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Accurate Removal of Implanted Gallium and Amorphous Damage from TEM Specimens after Focused Ion Beam (FIB) Preparation

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One of the most popular tools for TEM specimen preparation is gallium focused ion beam (FIB). It is well known that the FIB's high-energy Ga ions can damage a specimen's crystalline structure by introducing lattice defects (strain induction). FIB milling can also implant gallium ions into the specimen surface and cause surface amorphization [1]. An accurate and reproducible specimen preparation method that removes gallium and amorphous damage after FIB processing is necessary for TEM analysis. This work demonstrates how the quality of TEM specimens after FIB can be improved by using low energy argon ion milling.

FIB cross-section specimens were lifted out from monocrystal (100)[011] silicon using a 30 keV Ga ion beam and thinned to 100 nm using various acceleration voltages. The specimens and Ga implantation thickness were quantified by X-ray characteristic emission measurement generated by the electron beam in the specimen. Thickness can be determined using the $\varphi(\rho z)$ model (the XPP approach proposed by Pouchou, et al. [2]) by comparing predicted and measured k-ratios. $\varphi(\rho z)$ is a function that describes the distribution of emerging X-ray intensity as a function of depth, where z is the depth, ρ is the mass per unit volume, and ρz is the mass thickness.

The implanted gallium thickness layer was measured following FIB thinning and again following low energy 500 eV argon ion milling using the NanoMill® TEM specimen preparation system [Fischione Instruments]. X-ray characteristic emission measurement was performed using the 150 mm^2 X-Max^N detector [Oxford Instruments]. Specimens and Ga implanted layer thickness, before and after argon ion milling was evaluated using the AZtec software tool, LayerProbe [Oxford Instruments]. The results are shown in Table 1. The energy dispersive X-ray spectroscopy (EDS) data acquired before and after ion milling show complete removal of implanted gallium (Figure 1). The high quality of the specimen was then confirmed by high-resolution transmission electron microscopy (HRTEM) (Figure 2). The uncertainty of the thickness measurements was estimated, as described by the method in Ancey, et al. [3] and Nowakowski, et al. [4].
Quantification of amorphous layer damage vs gallium implantation by FIB preparation and its removal using NanoMill® argon ion milling will be also discussed.

References:

[1] J Mayer *et al*, MRS Bull. **32** (2007), p. 400.
[2] JL Pouchou, F Pichoir and D Boivin in "Microbeam Analysis", eds. JR Michaels and P Ingram (San Francisco Press, San Francisco), p. 120.
[3] M Ancey, F Bastenaire, and R Tixier, J. Phys. D: Appl. Phys. **10** (1977), p. 817.
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Table 1. Silicon specimen thickness and Ga-implanted layer thickness for each specimen side before and after ion milling.

Specimen preparation method	Silicon thickness	Gallium implanted layer (each side)
30 keV FIB	100 nm	1.5 ± 0.1 nm
5 keV FIB	130 nm	0.3 ± 0.08 nm
30 keV FIB and 500 eV + 300 eV Ar ion milling	70 nm	0 nm

