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Sample preparation by focused ion beam micromachining for transmission electron microscopy imaging in front-view

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ABSTRACT

This article deals with the development of an original sample preparation method for transmission electron microscopy (TEM) using focused ion beam (FIB) micromachining. The described method rests on the use of a removable protective shield to prevent the damaging of the sample surface during the FIB lamellae micromachining. It enables the production of thin TEM specimens that are suitable for plan view TEM imaging and analysis of the sample surface, without the deposition of a capping layer. This method is applied to an indented silicon carbide sample for which TEM analyses are presented to illustrate the potentiality of this sample preparation method.

1. Introduction

Analyses by transmission electron microscopy (TEM) are performed on thin specimens transparent to electrons. This implies in most cases to carry out a sample preparation prior to the specimen observation. The care paid to this crucial step directly affects the results obtained by TEM analysis. Numerous methods were developed and adapted depending on the nature of the material and on the required information (Thompson-Russel and Edington, 1977). For analyzing self-supporting specimens, observations are mostly achieved in cross-section, *i.e.* the electron beam being parallel to the sample surface (Bravman and Sinclair, 1984). This configuration is notably suited to the characterization of superficial nanostructures such as deposited layers onto a substrate and to study the interfaces. Various preparation methods, based either on mechanical polishing techniques and light-ion milling, can be used to obtain cross-sectional TEM samples (Goodhew, 1985). These methods have been widely applied and optimized for various types of inorganic materials and are now well established.

In other cases, plan-view observations (*i.e.* the electron beam perpendicular to the sample surface) are required to obtain structural or morphological information out of reach using cross-sectional samples. For instance, statistics about grain shapes, sizes and orientations in textured films, the characterization of compositional or structural homogeneities at the sample surface, or the study of elementary plastic deformation events produced by nano-indentation, may necessitate plan-view observations to be done. For all the previous examples, it is clear that the sample preparation must be fulfilled in such a way that the surface is kept intact and the superficial area of the specimen is prevented from any damage. To do this, the most common method consists in firstly mechanically thinning the back side of the specimen until the thickness becomes less than a few tens of microns, followed by an additional thinning method to reach the electron transparency. This can be done by chemical or electrolytic thinning provided the interested face is covered with a protective varnish, or by ion milling using dedicated devices.

The classical techniques for preparing TEM samples described before usually provide lamellas offering characteristics satisfying requirements for TEM observations but they are not suitable for the analysis of a specific micrometric area in the specimen. The need to prepare TEM samples in precise areas or containing nanometric objects led to the development of the focused ion beam (FIB) tools and next the combined focused ion-beam/scanning electron microscope (FIB/SEM) systems which allow to make the TEM lamellae on a localized zone with a spatial accuracy of about ten nanometers (Walker et al., 1995). In most cases, the TEM lamellae preparation consists to mill a thin slice of the material perpendicularly to the sample surface, to glue it on a TEM half-grid and to finish the thinning using low-voltage focused ion-beams at grazing incidence (Giannuzzi et al., 1998; Giannuzzi and Stevie, 1999). This technique, called the lift-out method, is then appropriated for cross-sectional

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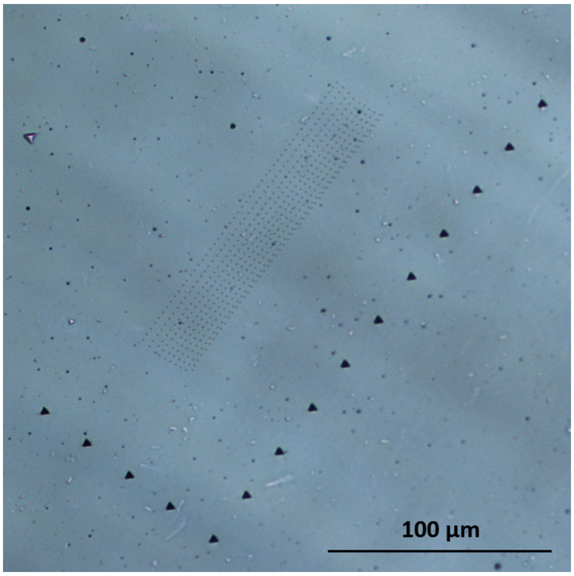

Fig. 1. Optical microscopy image showing the area of interest (in the center of the image) constituted of an array of nano-indents at the sample surface, surrounded by micro-indents forming a L-shaped pattern.

observations of the specimen. For preparing plan-view samples, an intermediate step is required. The sample surface is first covered with a sacrificial protective layer of platinum, tungsten or other weighty metal in order to protect it from deep damaging due to ion irradiation during micromachining. A large chunk of material is then cut by ion-beam milling; this wedge is taken off from the specimen and next rotated by 90° before being fixed onto the TEM grid. Both sides of the plan-view specimens are then milled using decreasing ion current like for the cross-section thinning using the lift-out technique. Nevertheless, the surface layer of the specimen cannot be analyzed using this method because the capping layer must be removed during the final thinning process, this later operation necessarily sputtering the superficial layer of the studied specimen.

