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► **To cite this version:**

A. Waugh, S. Payne, G. Worrall, G. Smith. IN SITU ION MILLING OF FIELD ION SPECIMENS USING A LIQUID METAL ION SOURCE. Journal de Physique Colloques, 1984, 45 (C9), pp.C9-207-C9-209. 10.1051/jphyscol:1984934 . jpa-00224414

HAL Id: jpa-00224414

<https://hal.science/jpa-00224414>

Submitted on 4 Feb 2008

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IN SITU ION MILLING OF FIELD ION SPECIMENS USING A LIQUID METAL ION SOURCE

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Résumé - Un processus d'érosion ionique contrôlée est décrit. Il permet des applications intéressantes telles que le "déplacement" d'un joint de grain en direction de la zone imagée à l'apex d'un échantillon de microscopie ionique de champ.

Abstract - A controlled ion milling procedure is described which permits a feature of interest, such as a grain boundary to be brought towards the imaging region at the apex of a field ion specimen.

1 - INTRODUCTION

One of the main limitations on the practical use of field ion microscopy and atom probe microanalysis is the small volume of material which can be examined. As a result of this, the probability of finding a feature of interest in any one specimen is often very low. This is especially true for studies of grain boundaries, which in the case of coarse grain size materials are almost prohibitively difficult to find. Inspection of field ion specimens by transmission electron microscopy is a useful aid to finding boundaries, and this can be combined with back-polishing of specimens by chemical, electrolytic or ion milling techniques [1-3]. However, the electron-transparent volume of a field ion specimen is still very small, and the back-polishing procedures used so far are not always as controllable as might be desired. New possibilities for controlled specimen preparation have become available with the development of micro-focussed liquid metal ion sources (LMIS). These sources permit imaging with a resolution of 500Å or better using reflected secondary electrons, and also allow ion milling to remove material from the specimen surface [4-6]. In imaging mode, it is found that the contrast obtained across grain boundaries and other phase boundaries is exceptionally good, and therefore the progress of the ion milling reaction can be monitored very accurately [6]. This opens up the exciting prospect of controllably bringing towards the apex region of a field ion tip a feature of interest which may initially be situated at almost any distance down the specimen shank. We report here some initial experiments designed to prove the feasibility of this idea.

2 - EXPERIMENTAL

The liquid metal ion source used in this work was a VG Scientific MIG 300 gallium ion gun. This was operated at 25keV, with a beam current of approximately 50pA, and a focussed spot size of 500Å. The gun was mounted in a high vacuum chamber, equipped with a tiltable stage for holding field ion specimens. Imaging was carried out by scanning the focussed ion beam across the specimen in a raster pattern, exactly as done in a scanning electron microscope. A secondary electron detector was used, biased to collect secondaries of energy below 100eV, and subtending a large solid angle at the specimen.

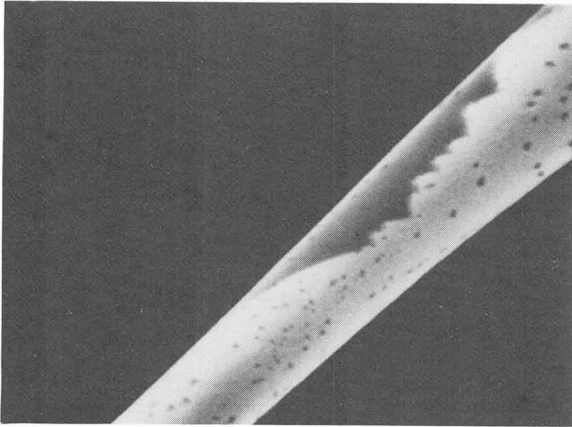


Figure 1
x 3000

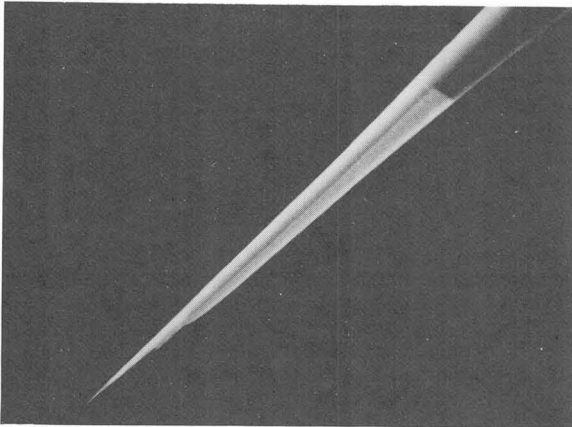


Figure 2
x 300

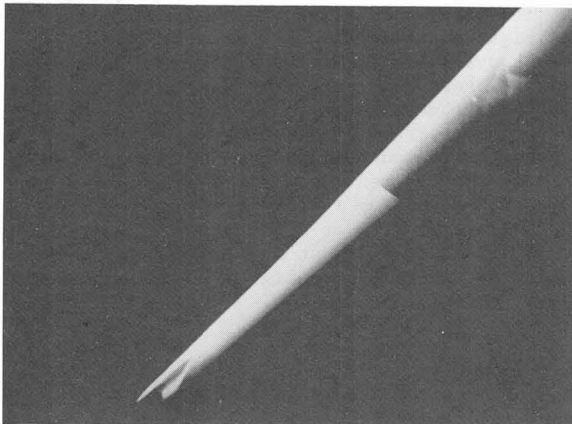


Figure 3
x 300

Scanning micrographs of an Inconel 600 FIM specimen obtained using a 25keV gallium liquid metal ion source. For details see text.

3 - RESULTS

The micrographs illustrated in Figs.1-3 were taken at various stages during the milling of a specimen of the alloy Inconel 600 (75% Ni, 15% Cr, 10% Fe). The material had been annealed for 1.6 hours at 1110°C and then held for 1 hour at 698°C before water quenching.

Figure 1 shows the contrast across a twin boundary which intersects the specimen surface. The faceted nature of the boundary can be clearly seen, with coherent and incoherent segments alternating along the boundary. (The much smaller dark spots are due to etch pits, arising during the initial electropolishing of the specimen).

Figure 2 shows a high angle grain boundary running transversely across the specimen, together with a second, longitudinal boundary (probably a twin), which emerges close to the tip apex. In Figure 3, the effects of a few minutes of ion milling this latter boundary "end on" (i.e. from the direction of the tip apex) are illustrated. This has an undesirable effect, in that a groove develops preferentially at the interface. It is evidently preferable to ion mill from a "sideways on" position, as most of the sputtering is then caused by ions striking the surface at relatively low angles of incidence, and a smoother profile is obtained. Rotation of the specimen is also necessary, as in conventional ion milling procedures for thin foils.

4 - DISCUSSION

These preliminary results demonstrate the feasibility of in situ ion milling of field ion tips, and open up new possibilities for controlled specimen preparation, not only for field ion microscopy, but also for transmission electron microscopy. The effects of the gallium ion beam on the specimens will have to be studied in detail. It may prove necessary to restrict the accelerating voltage to about 10keV in order to minimise beam damage and implantation effects. The combined use of argon ion beams (for milling) and low intensity metal ion beams (for imaging) also merits investigation, and may provide the best overall strategy for the preparation of specimens with the least possible damage.

ACKNOWLEDGEMENTS

Financial support for this work was provided by Science and Engineering Research Council, Central Electricity Generating Board and United Kingdom Atomic Energy Authority, Harwell. GMW wishes to thank United Kingdom Atomic Energy Authority for the provision of a CASE studentship.

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