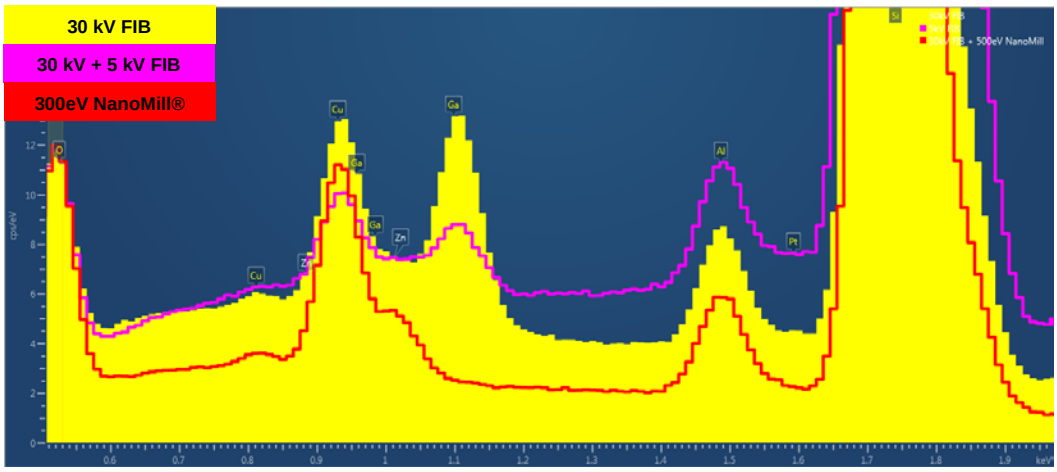

Figure 1. EDS spectra before and after ion milling. Specimen thinned at 30 keV (yellow), 5 keV (pink) Ga FIB, and 300 eV ion milling (red).

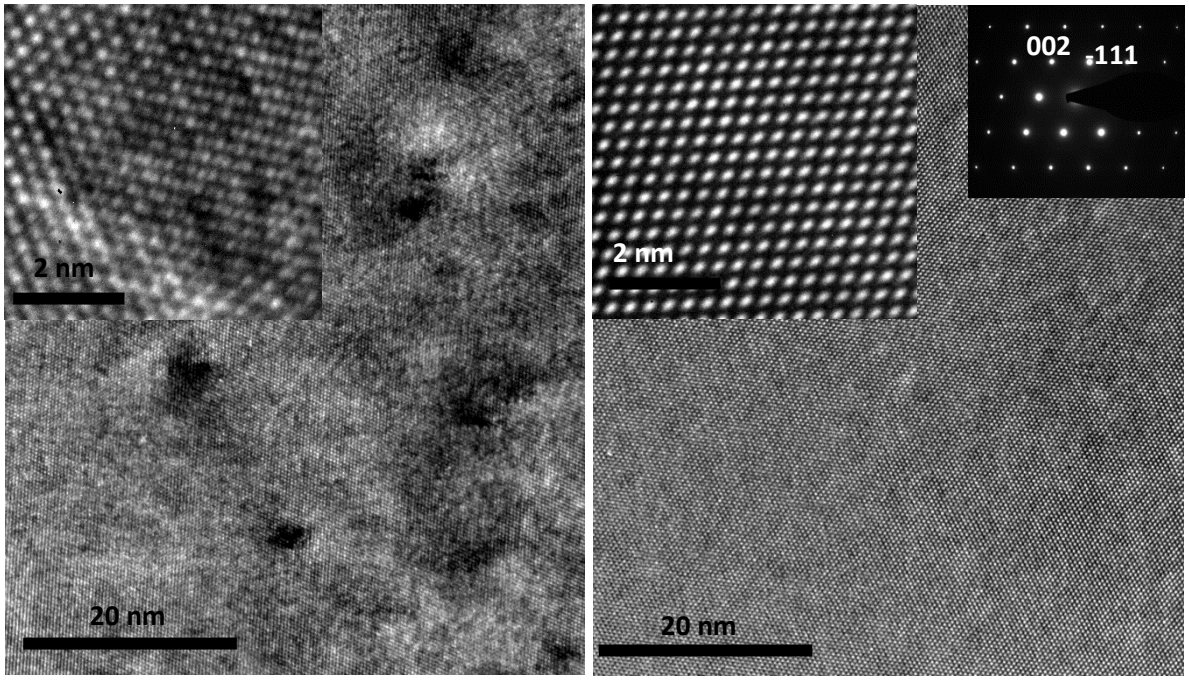

Figure 2. HRTEM images obtained at 200 kV from monocrystal silicon at [011] zone axis after 30 keV FIB gallium thinning (left), and after 300 eV argon ion milling (right).

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