JOURNAL OF MATERIALS SCIENCE 15 (1980) 681–684

Dynamic study of ion etching in a high resolution SEM

G. W. LEWIS, J. S. COLLIGON, M. J. NOBES
Department of Electrical Engineering, University of Salford, Salford, UK

On-line studies of surface topographical development have been made by mounting a saddle-field ion source into a standard scanning electron microscope. Preliminary results obtained during operation in both dynamic and static modes are presented.

1. Introduction

When an energetic ion beam bombards a surface, atoms of the material are sputtered. In many cases this process leads to the development of topographical features on the surface even for nominally pure amorphous samples. Sometimes such features are desirable, for example, where high coefficients of friction are required, or high absorption efficiency (e.g. for solar cells), or high surface areas (for catalysts). In other situations the roughening of a surface is undesirable, for example, when the ion bombardment is being used as a sectioning technique as in SIMS or Auger determinations of the depth concentration profiles of implanted layers.

At the present time, the mechanisms responsible for the roughening or smoothing of a particular surface are not well understood, although several theoretical attempts have been made to predict the development of amorphous pure materials during bombardment by a uniform ion beam [1–9]. The majority of existing experimental evidence of topographical development is based on the unsatisfactory technique of exposing a sample to an ion beam in one apparatus and then transferring it to a microscope for analysis. Whilst this technique provides excellent isolated examples of the dynamic cycle of surface development it cannot give the full picture, in particular the critical early stages of feature formation.

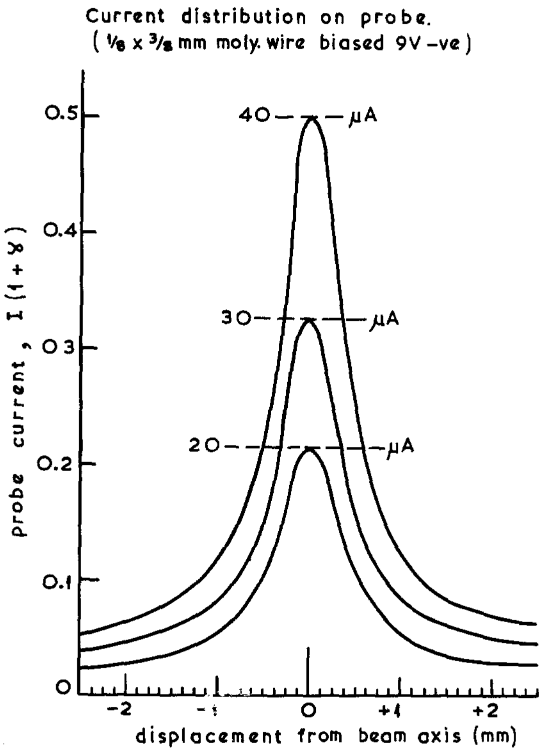
This paper describes preliminary results using an apparatus in which an intense low-energy ion source is mounted inside the standard target chamber of a high resolution SEM. This allows the surface features of a bombarded sample to be continuously monitored and has the advantage over the technique previously reported by Dhariwal and Fitch [10] of having much better resolution (up to x 10^5 magnification) which gives finer detail of the process.

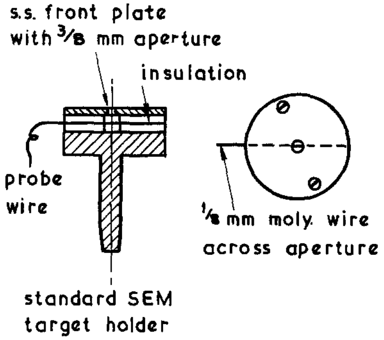
2. Experimental details

A standard Cambridge S 4.10 scanning electron microscope has been used for the present research programme. This instrument allows samples to be viewed at magnification up to x 10^5 and has several convenient blanked ports around its periphery, two of which have been used for the present additional assembly. A saddle-field ion source originally proposed by McIlraith [11] and later developed by Fitch *et al.* [12] and Rushton [13] has been mounted onto the specimen chamber as indicated in Fig. 1. Owing to the limited space in this chamber and the need to preserve the standard goniometer assembly for other experimental work, the source is mounted on guide rails placed at 5° to the horizontal plane. It can be slid back along these rails using a rigid locating rod which penetrates the vacuum flange via an O-ring shaft seal and, when not in use, the source is "parked" in the hollow cavity forming part of the standard SEM chamber wall, thus allowing the microscope to be used in its normal mode for other work. A second O-ring shaft seal allows manipulation of screen B which must be placed in front of the photomultiplier–scintillator assembly to protect it during sputtering of the samples.

