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Evidence of perfect dislocation glide in nanoindented 4H-SiC

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Abstract. Plastic deformation of a 4H-SiC wafer has been produced by nanoindentation at room temperature. The superficial layer of the specimen in the indented area has been lifted out thanks to a specific focussed ion beam micromachining method and the deformation microstructure close to the imprints has been investigated by means of both conventional and high-resolution transmission electron microscopy (TEM). The analysis of the images revealed the presence of various types of extended defects. Perfect dislocations and single stacking faults bounded by isolated Shockley partial dislocations have been observed on the basal plane. Perfect dislocations have also been evidenced out of the basal plane. These results highlight the competition between various activated systems involved during the plastic deformation of 4H-SiC in the brittle regime.

1. Introduction

Plastic deformation in covalent semiconducting materials is known to proceed by the motion of dissociated dislocations in the ductile regime, but very little is known about the nature of gliding dislocations in the brittle domain, particularly at low temperatures. In silicon, both calculations and experiments indicate that mobile dislocations below the brittle-to-ductile transition temperature are likely perfect dislocations gliding in the shuffle set. In 6H and 4H-SiC, deformation experiments performed above the brittle-to-ductile transition and microstructure analyses suggest that plastic deformation involves weakly-dissociated dislocations [1-2] whereas Shockley partial dislocations were evidenced in 4H-SiC deformed in the brittle regime [3-7,9]. However, some studies have also reported the presence of perfect or weakly-dissociated dislocations in prismatic and basal planes in 4H-SiC single crystals deformed in the brittle regime by microindentation [8] or under confining pressure [9-12].

In this work, a (11-20)4H-SiC wafer was deformed by nanoindenting the surface at room temperature. TEM thin foils were then prepared by dual-beam focused ion beam (DBFIB) micromachining parallel to the indented surface in order to perform conventional and high-resolution transmission electron microscopy imaging of the deformation microstructure viewed in plan view. The TEM observations revealed various types of defects in the investigated areas.



2. Experimental details

Plastic deformation of 4H-SiC wafer was achieved by nanoindenting the $(11\bar{2}0)$ surface. A periodic array of nano-imprints was performed in force-controlled mode using the CSM Instruments model NHT device equipped with a Berkovich indenter. The applied force was fixed to 15 mN leading to an average penetration depth (elastic plus plastic) of about 150 nm. After unloading, the dimensions of the residual nano-imprints were estimated to 60 nm in depth and 420 nm in lateral size. In order to facilitate the localisation of the nano-imprints, micro-indentations were performed at the vicinity of the nano-indented area.

Samples for TEM imaging in plan view were prepared by focussed ion beam (FIB) micromachining using an original method specifically developed for this study. The principle of this sample preparation method rests on the use of a Si shield fixed onto the sample surface during the micromachining process. This precaution permits to protect the superficial layer of the specimen during the ion beam milling and to obtain a thin lamellae suitable for TEM analyses in plan view, *i.e.* suitable for studying the microstructure just below the surface. Details of the method will be shortly described [13]. Final back-side thinning and cleaning of the FIB lamellae was carried out by Ar^+ ion milling at very low voltage (down to 100 V) using the Fischione model 1040 Nanomill system, allowing to investigate the deformation microstructure around the nano-imprints by TEM imaging.

C-TEM and HRTEM observations were respectively performed using a FEI Tecnai G2 microscope operating at 200 kV and a FEI Titan 80-300 Cs-corrected microscope operating at 300 kV.

3. Results and discussion

Plan view C-TEM observations performed in the thickest areas of the FIB lamellae allowed us to get an overview of the deformation microstructure around the nano-indentations. Characterization of the observed defects by the classical method based on their stereographic analysis and the use of extinction conditions has not been carried out on account of the very strong bending of the lamellae in the vicinity of the nano-indentations. However, different types of extended defects can be distinguished in dark-field images, as shown in figure 1. On the one hand, numerous straight lines are observed perpendicular to one of the three facets of the imprint. The direction parallel to these defects is the $[1\bar{1}00]$ direction, indicating that these defects extend in the (0001) planes, which are seen edge-on for this observation orientation. On the other hand, dislocation half loops out of the basal plane are also visible (see figure 2). It must be underlined that the observed dislocation loops do not drag planar defects indicating that they consist of perfect non-dissociated (or weakly-dissociated) dislocations, in agreement with previous observations reported in [4] where perfect dislocations have been identified in prismatic planes in 4H-SiC micro-indentation below the brittle-to-ductile transition temperature. In addition, the observed dislocation loops are very faceted; one of the segments of the largest loop shown in figure 2, parallel to the $[0001]$ direction, exhibits a very large kink suggesting that the growth of this loop preferentially proceeded by the nucleation and propagation of kinks along this direction.

