Characterization of Xe^+ PFIB Damage in Electron Beam Induced Deposition of Platinum, Tungsten and Carbon Chemistries for In-situ S/TEM Sample Preparation

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TEM/STEM sample preparation by focused ion beam (FIB) and FIB-SEM instrumentation has become routine in the last several years. Technology advances in automation and in-situ techniques have reduced preparation times for sub-50 nm lamellae to less than an hour and with state-of-the-art technology less than 30 minutes [1]. DualBeams (FIB-SEM) are also often used for micro- and nanoprototyping applications. Beam induced, site-specific chemical vapor deposition of materials like Pt, W or C inside the DualBeam has been used to fabricate three-dimensional structures [2]. For S/TEM sample preparation electron or ion beam induced deposition (EBID, IBID) is required to planarize the region of interest to minimize artifacts generated by the FIB [3]. Previous results using a Ga^+ FIB show that FIB damage into EBID of platinum, tungsten and carbon were 48 nm, 15 nm and 34 nm respectively when the ion beam is perpendicular to the EBID surfaces [4]. As plasma FIB-SEM DualBeams become more prevalent in materials research for their ability to perform large volume 3-dimensional characterization, so does the interest in using plasma FIBs for S/TEM sample preparation.

Many studies have investigated surface and sidewall lamella damage in silicon by FIB [5]. In addition to sidewall damage by FIB for cross-sections or FIB processed S/TEM samples, surface damage must also be considered for FIB preparation especially when characterization of the sample surface is required. It has been shown that low energy electron beam induced deposition (EBID) imparts the smallest surface damage when compared with Ga^+ ion beam induced deposition (IBID) [6]. However, the rate of deposition with EBID is ~ 20X slower than IBID, thus understanding the damage depth into EBID layer during the FIB deposition process will reduce process time for cross-section or S/TEM sample preparation.

Pt- and W- based gas injection (GIS) precursors are organometallic compounds (methylcyclopentadienyl trimethyl platinum and tungsten hexacarbonyl respectively). Neither of the deposition products are pure metals. The C-based GIS precursor is naphthalene and likely the purest material when EBID is used. However, for sample preparation, platinum deposition is the least preferred for planarization layer because of heterogeneity (nanocrystalline Pt grains) [7]. Carbon deposition may be preferred over tungsten deposition particularly for Z-contrast high angle annular dark field (HAADF) STEM imaging since the low atomic carbon layer will not dominate the contrast during analysis.

Approximately 100 nm EBID Pt, W and C layers were deposited onto Si using 2 keV; 51 nA (W), 5 keV; 51 nA (Pt) and 1 keV; 6.4 nA. 30 keV Xe^+ IBID Pt, W and C layers were deposited over the layers of interest and FIB prepared for S/TEM analysis. Each face of the lamella was prepared using a Scios DualBeam with Ga^+ ions at 30 keV and 88.5 degrees incident angle. Figures 1a and 1b are 200 keV STEM images of the Pt and C experiments respectively. Images were acquired on a Thermo Scientific Talos F200X. Measurements (Figure 2) reveal that IBID platinum penetrated approximately 16 nm into the EBID platinum while the IBID tungsten penetrated only 1/3 as much into EBID tungsten.

[1] D. Wall, "Ultra-Fast In-Situ S/TEM Sample Preparation." FEI Company Application Note P/N 04AP-FR0111, FEI Company, 2007.
[2] H.W.P. Koops et al., J. Vac. Sci. Technol. B, **13** (1995), p. 2400.
[3] Introduction to Focused Ion Beams, eds. L.A. Giannuzzi and F.A. Stevie, Springer (2005).
[4] B. Van Leer et al., IMC 18 Proceedings, **IT-13-P-2485**, (2014).
[5] L. A. Giannuzzi et al., Micros. Microanal., **11(Suppl 2)** (2005), p. 828.
[6] B.W. Kempshall et al., *J. Vac. Sci. Tech. B*, 20(1) (2002) 286.

[7] B. Van Leer et al., Micros. Microanal., **15(Suppl 2)** (2009), p. 336.

1a. 1b.

