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Protective Carbon Deposition for Superior FIB Prepared (S)TEM Specimens

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Focused ion beam (FIB) assisted chemical vapor deposited (CVD) metal layers such as Pt or W are routinely used to planarize specimens to avoid curtaining artifacts during FIB cross-sectioning for SEM or TEM specimen preparation [1]. Using a DualBeam™ (FIB/SEM) instrument, electron beam assisted deposition (EBAD) layers are often deposited as an alternative to ion beam assisted deposited (IBAD) layers to prevent ion implantation damage to the outer most ~ 50 nm region of the sample [2]. Since the EBAD process takes ~ 20 x longer than the IBAD process, thick (i.e., > ~ 200 nm) EBAD layers are seldom used for the entire protective layer. Thus, most protective layers consist of a deposit of ~ 50 – 200 nm thick EBAD layer followed by an IBAD layer.

The Pt-based GIS precursor is an organometallic-based material, and therefore, neither the IBAD nor the EBAD Pt is pure metal, and nominally consists of a heterogeneous mixture of nanocrystalline (Pt + C) grains [ref. 3 and see FIG. 1]. Excess oxygen may also be observed in the deposit, and of course, Ga is observed in the IBAD coating [3]. A Carbon GIS source is also available for DualBeam deposition use. A protective C layer may be preferred for specimen preparation over the higher atomic number Pt or W, particularly for Z-contrast high angled annular dark field (HAADF) STEM imaging since the low atomic number C layer will not dominate the contrast during analysis. Over the past few years, low energy Ga^+ ion FIB techniques using the DualBeam (FIB/SEM) have been exploited to prepare specimens that are capable of achieving the information limit (i.e., sub-angstrom) of aberration corrected (S)TEM instruments [4,5]. Below, we compare the use of EBAD and IBAD Pt versus C deposition protective layers for high quality (S)TEM specimens.

EBAD/IBAD C and EBAD/IBAD Pt were deposited on Si and FIB prepared for STEM analysis. Each face of the specimen was FIB milled using Ga^+ ions at 30 keV and 88.5 degrees incident angle, followed by 5 keV at 85 degrees incident angle, then 2 keV polishing at 82 degrees incidence angle. FIG. 1 shows 30 keV bright field (BF) STEM images of (left) Pt deposited on Si and (right) C deposited on Si. FIG. 2 shows 30 keV dark field (DF) STEM images of (left) Pt deposited on Si and (right) C deposited on Si. Note that the grain size of the EBAD Pt is 1-3 nm and the IBAD Pt is 10-20 nm. The larger grained IBAD Pt compared to the EBAD Pt is consistent with findings in [3]. In comparison, the EBAD and IBAD C layers yield sub-nanometer grains, with the IBAD C grains slightly larger than the EBAD C grains (see FIG. 2).

TRIM calculations [6] show that C sputters faster than Pt by ~ 24% at the defined conditions at 30 keV, but the difference in sputter yield between C and Pt increases to > 30% at 5 keV. Thus, during the low energy milling steps, the slower milling Pt grains sets up edge effects which manifests as curtaining artifacts observed in the specimen shown in FIG 1 and FIG 2. However, the Si samples protected with the smaller and more homogenous grain sized C deposition layer yield no observable curtaining artifacts in the Si protected by C deposition. Thus, the use of C deposition as a protective layer yields less curtaining FIB milling artifacts than Pt and is a better alternative for high resolution (S)TEM specimen preparation techniques.

