

TEM Specimen Preparation with Plasma FIB Xe⁺ Ions

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A liquid metal ion source (LMIS) of Ga⁺ ions has been the mainstay of focused ion beam technology over the past 20 years or so [1]. While state-of-the-art Ga⁺ ion FIB columns typically possess a resolution of < 5 nm, FIB-based transmission electron microscopy (TEM) specimen preparation techniques were initially developed using Ga⁺ ion beams with much broader beams (i.e., ~ 50-100 nm resolution) [2,3].

Inductively coupled plasma (ICP) sources [4] have recently been introduced into FIB columns. The ICP source yields worse ultimate resolution than the LMIS FIB within the low current regime, but greatly improved resolution in the high current regime [4]. Thus, the plasma FIB (PFIB) allows for faster removal rates of large volumes using a combination of higher beam currents plus larger mass Xe⁺ ions which increase the sputter yield [5]. The PFIB beam size in the low current regime is similar to the beam sizes first introduced in a LMIS FIB. Therefore, it seems plausible that the PFIB could be used for TEM specimen preparation. Indeed, we present PFIB-prepared TEM results below.

FIG. 1a shows a bright field (BF) 200 kV TEM image of a Cu Omniprobe grid that was ion milled to electron transparency using a PFIB source of 25 keV Xe⁺ ions using standard FIB milling techniques [1]. The numerous twin boundaries and high dislocation density are common features in an electrodeposited Cu metal. FIG. 1b shows a 200 keV high resolution TEM image of Si prepared with the PFIB. Note the amorphous layer on the Si surface where the Xe⁺ ion beam was at 0° (normal) incidence is ~ 30 nm thick. It is well known that 30 keV Ga⁺ will produce ~ 50 nm of amorphization damage when exposed at normal incidence to Si [6].

Since Xe is inert, Xe⁺ ion bombardment may be preferable for specimen preparation, particularly for cases where it is well known that deleterious Ga-rich intermetallic phases can precipitate on milled surfaces [7,8]. The high mass Xe⁺ ions also allows for greater throughput via higher sputter yields, and yields less surface damage due to the smaller ion range of Xe⁺ compared to Ga⁺ in a given target. Using SRIM, the sputter yield and longitudinal range for 30 keV Xe⁺ in Si at 0° incidence is 2.9 atoms/ion and 24 nm respectively, while that for Ga⁺ at the same conditions is 2.2 atoms/ion and 28 nm respectively. SRIM plots showing the ion range and recoil motion of 30 keV Xe⁺ and 30 keV Ga⁺ in Si at 0 degrees incidence are found in FIG. 2. The reduced ion range of Xe⁺ is consistent with the observation of less amorphous damage for Xe⁺ over Ga⁺ (despite the slight differences in ion energy herein). In summary, the PFIB can be used to prepare electron transparent specimens and Xe⁺ ions have tremendous potential as an alternative to Ga⁺ ions for TEM specimen preparation and other applications [9].

References

- [1] L.A. Giannuzzi and F.A. Stevie, *Introduction to Focused Ion Beams*, Springer (2005).
 [2] E.C.G. Kirk et al., *Inst. Phys. Conf. Ser.* **100** (1989) 501.
 [3] Kyung-ho Park, *Mat. Res. Soc. Symp. Proc.* **199** (1990) 271.
 [4] N.S. Smith et al., *J. Vac. Sci. Technol. B* **24(6)** (2006) 2902.
 [5] Paul Tesch et al., ISTFA, ASM International (2008) 7.
 [6] B. Kempshall et al., *J. Vac. Sci. Technol. B* **20(1)** (2002) 286.
 [7] J.D. Casey et al., *J. Vac. Sci. Technol. B* **20** 2682.
 [8] J.R. Michael, *Microsc Microanal* **12(Suppl 2)** (2006) 1248.
 [9] Thanks to USF NNRC and Portland State University for access to their TEM facilities.

