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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.

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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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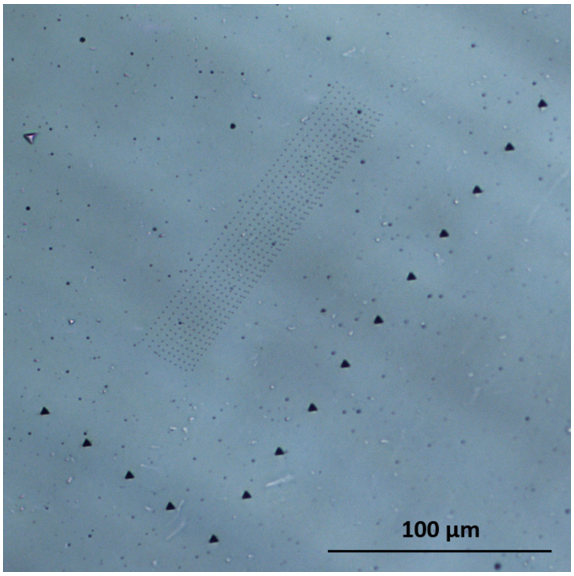

Fig. 1. Optical microscopy image showing the area of interest (in the center of the image) constituted of an array of nano-indents at the sample surface, surrounded by micro-indents forming a L-shaped pattern.

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^{TM} 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^{TM} 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^{TM} 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

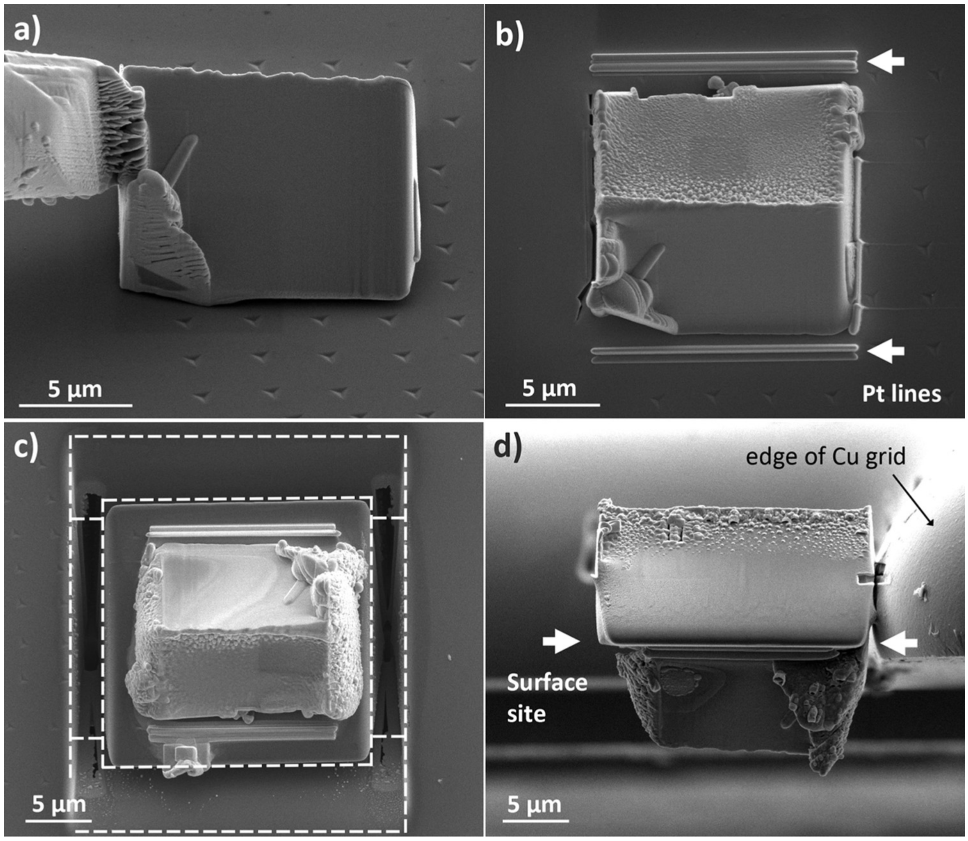

Fig. 2. SEM images showing the first steps of preparation of a FV-FIB lamellae: (a) the protective cap is deposited onto the sample surface, (b) the platinum lines are deposited on the sample surface, on both sides of the protective cap, (c) cutting of the protected sample; the two U-shaped cutting pattern are marked, and (d) the sample wedge is fixed on the TEM grid.