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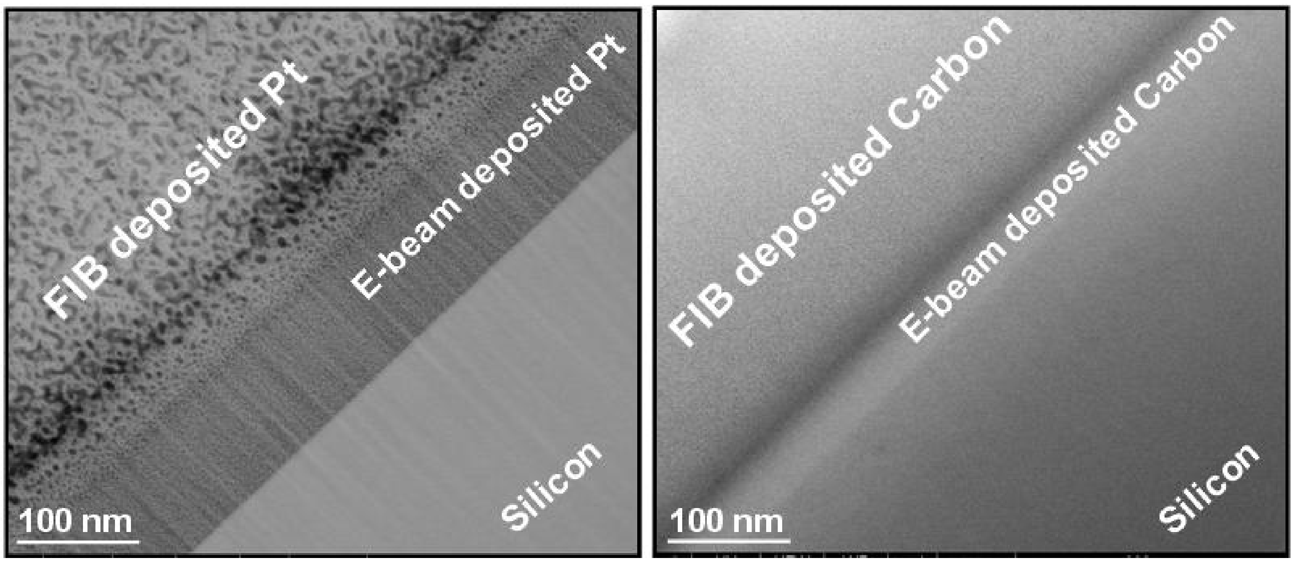

FIG.1. 30 keV bright field STEM images of FIB prepared specimens. The left image shows Pt on Si and the right image shows C deposition on Si.

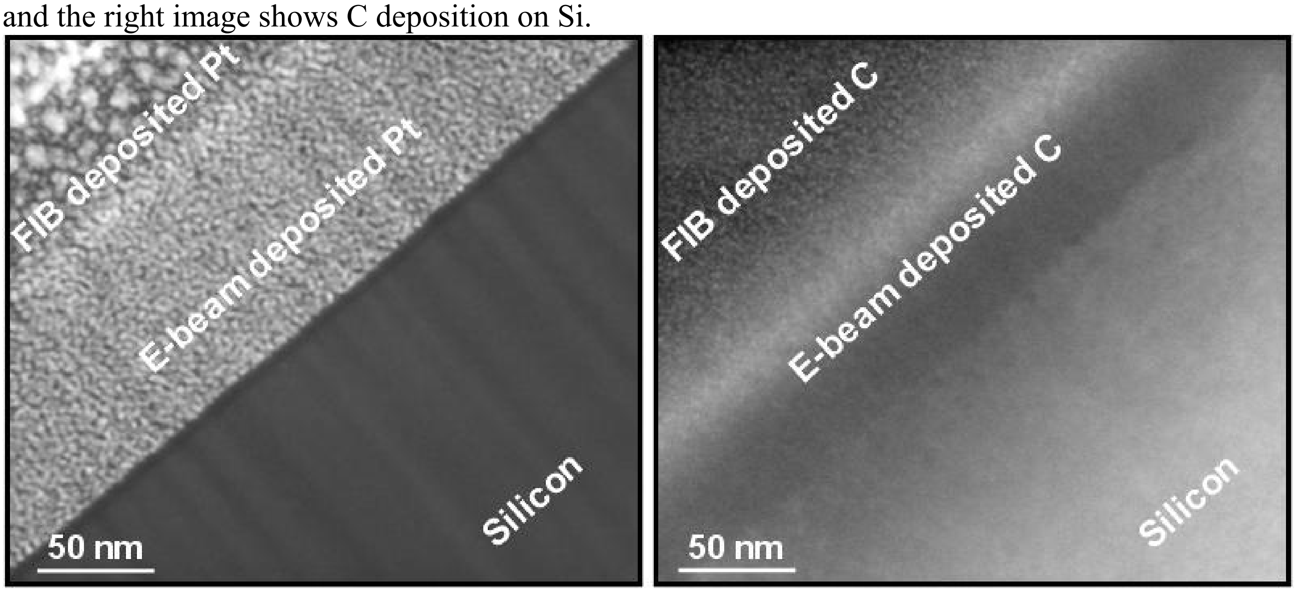

FIG.2. 30 keV dark field STEM images of FIB prepared specimens. The left image shows Pt on Si and the right image shows C deposition on Si.

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