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 outermost ~ 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.
References


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.