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Scott, J., Docherty, F.T., MacKenzie, M., Smith, W., Miller, B., Collins, C.L. and Craven, A.J. (2006) *Sample preparation for nanoanalytical electron microscopy using the FIB lift-out method and low energy ion milling*. Journal of Physics: Conference Series, 26 (1). pp. 223-226. ISSN 1742-6588

<http://eprints.gla.ac.uk/52636>

Deposited on: 7 June 2011

Sample preparation for nanoanalytical electron microscopy using the FIB lift-out method and low energy ion milling

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Abstract. Thinning specimens to electron transparency for electron microscopy analysis can be done by conventional (2 - 4 kV) argon ion milling or focused ion beam (FIB) lift-out techniques. Both these methods tend to leave “mottling” visible on thin specimen areas, and this is believed to be surface damage caused by ion implantation and amorphisation. A low energy (250 – 500 V) Argon ion polish has been shown to greatly improve specimen quality for crystalline silicon samples. Here we investigate the preparation of technologically important materials for nanoanalysis using conventional and lift-out methods followed by a low energy polish in a GentleMill™ low energy ion mill. We use a low energy, low angle (6 - 8°) ion beam to remove the surface damage from previous processing steps. We assess this method for the preparation of technologically important materials, such as steel, silicon and GaAs. For these materials the ability to create specimens from specific sites, and to be able to image and analyse these specimens with the full resolution and sensitivity of the STEM, allows a significant increase of the power and flexibility of nanoanalytical electron microscopy.

1. Introduction

One common method used to prepare specimens for electron microscopy utilizes argon ion beams to thin samples to electron transparency. The specimen is normally mechanically thinned first by and/or dimpled. Typically beams accelerated by 2 - 4 kV are used, with low (4 - 8°) incidence angles to minimize beam damage. In the last decade focused ion beam (FIB) systems have also been used to prepare electron microscopy specimens [1,2]. Both these techniques cause damage to the specimen surfaces [3,4]. Damage can be minimized in two ways: by using lower energy ions and by reducing the incidence angle to the surface. In both FIB and conventional techniques, it is usually not possible to reduce the ion incident energy below a few kV without unacceptable penalties in milling time due to the reduced sputtering rate. The main focus is therefore on reducing the angle of incidence to as low a value as possible. When using a Gatan PIPS ion mill this is governed by the thickness of the sample, and typically a value of ~4° is used.

In the FIB it is possible to tilt a specimen to any angle to mill the specimen surfaces. Preparing specimens using a FIB system has several advantages over conventional techniques. When examining silicon or GaAs based devices, the imaging capability of the FIB allows specimens from specific areas of interest (e.g. single transistors) to be prepared. When applied to steel samples, the orientation-dependant channeling contrast of grains in the material allows grain boundaries and other interesting areas to be selected for examination. A further advantage when preparing steel or other ferromagnetic

specimens with the lift-out technique is that only a small piece of material is subjected to the strong objective lens field of the TEM, and hence the forces acting on the specimen are negligible compared to traditional foil or cross-sectional specimens. The absence of strong fields from the specimen also means that the alignment of the TEM, and in particular the astigmatism, is not drastically affected when the sample and/or beam is moved, again in contrast to larger foil specimens.

Recently there has been interest in combining both FIB and argon ion milling techniques. The FIB is used to select and isolate a suitable specimen from a sample, and then argon ion milling is used to remove the damage layers caused by the FIB. This process has the potential to combine the precise site selectivity of the FIB with the detailed structural and chemical analysis possible with thin, low-damage specimens, and has been successfully applied to various material systems [5]. However, the resulting specimens still have a damage layer caused by the argon ion milling, despite the low milling angles achievable using this method. This suggests that lower energy argon ion milling is desirable.

It is normally not possible to reduce the accelerating voltage of a conventional argon ion mill without causing the beam current to drop to impracticably low levels. Several manufacturers have now designed ion guns specifically for use at low accelerating voltages [6]. We have used one such system, the GentleMill ion beam thinner [7], to thin a variety of technologically relevant materials, including steels, GaAs-based devices and silicon-based devices. By using this system, a low energy ion beam can remove most of the specimen surface damage caused by previous ion milling steps, whether these be high energy argon ion milling or FIB milling [8].