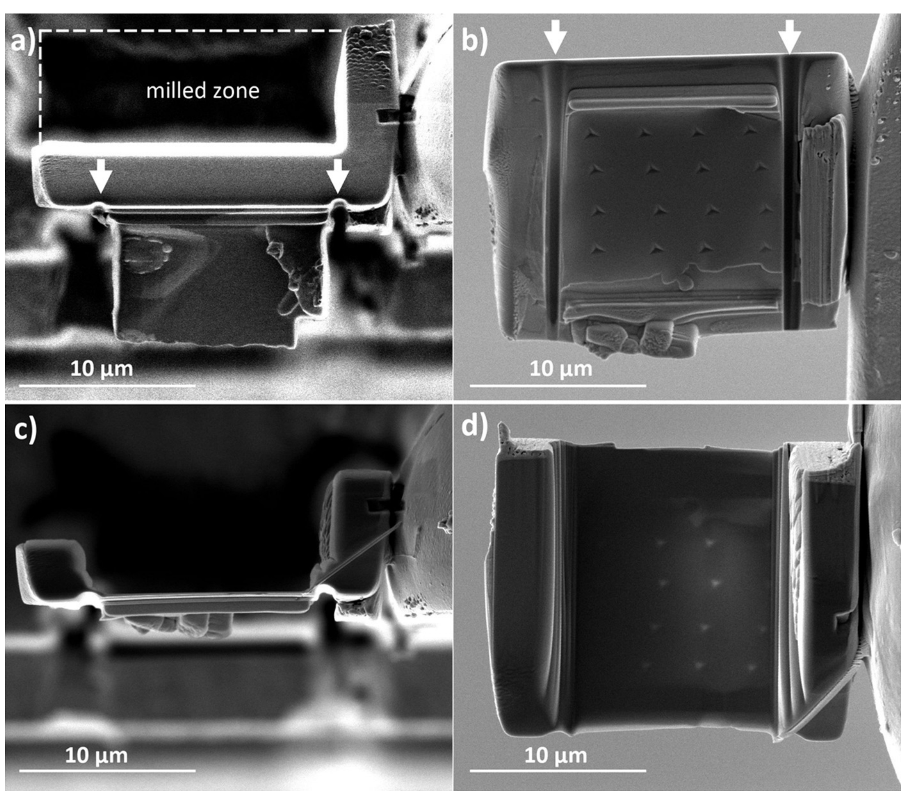

Fig. 3. SEM images showing the FV-FIB lamellae during the thinning process: the initial thick lamellae is observed: (a) edge-on and (b) tilted of 52°, without its cap. The thinned lamellae is observed: (c) edge on and (d) tilted of 57°.