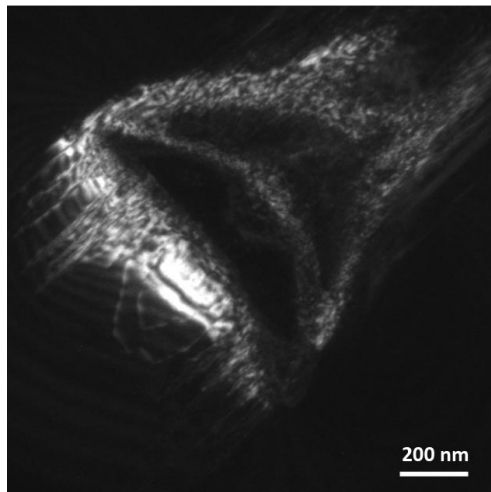


Figure 1. Dark-field TEM image of a nano-imprint seen in front view. Numerous extended defects parallel to the $[1\bar{1}00]$ direction are visible.

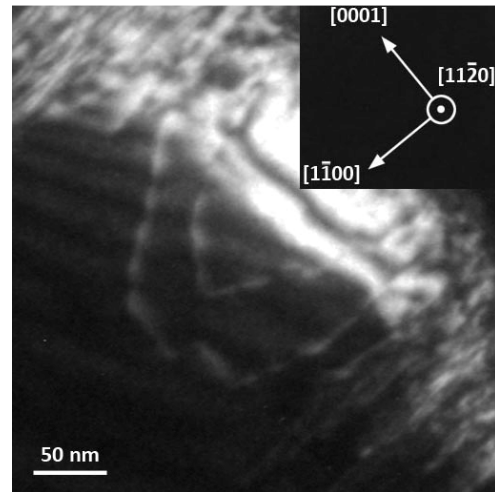


Figure 2. Detail of the dark field image shown in figure 1. Two perfect dislocation loops extend out of the basal plane which is seen edge on for this crystal orientation.

HRTEM analyses have also been performed in very thin areas of the sample, close to the nano-indent. The HRTEM images and the associated strain mapping obtained by the geometrical phase analysis (GPA) method applied to the experimental images, revealed the presence of many isolated stacking faults (SFs) extended in the basal plane, as shown in figure 3. A careful examination of the observed stacking disorder introduced in the 4H-SiC structure (see for instance the disrupted sequence of the C-centred tetrahedra superimposed to the image in figure 3) indicates that these defects systematically correspond to single SFs. It must be underlined that multiple SFs have not been identified in this study, contrary to previous studies performed in 4H-SiC deformed in the brittle regime [6-9].