2. Specimen preparation methods

The GentleMill consists of a computer-controlled oil-free vacuum system, sample stage mounted on a 2-axis goniometer, sample-loading airlock, sample imaging video camera and fixed argon ion gun. The ion gun is designed to give a useful ion beam current from ~200 V to 2 kV, and thus can be used for 'conventional' ion milling as well as low-energy polishing. Samples are held in a titanium annulus with a sprung bayonet locking plate. This arrangement gives good mechanical stability and thermal contact with the specimen disc. One disadvantage is that the minimum tilt angle is restricted for thicker dimpled samples. For example, samples with edge thickness 120 μm are restricted to about 8°, as below this value the holder shadows the center of the specimen. With 50 μm thick specimens the minimum angle is reduced to ~4°.

Specimens are prepared using a variety of routes. Planar and cross-sectional specimens are initially mechanically thinned to ~120 μm , and then dimpled from both sides to give a ~10 μm thick section in the center of the specimen disc. Thinning to electron transparency is then performed either using the GentleMill (typically using a 1.5 kV beam at 8° incidence angle) or using a Gatan model 691 PIPS (using 4 kV beams at 4°). The specimens are then transferred to the GentleMill (if required) and polished on both sides using beams of 200-400 V. Polishing is usually done with a tilt angle increased by 1-2° over that used for the previous stages.

FIB specimens were initially prepared by the conventional trench lift-out method [9]. To mount the lamella for further ion milling a copper grid was cut in half. The lamella (approx 20x5x0.2 μm) was then mounted onto a bar in the middle of the cut edge of the grid. To hold the lamella in place the FIB was used to deposit platinum over the end of the lamella resting on the grid bar, thereby welding it in place. Specimens prepared in this way were then ion milled in both PIPS (2 kV, 3°) and GentleMill (500 V, 15°) systems. The GentleMill is usually preferred for this purpose, as the lower ion energies used reduce the sputtering rates and therefore make the thinning process more controllable. This is important with the small area of interest being thinned. Initially the process of lifting out was done using an optical microscope and micromanipulator. The grid and lamella were then transferred back into the FIB for welding, prior to argon ion milling.

The specimens were analyzed in a Tecnai F20 FEG TEM. Available analysis capabilities on this instrument include STEM imaging with bright field, dark field and high angle annular dark field (HAADF) detectors. EELS spectrometry is provided using a Gatan Enfina 1000 spectrometer, and EXD spectrometry is also available.

3. Results

Low energy argon ion milling has previously been shown to reduce surface damage of silicon and GaAs-based samples [8]. We have found that cross-sectional specimens prepared from silicon and GaAs-based hetrostructures using low energy ion milling show marked improvements when polished at low kV. In addition, the broader beam of the GentleMill compared to the PIPS tends to yield larger electron transparent areas with good thickness uniformity, which is advantageous for Z-contrast and EELS analysis. As an example, figure 1 shows a thickness map of a planar silicon sample prepared using the GentleMill.

Several samples from a variety of processed steels were prepared using the FIB lift-out method, followed by thinning using low energy ions in a GentleMill. An example of a FIB lamellae welded on to a copper support grid using in-situ platinum deposition is shown in figure 2. After low energy ion milling some areas of the lamellae were thinned sufficiently for HRTEM examination. Figure 3 is an example of a specimen thinned using the FIB only, and it is obvious that the surfaces have been badly damaged, making any attempt at high resolution analysis a non-trivial challenge. By contrast, figure 4 shows an example of an area thinned by the argon ion beam. Analysis of these areas reveals that there is little amorphous material present, thus enabling the lattice structure of the material to be seen clearly (figure 5). Having such high-quality thinned areas available allows sensitive elemental analysis to be performed with high spatial resolution using EELS. An example of this is illustrated in figure 6, where an EELS spectrum image of the vanadium $L_{2,3}$ core loss edge was used to map the distribution of vanadium precipitates in the steel. In this case particles down to 2 nm in size are visible.