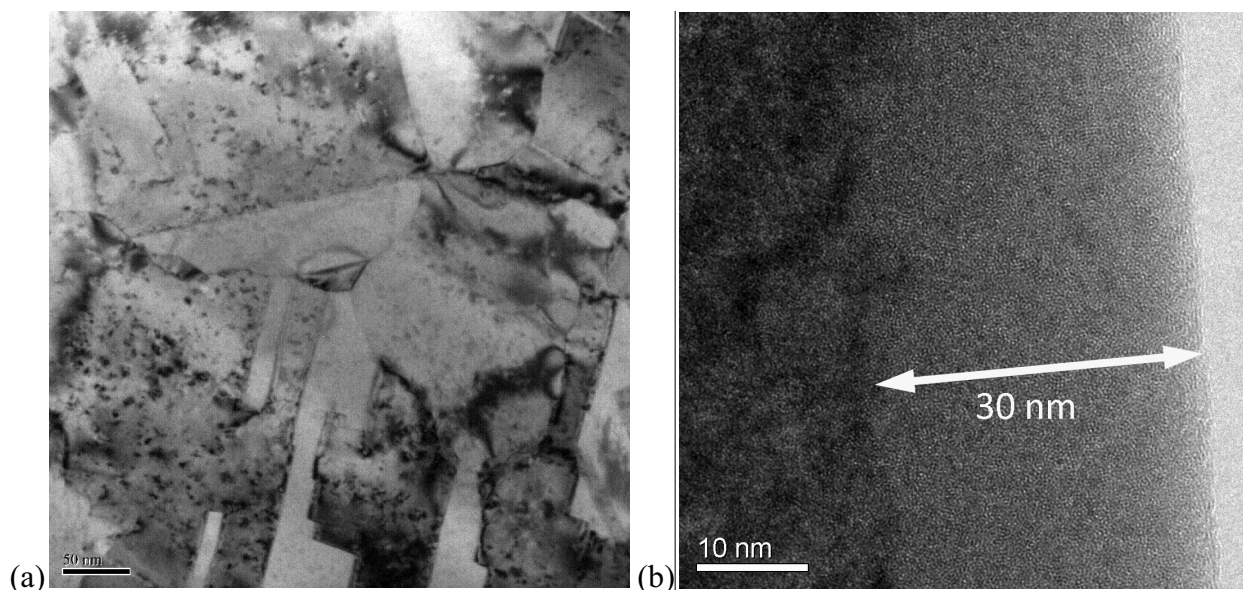


FIG. 1. Examples of TEM specimen preparation using Xe^+ ions from a PFIB. (a) 200 kV BF TEM image of Cu (b) HRTEM image of 25 keV Xe^+ amorphous damage layer in Si exposed at normal incidence (e.g., 0°).

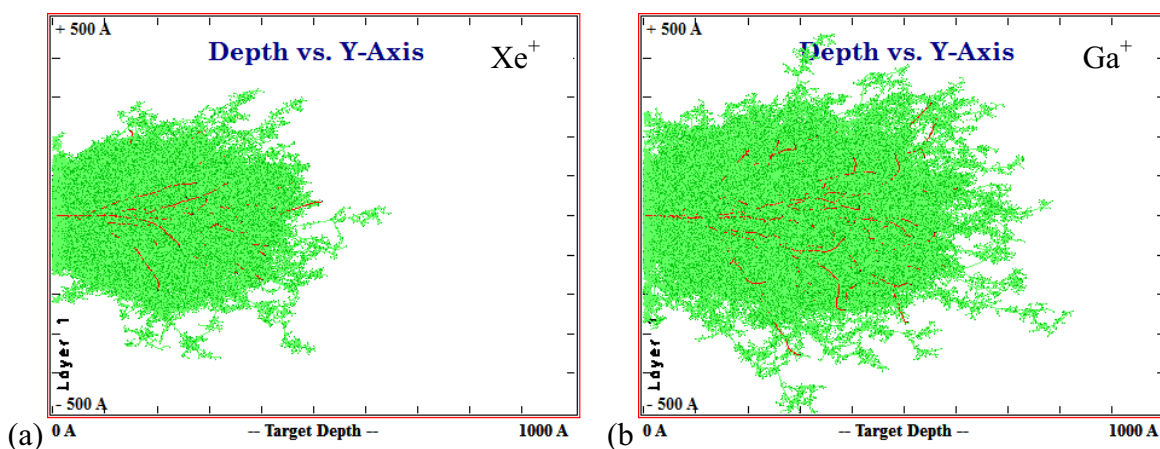


FIG. 2. SRIM plots for 30 keV 0° incidence angle ion bombardment in Si. (a) The range (and damage) of the larger mass Xe^+ ions is less than (b) the range (and damage) of Ga^+ ions at the same energy.