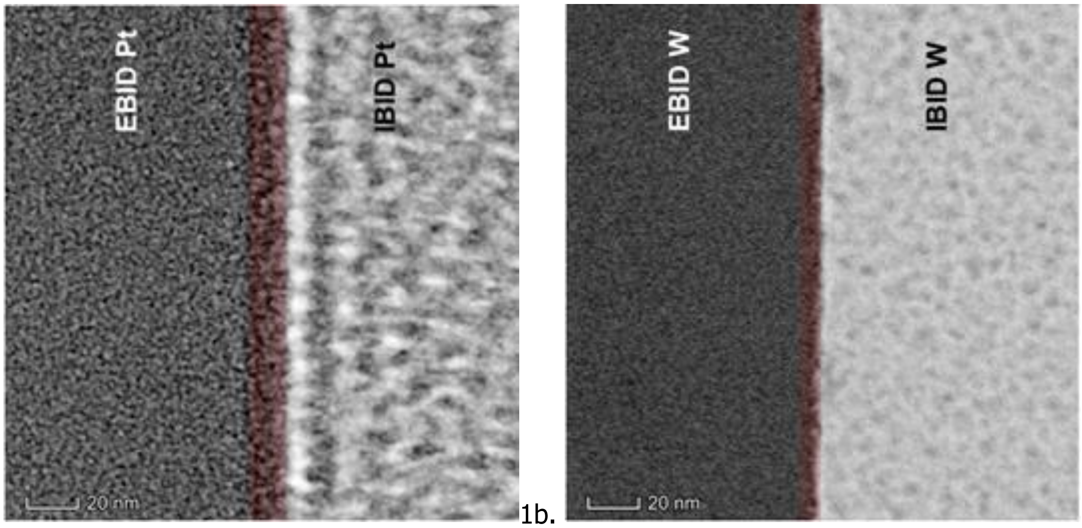

Figure 1. 200 keV STEM images of surface 30 kV Xe^{+} FIB damage during IBID into a) EBID Pt and b) EBID W.

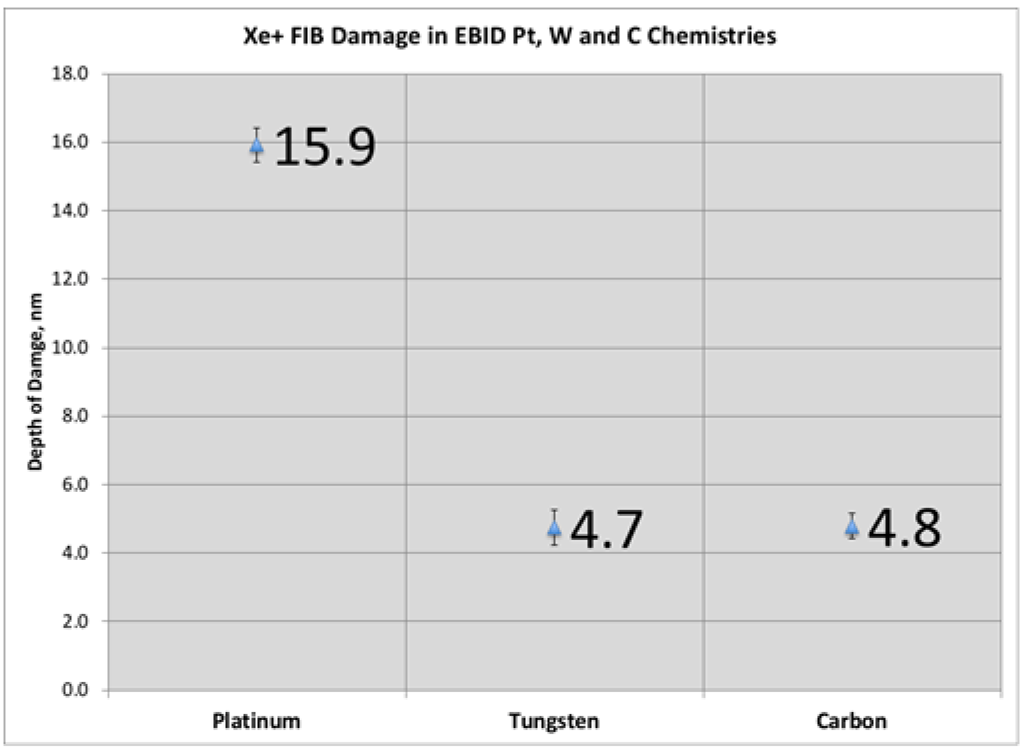

Figure 2. Mean FIB damage depth (nm) during IBID of Pt, W and C over EBID Pt, W and C.