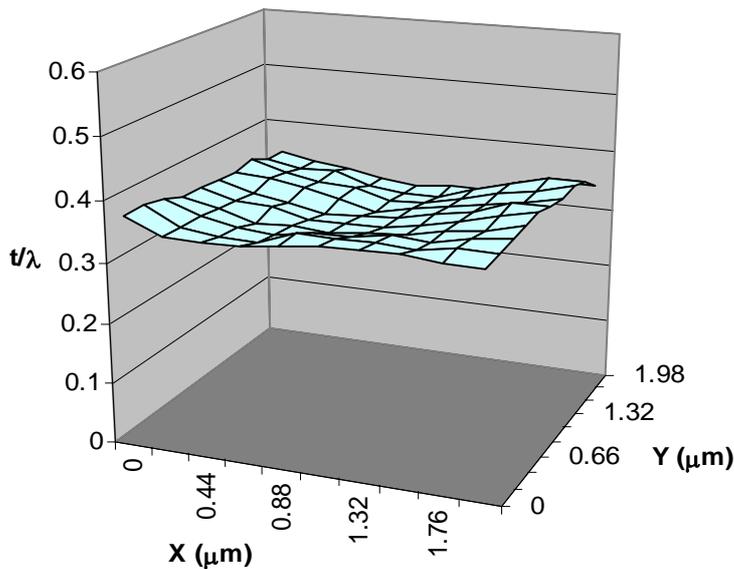


Figure 1. Thickness map calculated from EELS low-loss spectrum image taken over a 2 μm square area of a planar silicon specimen thinned using a GentleMill.

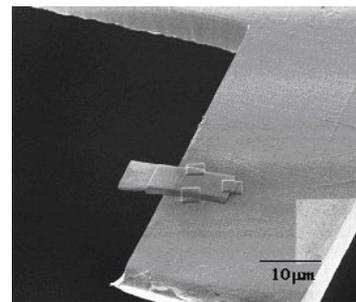


Figure 2. FIB image of a FIB lamella from steel sample.

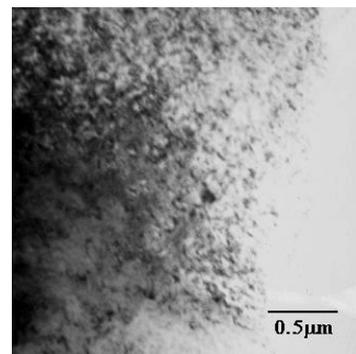


Figure 3. TEM micrograph of steel FIB lamella thinned using FIB only.

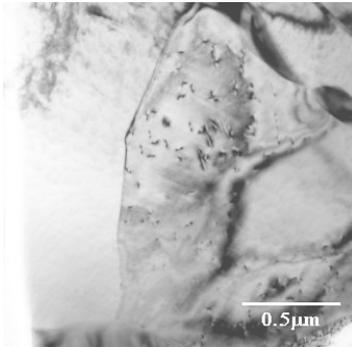


Figure 4. TEM micrograph of steel FIB lamella as shown in Figure 2, after thinning using low energy argon ions in GentleMill.

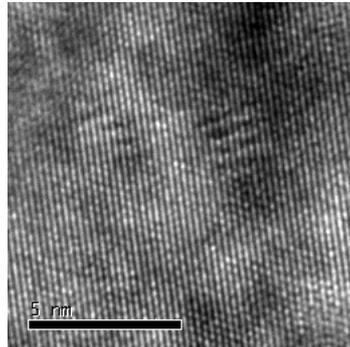


Figure 5. HRTEM micrograph of a steel FIB lamella, illustrating the clarity and resolution of imaging achievable.

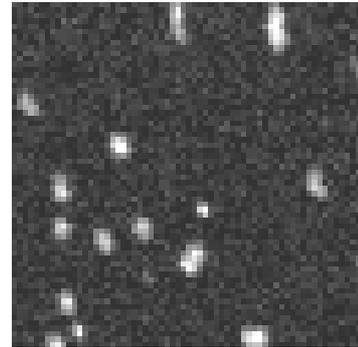


Figure 6. EELS core loss map (64 nm square) of FIB lamella showing Vanadium core loss signal. The smallest precipitates are ~2 nm in size.

4. Conclusions

Low energy ion milling has already been shown to improve the quality of TEM specimens, especially with regard to surface damage and uniformity. Use of a FIB to create TEM specimens is a powerful technique when sub-micron site specificity is required. The combination of these methods can take advantage of the strengths of both, allowing analysis of specimens taken from very specific sample areas. This capability will be extremely useful, not only for analysis of material samples as shown here, but also for present and future semiconductor devices of ever-decreasing size.

Acknowledgements

The authors would like to thank the staff at Technoorg Linda, Budapest for their assistance in preparing many of the samples used in this study. This work was performed with funding from the EPSRC.

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