In preliminary tests of the ion source it was found that its insulators became contaminated after a few hours operation. This appeared to be associated with the excessive heating of the source

0022–2461/80/030681–4$02.40/0





Typical running conditions

Anode Voltage kV	Discharge Current mA	Total Current on target μA
8.5	2.0	40
7.3	1.6	30
6.0	1.2	20
4.0	0.4	5

Figure 1 Schematic diagram showing mounting of ion source in SEM.

which dissipates a significant amount of electrical power and, in its present mounting position, does not have a good thermal contact with a large mass of metal. Fortunately this problem was overcome by arranging for the guides to be constructed of hollow tubes through which cooling water could be passed. With this modification the source has now operated for over 100 h without the need for overhaul.

Because the SEM can examine particularly small areas of the bombarded sample over which the current density must be very nearly uniform, the actual beam density distribution is not critical. However, inspection of bombarded samples at

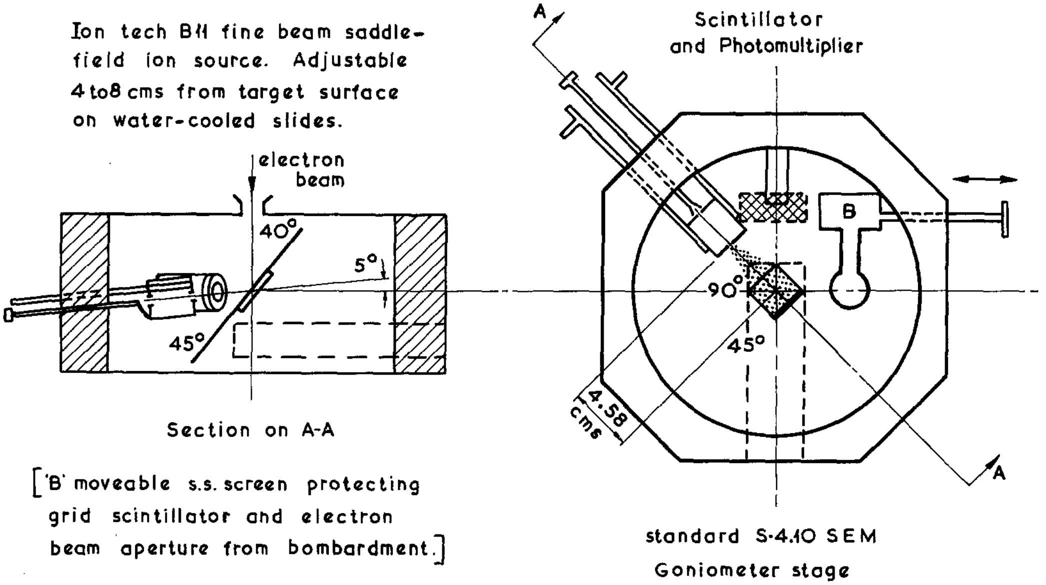


Figure 2 Ion beam profiles.

low magnification clearly showed an egg-shaped depression indicating that the beam density was far greater in centre than at its periphery. A current probe constructed of a $\frac{1}{8}$ mm wire mounted in a closed box behind a $\frac{3}{8}$ mm diameter aperture (see Fig. 2) was used to measure this current density distribution and gave results consistent with the etch-pit shape (see also Fig. 2). It was decided to utilize this property of the ion source since, at different radii, the sample has experienced a unique fluence of ions, i.e. as radius increases, the corresponding total ion dose "seen" by a sample decreases. Results could therefore be obtained in the so-called "static" mode following one specific dose of ions, where pictures of the surface were recorded at various radii or they could be obtained in a "dynamic" mode where a specific point on the surface was repeatedly inspected after periods of ion bombardment. However, due to a finite beam divergence angle of ± 4° and possible influence of this on the topographical development some inconsistency may be introduced in the interpretation of the "static" morphology, especially towards the beam periphery.

