot be carried out at high temperature (and the detector must be retratced) because the EDX detector is also sensitive to heat.

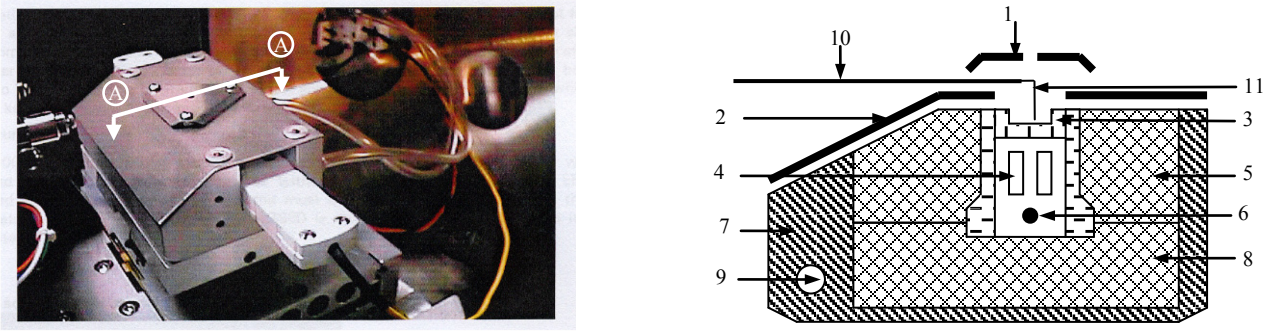
A number of researchers have used the ESEM with a heating stage to carry out *in situ* studies of dynamic processes in metallic systems at high temperatures [2-4]. One of these papers [2] represents a preliminary study of a metallic system in the semi-solid state. None of these studies has involved micromanipulation of the sample. Here we will present results for an aluminium alloy which has been produced by magneto-hydrodynamic stirring (and thus has a spheroidal microstructure) and then reheated into the semisolid state in the heating stage in the ESEM. In addition, we have carried out some probing of the sample surface with a one-axis manipulator. The issue here is that the micromanipulator is driven by a piezoelectric transducer. Such transducers are sensitive to temperature and cannot be operated above about 100˚C. However, the probe tip, if we are dealing with a semisolid aluminium alloy, is at around 600˚C or more. Thus, there is an engineering design challenge in protecting the piezoelectric transducer from the heat. We will show here that this can be achieved.

Experimental Procedure

Alloy Material. The material studied here was a silver-containing high strength casting aluminium alloy A201 supplied in the Magnetohydrodynamically (MHD) stirred state by SAG. The composition is 4.8% Cu, 0.25% Mg, 0.087% Si, 0.145% Fe, 0.29% Mn, 0.0026% Zn, 0.25% Ti, 0.564% Ag, 0.0012% Be, 0.0001% Sr. All percentages are in wt.%. The samples were polished to a one micron diamond finish before being placed in the heating stage.

ESEM. A Philips XL-30 ESEM from FEI was used with the FEI 1000˚C heating stage. The heat shield above the crucible (Fig. 1) restricts how close the GSED detector can be to the sample. The working distance was generally between 13 and 14 mm. Smaller working distances would improve image quality but this was not physically possible. The sample was heated in a water vapour atmosphere with a pressure of 2.0 torr. The imaging voltage was 25kV. The sample was heated at a rate of 20˚C/min from room temperature to 500˚C and then at 5˚C/min to 580˚C with a 3 min hold at 580˚C. It was then heated at 5˚C/min to 650˚C and then cooled. From differential scanning calorimetry [5], the semisolid state is expected to initiate at around 530˚C. In the experiments with the manipulator, the same heating routine was applied. The heating stage was calibrated using Omegalaq heat sensitive paint, showing that the temperature readings were accurate to within about 10˚C at 600˚C. This is within the manufacturer's specification. The crucible is 5 mm in diameter and 2 mm deep and thus the sample must fit within this.

Micromanipulation. A one-dimensional micromanipulator from PiezoMotor was used. This gives movement in one axis in steps of ~5 microns. The manipulator is mounted on a turret to one side of the heating stage and a probe arm extension is fitted made of alumina, which inhibits heat conduction back to the piezoelectric. The extension has to fit beneath the heat shield (in a space of about 2mm) and has a protruding tip, angled downwards into the crucible. The probe tip was made of tungsten so as not to react with the sample and had a diameter of about 90 μm. In due course, finer probe tips could be used.



a.) Heating Stage Installed Inside ESEM

b.) Section A-A

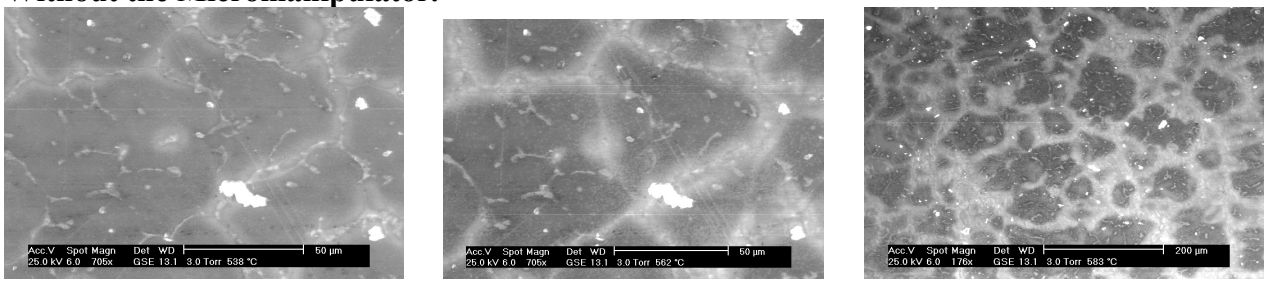
1 – Stainless Steel Heat Shield 2 – Stainless Steel Cover 3 – Alumina Crucible
4 – Heater 5 – Upper Zircar Block 6 – Thermocouple
7 – Aluminium Housing 8 – Lower Zircar Block 9 – Water Pipes
10 – Extension Arm 11 – Probe Tip

Fig. 1 ESEM heating stage (a) Heating stage installed in the ESEM, (b) Cross-section through the stage showing the component parts (courtesy of FEI).