In this article, we describe a novel original technique that makes use on a standard FIB/SEM device to prepare a plan-view TEM lamellae from the sample surface of a specific area without damaging of this surface. Inspired by the previously described method, it rests in covering the surface site with a removable piece of material in place of the usual protective layer. In this article, this method is applied to the preparation of a plan-view TEM lamellae from an indented area of a (11-20)4H-SiC wafer. *Post-mortem* TEM analysis of the microstructure of defects which extend from the nano-indents has been successfully carried-out using this front-view (FV) FIB sample geometry.

2. FIB lamellae preparation method for front view observations

The dual beam FIB/SEM system used is a FEI Helios 600 NanoLab microscope, equipped with a gallium ion source operating in the accelerating-voltage range 0.5–30 kV and an omniprobe™ micromanipulator. The preparation technique is demonstrated on a (11-20)4H-SiC wafer patterned with nano-indents of various loads. The indented surface covers 26 μm × 148 μm, the smallest nano-indents being roughly spaced of 2 μm each other, with an edge size of 1 μm (see Fig. 1).

The FV-FIB preparation is composed of 4 steps: (i) the deposit of the protective material onto the specimen surface in the selected area, (ii) the extraction and rotation of the wedge containing the selected area, (iii) the removal of the protective material and (iv) the final cleaning step. First, during the FIB micromachining, the surface of the selected area has to be protected from ion impacts and from the redeposit of sputtered material. For this, a wedge exhibiting a flat face is taken from an irrelevant zone of the specimen or from another sample to be used as a protective cap of the specimen. After extraction, the wedge is rotated of 180° and its flat side is next placed on the selected area as shown in Fig. 2a. The 180° rotation is done by fixing the extracted pyramidal wedge to the tip of a needle parallel to the working plane, rotate the needle of 180° and get back the wedge with the omniprobe™ micromanipulator onto the selected area of the specimen surface.

The left and right sides of the piece are fixed to the sample using *in situ* platinum injection. Note that the lateral sides of the protective cap are preferred for fixing it on the specimen surface to its top and bottom sides in order to facilitate its later removal (step 3). In the work presented here, the surface of the protective cap presents a square-base pyramidal shape having a basal surface of 13 μm × 13 μm. After fixing the protective cap on the selected area, two 0.9 μm height lines of platinum are deposited on the sample, as indicated by the arrows in Fig. 2b. They are disposed closely to the protective cap, about 0.5 μm apart from its bottom and top edges. The deposited lines protect the capped surface avoiding both redepositing sputtered material on it and damaging the surface during ion imaging. In addition, they also prevent the lamellae to bend after thinning. Moreover the two platinum lines allow to reduce the redepositing of sputtered matter between the cap and the sample surface during the micromachining steps. Second, a new wedge is milled from the specimen covered by the protective cap, as seen in Fig. 2c. It is done in two steps with an U-shaped ionic etching, tilted of 52° with the normal surface. Alike the protective cap, the obtained pyramidal-shaped sample is extracted from the specimen after cutting and is next rotated in the FIB chamber using the omniprobe™ micromanipulator, the rotation angle being 90° at this step. The new wedge is then fixed to a lift-out copper grid thanks to a platinum welding. Fig. 2d shows the wedge fixed to the grid with the surface perpendicular to the image plane. The area of interest is located between the arrows, the top and bottom pieces corresponding respectively to the sample and the protective cap.

When the sample is fixed on the lift-out copper grid, the non-protected side of the wedge is milled using large beam currents (0.9–2.8 nA), as shown in Fig. 3a. A thickness of 1 μm is kept to avoid a strong bending of the sample during the next step which consists in removing the protective cap. For this, the platinum welding used to fix the cap onto the sample is first smoothly milled using a 48 pA ion beam. The two removed fixing points are identified in Fig. 3a by arrows. The micromanipulator needle is then glued to the capping piece and moved away to free the sample surface. This one is imaged in Fig. 3b with a tilt angle of 52°. On this figure, it is seen that the indents are prevented of damage or redepositing of sputtered material. This step of the FV-FIB lamellae preparation method is somehow critical as, unless special care is taken, sputtered material may redeposit on both edges of the protective cap and may glue it to the sample. It must be noticed that in this case, the sputtered material redeposited on the top and bottom sides of the cap could not be ion milled at this step, without complicated translations and rotations of the lift-out copper grid.

The last step of the FV-FIB lamellae preparation corresponds to the final thinning and cleaning of the sample. The technique commonly used for classical FIB lamellas consists in scanning the lateral face of the lamellae using a low-current beam (Kato, 2004). In the specific case of FV-FIB lamellae preparation, the only difference is that the thinning must be performed only on the sample side opposite to the surface of interest. In the example shown here, the low-current thinning process is continued until the nano-indents appear across the FV-FIB lamellae when imaged with electrons