Xe⁺ FIB Milling and Measurement of Amorphous Damage in Diamond

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Micro- and nanomachining of diamond using focused ion beam (FIB) continues to generate interest in applications such as diamond anvil cells, photonic devices, micro-cantilevers and tools for imprinting applications [1,2]. However, the milling rate of diamond by FIB is approximate 4X slower when compared to silicon using 30 kV Ga⁺ FIB [3]. Recent instrumentation using PFIB technology and Xe⁺ ions offer increased milling rates because of their ability to deliver up to 30X more current compared to Ga⁺ FIBs. While the sputter rate of diamond using Ga⁺ and Xe⁺ differs only slightly (0.07 µm³/nC [Ga] and 0.09 µm³/nC [Xe]), the ability to use more current for micromachining will allow users to increase throughput significantly. Therefore, it is of interest to understand the amount of amorphous damage introduced into a sidewall of diamond. Previous results indicate that for a glancing angle ~0 degrees, up to 35 nm of amorphous damage is introduced by Ga⁺ FIB in single crystal diamond [4].

Cross-sections of an octahedron, rough-rough cut natural diamond was prepared using the Helios PFIB DualBeam™ using Xe⁺ ions. Specimens were polished with energies of 30 kV, 5 kV, and 2 kV using incident angles of 88.5°, 87° and 87° respectively. After protecting the cross-section surface with 2 keV Pt EBID, conventional in-situ lift-out TEM samples of the milled cross-sections were prepared using a Helios NanoLab™ 460F1 DualBeam equipped with an EasyLift™ nanomanipulator. Amorphous silicon damage was analyzed by HRTEM on a Talos™ F200X TEM operating at 200 keV.

Figure 1 shows HRTEM images of the amorphous sidewall damage in single diamond using Xe⁺ FIB milling with 30 kV, 5 kV and 2 kV, respectively. Experimental results differ from SRIM calculations by as much as 50%; likely due to SRIM’s simulation not taking into account crystal orientation [5]. Table 1 shows amorphous sidewall damage for diamond and silicon (originally reported in 2013) [6]. As expected, decreasing accelerating voltage will decrease amorphous damage.

References:
Figure 1. HRTEM images of sidewall amorphization damage in diamond from a Xe$^+$ PFIB with 30 kV, 5 kV and 2 kV accelerating voltages.

<table>
<thead>
<tr>
<th>Accelerating Voltage of PFIB Xe$^+$ ions (kV)</th>
<th>Target Material</th>
<th>30</th>
<th>5</th>
<th>2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diamond</td>
<td>~9 nm</td>
<td>~6 nm</td>
<td>~4 nm</td>
<td></td>
</tr>
<tr>
<td>Silicon</td>
<td>~13 nm</td>
<td>~4 nm</td>
<td>~2 nm</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. Summary table of sidewall amorphization damage layer thickness (nm) in Diamond and Si after Xe$^+$ milling with 30 kV, 5 kV and 2 kV.