At the start of the experiment with the manipulator, the probe tip was placed onto the sample with an initial downwards force applied, so that with any melting/softening of the sample, the tip would remain in contact. This is a particular issue with the one-dimensional manipulator which would not exist with a 3-D one, where vertical movement would be possible. The same heating routine was used as for the experiment without the manipulator, but at 578˚C the probe tip was moved and then again at 622˚C.

Results

Without the Micromanipulator.



(a) (b) (c)

(d)

Fig. 2 ESEM ımages of MHD A201 aluminium alloy reheated into the semisolid state. (a) 538˚C, (b) 562˚C, (c) 583˚C, (d) 604˚C.

Fig. 2 (a) shows phases at the grain boundaries. These are likely to be intermetallics and complex eutectics [6]. According to differential scanning calorimetry information [5], melting should barely have initiated at this temperature. At 562˚C (Fig. 2 (b)), a few percent of liquid should be present. There is clearly some change in the microstructure in comparison with Fig. 2 (a). (Note that the bright white specks are dust but act as markers). By 583˚C (Fig. 2 (c) – note the change in magnification), considerable contrast is apparent in the microstructure. According to DSC, about 5% liquid should be present now. If the pale areas correspond to liquid, then the fraction appears to be higher. Alternatively, the pale areas might correspond with enhanced oxidation at regions which are enriched in alloying elements, given that the imaging atmosphere is water vapour. This requires further investigation. At 604˚C, the microstructure should contain about 10% liquid according to DSC, but it appears that the surface of the pale areas is puckering as though these are liquid (Fig. 2 (d)). At higher temperatures (620˚, 630˚C) the puckering is evident right across the surface.

With Micromanipulator.

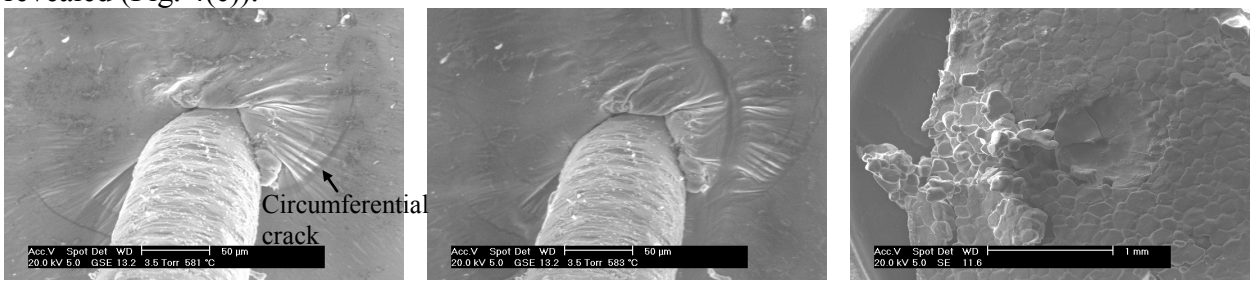
(a) (b) (c)

Fig. 3 ESEM images of MHD A201 aluminium alloy reheated into the semisolid state and probed with a micromanipulator tip, (a) 579˚C, (b) 622˚C, (c) 623˚C with tip moved back and forth.

In the micromanipulator experiment (Fig. 3), the probe was first moved at 579˚C (Fig. 3(a)). This temperature was chosen because it was thought that some liquid would be present from the above results and it would be possible to distort the sample. However, the tip would not move and it was only at a higher temperature of 622˚C that significant distortion was seen (Fig. 3 (b)). At this temperature, the probe tip was moved back and forth. The consequence was that a crater developed in the specimen (i.e. liquid did not flow back into the crater), (Fig. 3(c)), and the spheroidal structure was

clearly revealed. The alumina 'skin' on the surface of the sample is clearly being broken in Fig. 3(b); in effect, the probe tip is indenting the surface.

The micromanipulator experiment was repeated under very similar conditions. Fig. 4 illustrates the softness of the material at these temperatures and the classical development of circumferential cracks as though the probe tip were an indenter. It is not clear whether these lateral cracks are restricted to the oxide skin. At low magnification, after the experiment, the break up of the structure under the stress is revealed (Fig. 4(c)).



(a) (b) (c)

Fig. 4 ESEM images of MHD A201 Al alloy reheated into the semisolid state and probed with micromanipulator tip, (a) 581˚C, (b) 583˚C, (c) post-experiment at room temperature and low mag..

Summary

ESEM has been used with a heating stage and a micromanipulator to probe the behaviour of A201 metallic alloy in the semi-solid state. Contrast develops at the spheroid boundaries as the temperature increases, which may be liquid. The semisolid material is deformed by the probe tip and cracks develop in what is probably the oxide skin. At low magnification, the disruption of the structure is visible.

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