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LOCALIZED ION MILLING OF METALLIC AND CERAMIC TEM SPECIMENS

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The utility of localized ion milling in the preparation of metallic and ceramic specimens for TEM and AEM is being evaluated from the standpoint of optimizing procedures and identifying limitations and milling characteristics. The equipment used was a Gatan model 645 precision ion milling system (PIMS). This device is a scanning ion beam instrument with which selected areas of a sample can be imaged by either secondary electron or secondary ion signals and selectively ion milled in a small region within the imaged area. The 1 to 10 keV ion beam can be focussed to a 2 μ m spot. Specimens are thinned without removing them from the electron microscope specimen holder.

Metallic alloys (Fig. 1) and semiconducting ceramic specimens (Fig. 2) have been successfully milled. Insulating ceramics have posed more of a problem because of secondary electron suppression due to a buildup of charge. The loss of a reasonable quality secondary electron image precludes the accurate location of the ion beam for localized milling. A thin coating of carbon, which is normally required for TEM examination anyway, may help but can soon be milled away at high magnification operation. We are exploring the possibility of installing an electron flood gun or other charge neutralizing device to overcome this serious problem.

The system is expected to greatly enhance the specimen preparation process for cross-sectional specimens of ion-implanted ceramics (and metals), oxidized alloys, and rapidly solidified splats. The instrument should also be extremely useful in obtaining electron energy loss spectroscopy (EELS), high spatial resolution x-ray microanalysis and lattice imaging data on regions that would otherwise be too thick. One example is the analysis of second phase particles in structural materials. At the moment, EELS analysis of such particles is limited to ones at the edge of the foil (usually an infrequent occurrence) where information on the surrounding microstructure, that is often vital for interpretation of the results, is lost. Another application we are interested in is the correlation of grain boundary segregation with grain boundary structure. We envisage performing diffraction contrast experiments on relatively thick regions (~100nm) where such experiments are easier than in the thinnest regions, followed by localized ion milling for optimizing high spatial resolution x-ray microanalysis. Figures 1 (a,b) and 2 show that such thinning of grain boundaries is possible. However, in Fig. 1 (c) which shows a region containing an annealing twin, selective milling that is strongly orientation dependent is evident. Such crystallographic orientation dependence is not unexpected and is mitigated in conventional broad-beam ion millers by employing specimen rotation and milling with ion beams at small angles of incidence.

The limited resolution and secondary electron or ion contrast that is very different from a TEM image can make the location of the desired area difficult. However, area recognition is usually possible if characteristic features are sought at low magnification in the TEM and PIMS. We have had limited success in milling small areas that are not already electron transparent [Fig. 1(d)]. Perforation is certainly possible but little thin area is produced. Again perhaps the limited tilt angle for milling and the absence of specimen rotation contribute to this undesirable situation. Finally, the technique suffers the same problems of "black spot" damage or the formation of amorphous surface layers that can be limiting factors in conventionally ion-milled specimens.¹

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FIG. 1.—Micrographs showing locally milled areas of $(\text{Fe,Ni})_3\text{V}$ specimen. (a) Region near grain boundary that was originally $\sim 150\text{nm}$ thick. (b) Same region, shorter photographic exposure to show thin area around secondary perforation. (c) Region containing annealing twin. Note selective thinning. (d) Secondary perforation with little thin area in region not originally electron transparent.

FIG. 2.—Micrographs showing same region of silicon carbide (a) before and (b) after localized ion milling. Diffraction conditions are identical in both micrographs.