In-Situ Focused Ion Beam (FIB) microscopy at high temperature

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Abstract. Materials Scientists need information on the kinetics of the microstructural evolution processes that determine the finished microstructure, and hence the properties, of any material. E.g. recrystallisation, grain growth and phase changes. Such kinetic information requires reliable discrimination of differently oriented crystallites and/or different crystal phases coupled with useful spatial resolution and temporal resolution (i.e. high frame rates). These imaging results must be realised from a hot and changing specimen, in an instrument that is compatible with that hot specimen and with a practical specimen heater. Focused Ion Beams (FIB) offer strong contrast between crystallites and phases, and hence offer the ability to discriminate between these features even while imaging at fast frame rates, however their compatibility with hot specimens was unproven. Here we report results from a novel combination of FIB with an in-situ heating stage, to produce in-situ, real-time microstructural imaging from a variety of metallic specimens at temperatures up to about 700°C. FIB has additional capabilities, such as site-specific milling, which we show remains practical with hot specimens and which can be very useful with certain materials.

1. Introduction

The object of this work was to place a modification of FEI’s ESEM heating stage in an FIB/SEM instrument, specifically an FEI Quanta 3D DualBeam™. The intent was to demonstrate the feasibility of using such an instrument with a hot specimen, to discover the problems and opportunities raised by the combination of FIB with a hot specimen and to demonstrate its capability to give insight into the dynamic processes of microstructural evolution in model and industrial materials.

An FIB was used because one of the signals produced by specimens under an FIB is “ion induced secondary electrons” (ISE), which is analogous to the secondary electrons produced by an incoming electron beam. I.e. it is composed of low energy electrons “shaken loose” from the specimen by high-energy particles passing through the specimen surface layers. This ISE signal is of interest because the ISE yield is strongly dependent on the orientation of the local crystal lattice to the incoming ion beam [1]. The penetration of an incoming ion into the sample is small, but will be greater when the ion is travelling parallel to a low-indexed crystal plane, allowing it to “channel” through the crystal lattice. This greater penetration will result in very much lower ISE yield per incident ion, giving “channelling

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contrast” or “orientation contrast” (OC) in the resulting image. An analogous process occurs with backscattered electrons, but the penetration depth for an electron is relatively much larger than for an ion and the orientation-dependent change in penetration is proportionally much smaller. Therefore the maximum orientation-dependent change in yield of backscattered electrons per incident electron is much smaller than the orientation dependent change in ISE yield. Hence, the available OC is much lower in electron imaging than in ion imaging – particularly in light elements such as aluminium.

In-situ SEM with hot specimens has been proved practical [2] and has given good results [3]. However, it has limitations when applied to light metals (e.g. aluminium) and soft materials (e.g. gold).

2. Experiment
A modified hot stage (figure 1) was fitted into an FEI Quanta 3D Dualbeam™ instrument with an electron column at 15mm working distance and 90° to the specimen and a Ga+ ion column at 30mm working distance and 38° to the specimen. The microscope had additional vacuum gauges fitted to a chamber port in order to give continuous direct monitoring of the chamber vacuum (figure 2). The instrument was operated in high-vacuum mode, with secondary electron and ISE signals detected via the Everhart-Thornley detector.

Fig. 1. The hotstage, modified for FIB use. Fig. 2. The Quanta 3D DualBeam & instrumentation.

2.1. Specimens
Experiments were conducted on a variety of cold worked metal specimens, prepared by mechanical polishing. These were Copper (99.99+ mass% Cu, subjected to 4 passes of equi-channel angular pressing), Aluminium-Manganese (Al, 1 mass% Mn, cold rolled to 40% reduction), Gold (99.99+ mass% Au, cold worked to the “fully hard” condition) and commercial “2101 low-alloyed-duplex” stainless steel (nominally Fe, 21.5 mass% Cr, 1.5 Ni, 5 Mn, 0.3 Mo, 0.22 N, <0.03 C, cold rolled to 50% reduction).

Equi-Channel Angular Pressed (ECAP) Copper was selected as it was expected that this would produce high-quality results that the other specimens could be compared against.