3. Results and discussion

It will be apparent from Fig. 1 that the incident ion beam for sputtering was always at 45° to the sample surface whilst the axis of the electron beam of the microscope intersected the sample at 40° to its surface. The scintillator "viewing" angle is complex being the resultant of two angles in different directions and turns out to be 33°. Ion energy from the saddle field source is not well defined. Experiments were usually carried out with the anode voltage at 6 to 8 kV which, from retarding potential experiments, implied an average beam energy of 3 keV.

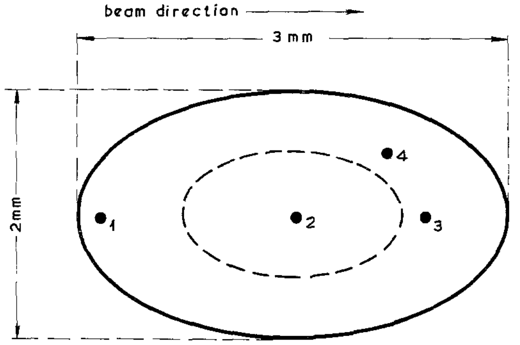


Figure 3 Static experiment for Ar^+ on silicon. Micrographs refer to surface structure appropriate to areas indicated. Total dose at centre (2), neglecting secondary electron and neutral atom contributions, $= 7.4 \times 10^{19}$ ions cm^{-2}. In all micrographs, ion-beam projected direction is towards lower right-hand corner. Interval indicated is 1 μm.

Two sets of experiments demonstrate the flexibility of the apparatus and are typical of many others obtained using this facility. Fig. 3 shows the results for a static experiment using Ar^+ on silicon where different positions on the surface

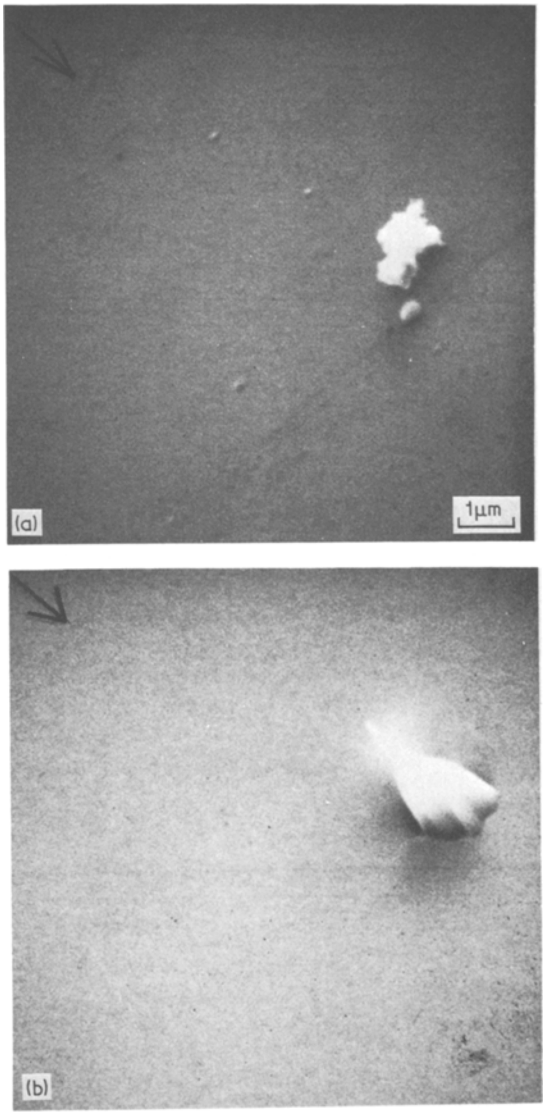
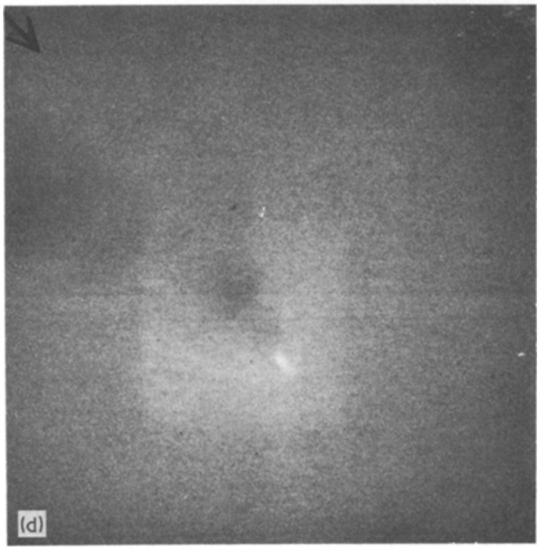


