AMTC Letters
Vol. 2 (2010)

Optimisation of the TEM Sample Preparation from Diamond Using Focused Ion Beam Technique

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The progress in fabrication of synthetic diamond results in increasing number of its potential engineering applications. However, due to its extreme properties, it is very hard to prepare TEM sample from diamond using traditional methods of preparation including mechanical thinning, ion milling or chemical etching. At the same time diamond can be relatively easy micro-machined using FIB (focused ion beam) technique. Using this technique cross-sectional TEM sample of diamond can be prepared in a few hrs [1]. However, FIB milling has an unavoidable result in formation of the damage layers [2-3] which can significantly aggravate the quality of high-resolution imaging. In this work the damage layers after 5, 10 and 30 keV FIB milling in the single crystal diamond were studied. Also the method of optimisation of the diamond TEM sample preparation was demonstrated.

Synthetic single crystal (001) diamond samples produced by Sumitomo were used in this study. FEI Nova Nanolab 200 dual beam FIB system was used for both damage formation in diamond and cross-sectional TEM sample preparation. Rectangular trenches 4x4 μm^2 and 2 µm deep were milled using ~100 pA Ga^+ ion beam current at 5, 10 and 30 keV ion energy. After FIB milling the near surface regions of the trenches contain two types of damage: the bottom-wall damage where the Ga beam was normal to the surface and the side-wall damage where the Ga beam was at low angle to the surface of the created trench walls. Next cross-sectional TEM samples were prepared using lift-off technique described elsewhere [1, 4] and were examined using FEI Tecnai TF20 and spherical aberration corrected Titan electron microscopes.

Cross-sectional HREM images of the side-wall damage layers created during 5, 10 and 30 FIB milling are shown on FIG. 1(a-c). There are clearly visible crystalline diamond substrates, Pt containing protective layers and amorphous damage layers in between. The crystalline-amorphous interfaces are rather sharp and uniform. The thickness of the amorphous damage layers was measured to be 3.5, 5 and 16 nm for side-walls and 10.5, 15 and 44 for the bottom-walls. There are also visible the areas with darker contrast which are situated just below amorphous-crystalline interfaces. Despite diamond in these areas contains large number of point defects their density is below amorphisation threshold and material remains crystalline. However, these point defects create the local distortion of the diamond lattice due to small displacement of carbon atoms from their ideal positions in the vicinity to the point defects. Such atom displacement (“crystalline damage”) can significantly aggravate the sharpness of atomic columns on high resolution images.

The TEM sample preparation process was optimised by using 5 keV FIB milling on final stage. High-resolution imaging of the (110) diamond lattice after 30 keV FIB milling and after 5 keV milling are shown on FIG. 3. Reduction of the thickness of the damage layer after 5 keV milling (FIG. 3b) results in the reduction of the background noise on the HREM images. Also the thickness of the area of local distortion of diamond lattice near amorphous-crystalline interface is significantly narrow after 5 keV cleaning. This increases the fraction of the pristine undamaged diamond in the TEM membrane which results in much sharper image of atomic columns on HREM image. This is also confirmed by corresponding FFT images on inserts. On FIG. 3c the higher magnification image of the diamond sample after 5 keV FIB milling is shown. The individual atomic columns with spacing 0.089 nm are clearly visible. The reduction of the damage layer thickness in the TEM diamond samples with 5 keV FIB milling drastically improved the quality of the lattice images and allowed to resolve individual atomic column (diamond dumbbells) with sub-angstrom spacing.

References

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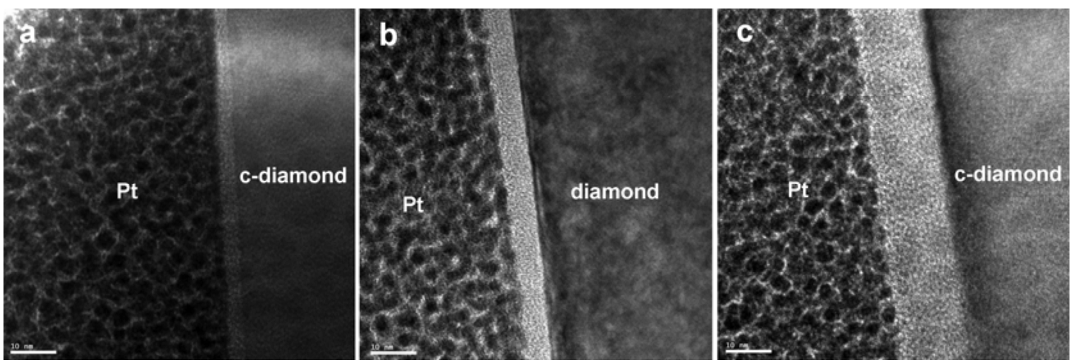

FIG. 1. Cross-sectional images of the side-wall (a-c) damage layers in the diamond samples after FIB milling with 5 (a), 10 (b) and 30 (c) keV Ga^+ ions.

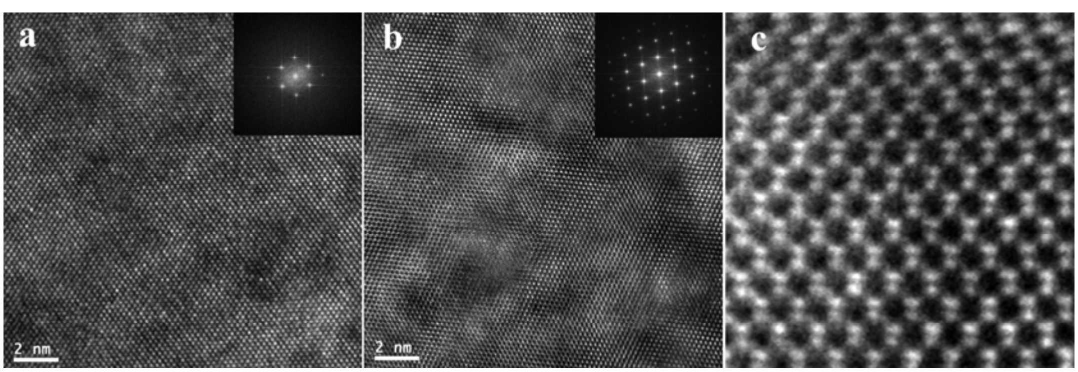

FIG. 2. High-resolution images of (110) diamond lattice after 30 keV FIB sample preparation (a); after using 5 keV FIB milling on final stage (b); magnified image of central area of previous image (c). Corresponding power spectrums are shown on inserts.