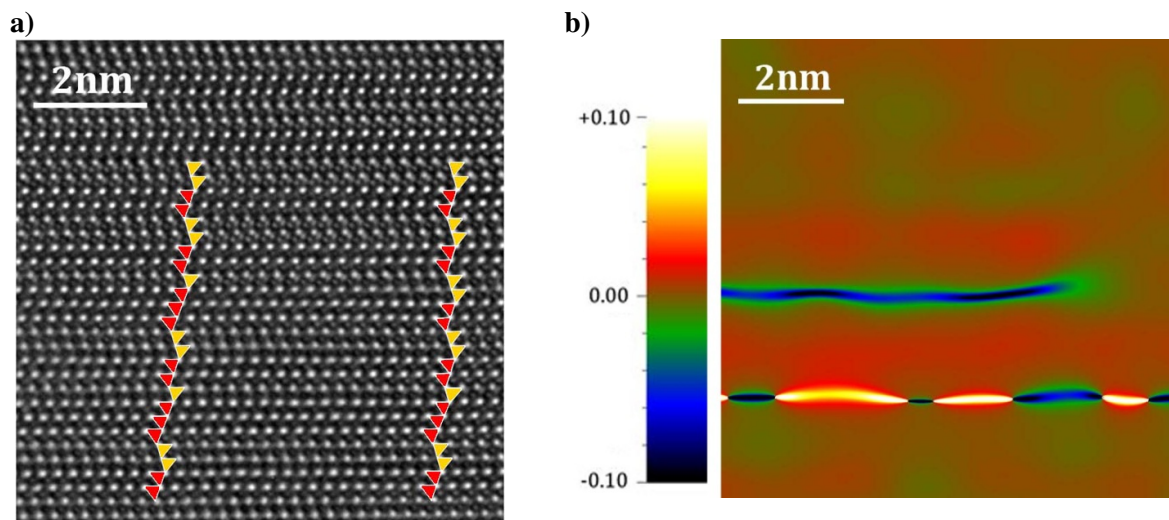


Figure 3. a) HRTEM image of the area close to a nano-indent. Two single SFs are visible in this image. One can note that the comparison between the stacking sequences at the left and right sides of the image evidenced the presence of a Shockley partial dislocation in the field of view of the image. b) Shear map (ϵ_{xy}) measured using the GPA method ($e_x = [1\bar{1}00]$, $e_y = [0004]$) in the same area.

In addition, the presence of non-dissociated perfect dislocation in the basal plane was also revealed by HRTEM imaging. The structure shown in figure 4 contains a dislocation parallel to the observation direction which can clearly be evidenced thanks to the GPA strain mapping. In order to unambiguously identify the character of this dislocation, multislice simulations based on elastic calculations (isotropic approximation) for various dislocation types have been compared with the experimental image. From these simulations, this dislocation has been identified as being a 60° perfect dislocation.

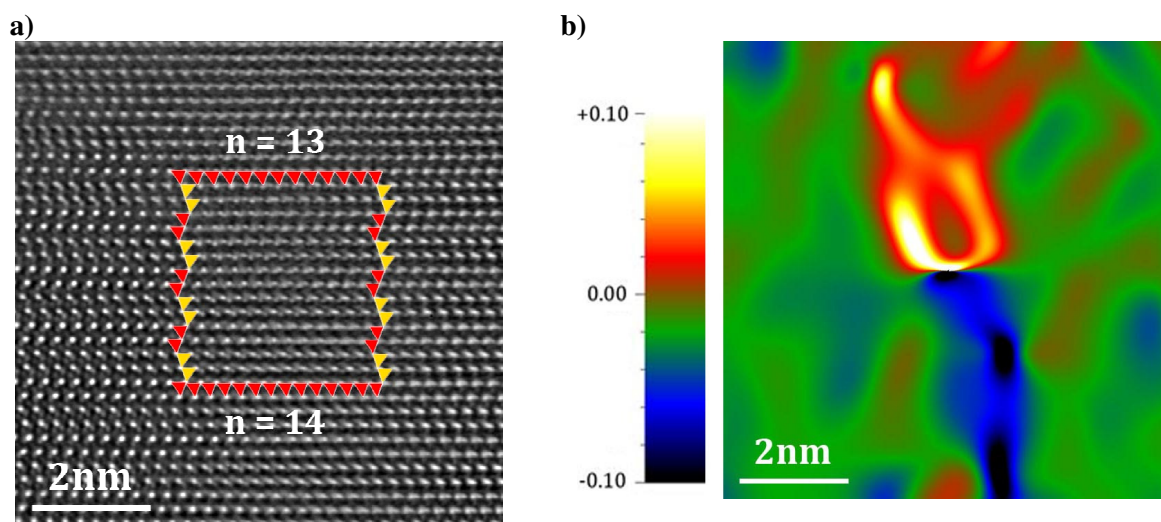


Figure 4. a) HRTEM image of a perfect 60° dislocation. b) Corresponding strain map ($\epsilon_{1\bar{1}00}$) measured using the GPA method.

4. Conclusion

Defects produced by nanoindentation of 4H-SiC at room temperature have been studied by TEM imaging. This work evidenced the concomitant nucleation and propagation of various types of defects for the low-temperature plastic deformation of this material, in agreement with [9]. Isolated Shockley partial dislocations and resulting single SFs have been frequently observed in the basal plane. Perfect dislocations have been identified in and out of the basal plane. These observations highlight the possible motion of perfect dislocations in the shuffle set in the brittle regime.

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