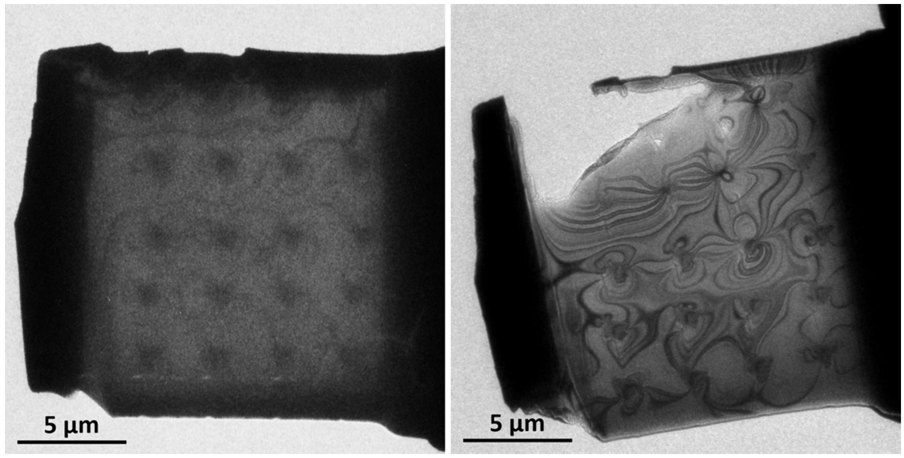

Fig. 4. TEM micrographs showing the FV-FIB lamellae before (left) and after (right) the post-FIB Ar^+ ion milling process using the Fischione Nanomill device.

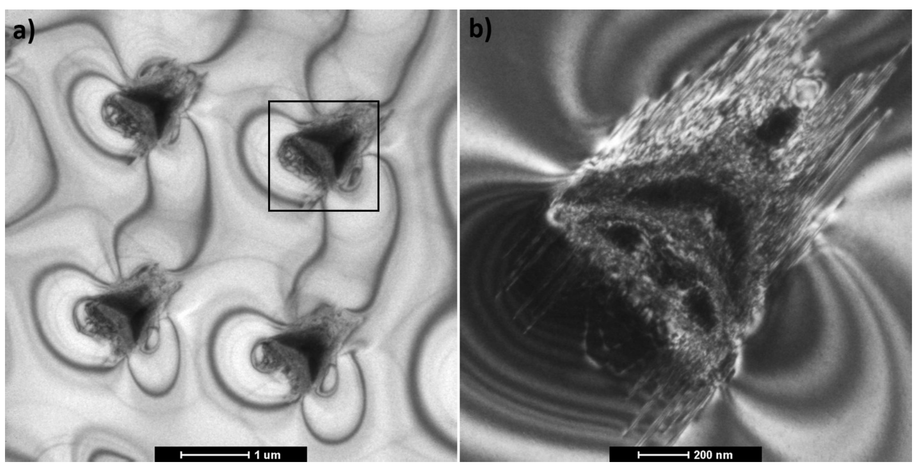

Fig. 5. (a) Bright-field TEM micrograph of a thin area of the sample, containing four nano-indents, observed in front-view. (b) Dark field TEM image of the nano-indent framed by a black square in (a). Numerous dislocations are clearly visible close to the imprint.

accelerated at 5 kV. The obtained lamella was then about 100 nm thick. As far as the thickness decreased, the ion beam current was progressively decreased, from 920 pA to 48 pA. The line scan thinning is ended using an accelerating voltage of 5 kV and a cleaning step is done using a 1 kV ion beam. Fig. 3c and d shows respectively a SEM image of the final FV-FIB specimen seen edge on and a 57° tilted SEM image of the same specimen. The lamellae presents a thin surface area of 11 μm × 12 μm wide containing a few indents.

3. Front view TEM observations of the nano-indented area

In spite of the high internal stresses of the specimen accumulated in the nano-indented areas, the thinnest part of the FV-FIB lamellae remains straight after thinning, as seen in Fig. 3c. This observation demonstrates the great influence of the platinum lines deposited during the preparation process on the stiffness of the FV-FIB lamellae. Nevertheless, even for unstressed specimens, a platinum line support is also necessary to thin lamellas until 50 nm. This precaution is notably essential for ductile materials as metals, III–V semiconducting compounds, *etc*.