Aluminium-Manganese was selected: 1) Because aluminium had caused difficulties during in-situ electron imaging, due to its low back-scattering coefficient. 2) In order to investigate the imaging of a two-phase system consisting of differing elements. 3) To investigate reactions between the gallium ions and the aluminium and show their effect on imaging at high temperature.

Gold was selected because it had proved problematic during in-situ electron imaging. Gold is difficult to prepare to a good mechanical polish due to its softness, even in the “fully hard” cold worked condition. Previous experience had shown that while it was possible to produce a well polished specimen at room temperature, the combined action of residual stress and surface diffusion
degraded the specimen surface to an unacceptable degree when heated. Gold also showed very pronounced thermally etched grooves at its grain boundaries when heated.

Duplex stainless steel was selected in order to investigate the imaging of a system with distinct crystalline phases but with a broadly isotropic distribution of elements.

3. Results

ISE images were captured at a variety of resolutions and proved well able to resolve the sub-micron crystallites in the ECAP copper (figure 3). The expected finished microstructures of the specimens dictated imaging at frame sizes of tens to hundreds of microns width, i.e. a maximum spatial resolution in the order of 200-1000nm per pixel. It was clear that higher magnification could easily have been used, if it had been appropriate to investigating grain growth in these specimens.

Image quality was good, with strong contrast between neighbouring grains (figure 4). Images were captured with no apparent temperature dependant effects, other than that noted below.

At a specimen temperature of approximately 750°C, specimens became progressively obscured by some deposit (presumably solid gallium oxide or liquid gallium films). The deposit appeared initially at the edge of the field of view and grew rapidly towards the centre (figure 5). This has been shown to occur at approximately the same temperature with copper, gold and stainless steel specimens and therefore seems to be specimen independent. The deposition is reversible and disappears when imaging is continued and the specimen temperature is reduced. The effect of further heating was not investigated, neither was the effect of cooling the specimens without ion-beam scanning.

The aluminium proved incompatible with Ga⁺ ion beam imaging, even when heated (figure 6). Material was deposited on the specimen surface and obscured the microstructure despite heating and ion beam milling effects.
The total material eroded by the ion beam during the course of the experiments (about 3 hours) was in the order of tens of nanometres, compared to finished grain sizes in the order of tens of micrometres.

Images of 1024 X 884 pixels (0.9 Megapixels) were captured at frame rates up to 0.35 frames/second (0.31 Megapixels/second) and retained good quality. Electron imaging in earlier work [2, 3, 4] captured images of 634 X 480 pixels (0.3 Megapixels) at frame rates up to 1 frame/second (0.3 Megapixels/second) but with much lower contrast and higher image noise than in this work.

4. Discussion
Temporal resolution of in-situ microscopy and other moving image techniques is normally stated in human-friendly terms such as “frames per second”, “seconds per frame”, or similar. However, when comparing the capabilities of different techniques this is generally not a useful objective measure as it fails to take into account the fact that different techniques and different implementations of the same technique will probably have different numbers of pixels in each frame. We therefore propose that a measure of “(mega)pixels per second” be used to quantify the temporal resolution of a microscopy technique. This is an objective measure that is independent of frame size and clearly shows the trade-offs that must be made in terms of spatial resolution (scanned area & number of pixels per frame) against temporal resolution (number of pixels per frame and number of frames per second).

To enable completely objective comparison of different techniques it seems necessary to develop some quantitative measure of image quality either in terms of contrast and noise or in terms of the ability of automated microstructure characterisation software to correctly recognise grains, boundaries and other microstructural features.

5. Conclusions
The combination of FIB with hot specimens is practical and capable of producing valuable insights into microstructural evolution processes.

Above some temperature (about 750°C), Ga⁺ ion beams interact with specimens so as to deposit material. This is reversible on cooling and appears to be a specimen independent temperature effect.

Ion beam milling of hot specimens is practical and its behaviour is not significantly different from that encountered at room temperature.

In-situ ion beam milling is a highly practical technique for preparing distortion-free, stress-free polished surfaces on hot specimens.

ISE imaging simultaneous with a modest rate of ion beam milling can suppress the formation of thermally etched grain boundary grooves.

Simultaneous imaging and milling can also suppress formation of oxide films on the hot specimen.

The in-situ microscopy research community needs to generate a consistent and practical means of quantifying and comparing temporal resolution from dynamic microscopy techniques.

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