Figure 4 Dynamic experiment for Ar^+ on silicon. Bombardment time associated with each micrograph is: (a) 0 (unbombarded), (b) 35 min, (c) 60 min, (d) 120 min, (e) 270 min, where, neglecting secondary electron emission and neutral atom contributions, 125 min is equivalent to 1.5×10^{18} ions cm^{-2}. In all micrographs ion-beam projected direction is towards lower right-hand corner. Interval indicated is 1 μm.



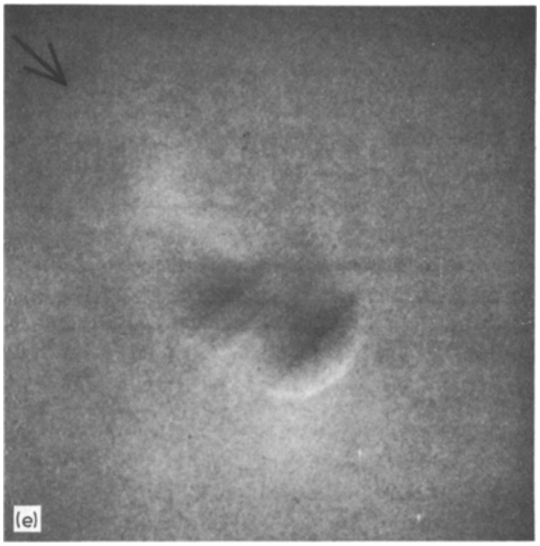


Figure 4 (continued)

correspond to different total fluences. In the centre (2) the silicon relief is most developed having experienced a high total dose of ions whilst at the edge (1) no appreciable relief exists. These results are consistent with those obtained for the same sample bombarded in the dynamic mode.

One of the most interesting features of the present apparatus is its ability to follow the development and subsequent annihilation of a particular feature on the surface, such as a cone. Such a progression is shown, again for Ar$^+$-silicon, in Fig. 4 and clearly indicates that, even when the cone has disappeared, the crater associated with it remains.

4. Conclusions

It has been shown that the addition of a saddle-field ion source to a standard SEM allows the attainment of a series of correlated pictures of an ion-bombarded surface from which not only a feature can be seen, but also a development of that feature with time can be followed. This extra dimension of time will greatly assist in the development of a better understanding of the problems of surface erosion and will assist evaluation of theoretical models proposed to describe surface topographical changes.

References

1. A. D. G. STEWART and M. W. THOMPSON, *J. Mater. Sci.* 4 (1969) 56.
2. M. J. NOBES, J. S. COLLIGON and G. CARTER, *ibid* 4 (1969) 730.
3. G. CARTER, J. S. COLLIGON and M. J. NOBES, *ibid* 6 (1971) 115.
4. C. CATANA, J. S. COLLIGON and G. CARTER, *ibid* 7 (1972) 467 Letters.
5. G. CARTER, *ibid* **11** (1976) 1091.
6. D. J. BARBER, E. C. FRANK, M. MOSS, J. W. STEEDS and I. S. T. TSONG, *ibid* 8 (1973) 1030.
7. J. P. DUCOMMUN, M. CANTAGREL and M. MARCHAL, *ibid* 9 (1974) 725.
8. G. CARTER, M. J. NOBES and J. L. WHITTON, *ibid* **13** (1978) Letters.
9. G. CARTER, M. J. NOBES and K. I. ARSHAK, *Wear* 53 (1979) 245.
10. R. S. DHARIWAL and R. FITCH, *J. Mater. Sci.* **12** (1977) 1225.
11. A. H. McILRAITH, *Nature* **212** (1966) 1422.
12. R. K. FITCH, T. MULVEY, W. J. THATCHER and A. H. McILRAITH, *J. Phys. D* 3 (1970) 1339.
13. G. J. RUSHTON, K. R. O'SHEA and R. K. FITCH, *J. Phys. D* 6 (1973) 1167.

Received 4 July and accepted 24 July 1979.