In the case of the example presented in this article, the final back-side thinning and cleaning of the FV-FIB lamellae was carried out by Ar^+ ion milling at very low voltages (down to 100 V) using the Fischione model 1040 Nanomill system. This additional thinning step allowed us to slightly reduce the thickness of the sample (see Fig. 4) and also to remove the residual superficial amorphous layer produced by the Ga^+ ion milling during the FIB micromachining. Finally, the obtained FV-FIB lamellae contains very thin areas (<20 nm thick) suitable for both conventional and high-resolution TEM imaging. However, as shown in Fig. 5a, TEM observations

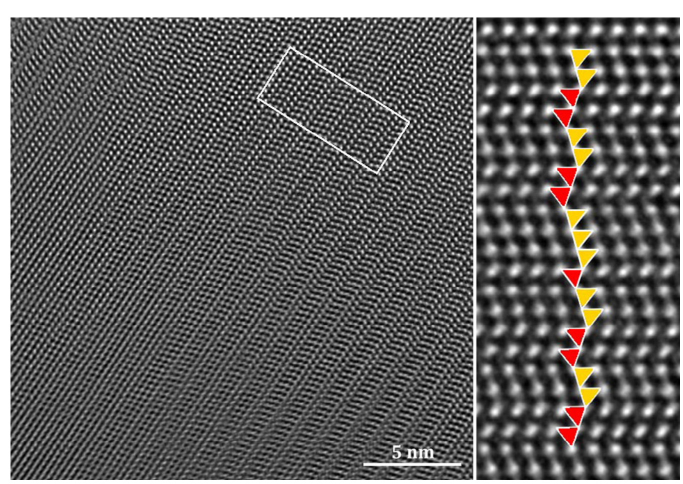

Fig. 6. High-resolution TEM image (left) and magnified framed selection (right) performed in the vicinity of an indented zone. The presence of a stacking fault is evidenced by the modification of the 4H-SiC stacking sequence.

demonstrate the very good thickness homogeneity of the sample over wide areas, even for FIB lamellae having such a low thickness.

The observed bend contours bear witness of the very high stresses accumulated around the nano-imprints and clearly show that the sample preparation method does not release these stresses. In addition, the dislocations produced by nanoindentation, known to be weakly mobile at low temperatures in silicon carbide (Pirouz et al., 2003; Lancin et al., 2009), are distinguishable around the imprint in Fig. 5b. Their observation indicates that the indented surface has not been eroded by the ion beam micromachining of the FV-FIB lamellae.

High-resolution TEM observations have also been performed in the thinnest areas of the FV-FIB lamellae (*cf.* Fig. 6). A detailed analysis of the observed defects has been carried out and can be found elsewhere (Texier et al., 2013).

4. Conclusion

The sample preparation method described here was successfully applied for studying the deformation microstructure of a nano-indented 4H-SiC wafer. This work demonstrates the high efficiency of FIB micromachining for TEM sample preparation even for front-view observations, provided the preparation method is properly adapted to the experimental constraints imposed by the targeted analysis.

The most difficult parts of the preparation method presented here are the steps 1 and 3, which correspond to the cap manipulation. The flat surface has to be snugly fixed to the sample surface, and the detachment procedure depends on the cap takeoff and the micromanipulator handling. Moreover, the achievement and the manipulation of this piece is time consuming. For instance, the 180° rotation requires to open the FIB chamber and to manually turn the needle support. The steps 2 and 4 are common to the classic “microsampling technique”. Nevertheless they take more time than a classic plan-view specimen preparation because of the larger size of the needed wedge base. To make the front-view specimen described in this demonstration, 5 h was nearly used. The time required may be reduced of 1 or 2 h if the sample dimensions are smaller and the material is softer.

This FIB micromachining method results in TEM front-view lamellae of a surface avoided of ion impacts and contamination. The key point is a full protection *via* the use of both a temporary cap and platinum lines around. The matchless advantages of this technique over the mechanical and electrolytic techniques are that the plan-view specimen can be prepared from a specific site and that the surrounding surface is left intact. This can be opportunely applied to the micro or nanoscopic characterization of materials for studying the surface changes resulting from implantation, etching, materials deposition or indentation experiments for instance.

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