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Raman Spectroscopy and Electron Microscopy Studies of Ga FIB and Post-FIB Ar Ion Milling's Impact on Si TEM Specimens

C.S. Bonifacio[1], S. Tan[2], P. Nowakowski[1], M.L. Ray[1], and P.E. Fischione[1]

[1.] E.A. Fischione Instruments, Inc., Export, PA, USA
[2.] Department of Electrical and Computer Engineering & Nanoscale Fabrication and Characterization Facility, University of Pittsburgh, Pittsburgh, PA USA
*Corresponding author: cs_bonifacio@fischione.com

Transmission electron microscopy (TEM) specimens are prepared for site-specific thinning and extraction using focused ion beam (FIB) using Ga ions or by inductively coupled plasma using Xe, O, N, or Ar ions. For all types of FIB milling ion species, higher milling energies results to specimen damage and reducing ion energies for the final thinning is critical to minimize specimen damage [1]. However, even using low energies in the final FIB polishing step, Ga implantation and amorphous damage [2] are still present. Alternatively, post-FIB concentrated Ar ion beam (CIB) milling at low energies (< 2 keV) removes FIB-induced damage (both amorphous damage and Ga implantation) [3,4]. Here, we use Raman spectroscopy to characterize not only the damage layers, but also residual strain on Si specimens prepared by Ga FIB and post-FIB Ar ion milling. Raman spectroscopy is a nondestructive characterization technique that is receptive to crystal quality, residual strain, and doping concentrations in semiconductors [5]. Amorphous damage from Raman studies will be correlated to TEM measurements from specimens prepared to show sidewall damage.

Specimens from bulk Si sample material were prepared in a Ga FIB by conventional in situ lift-out technique with polishing steps of 30 keV (specimen thickness of 200 nm) and 5 keV (specimen thickness of 130 nm). Using separate Si specimens, post-FIB thinning by CIB Ar ion milling [Fischione Instruments] was performed at low energies. To determine thickness of the amorphous damage in the sidewalls of TEM specimens, FIB preparation using the method in [6] was performed on Si specimens exposed to 30 keV Ga FIB milling and 900 eV Ar CIB milling (Fig.1a) and 30 keV Ga FIB milling followed by 5 keV Ga FIB milling and 500 eV Ar CIB milling. In addition, energy-dispersive X-ray spectra (EDS) and TEM images were acquired across the resulting sidewall damage by the Ga and Ar beams. Subsequently, Raman spectra were acquired on all specimens.

Figure 1 shows scanning electron microscopy (SEM) of the Si specimen during FIB preparation and high resolution TEM images of the resulting sidewall damage after 30 keV Ga FIB milling and after 30 keV Ga FIB milling followed by 900 eV CIB Ar milling. Based on the TEM images, the initial 22 nm thick amorphous layer after 30 keV Ga FIB milling (Figure 1b) was removed completely (0 nm) after Ar CIB milling (Figure 1c). Figure 2a is Raman spectra acquired at 50X magnification from a bulk Si wafer, specimen after 30 kV Ga FIB milling, and specimen after 900 eV Ar CIB milling (Figs. 2c-e). The sharp peak for bulk Si corresponded to 521 cm^{-1}, which is related to crystalline phase. The sharp peak for 30 keV Ga FIB milled and 900 eV Ar CIB milled specimens both aligned to 519 cm^{-1}. However, the specimen after 30 keV Ga FIB milling is the sole specimen with a broad peak centered on 470 cm^{-1}(Fig.2b). Such Raman shift correlated to the amorphous layer on the surface of Si [7], which is in agreement with the sidewall damage observed after 30 keV Ga FIB preparation (Fig.1b). Preparation of the sidewall specimens using 5 keV Ga FIB milling and the 500 eV Ar CIB milling, as well as the Raman stress analysis of specimens following Ga and Ar ion milling are in progress. Calculations of the sharp and broad peak (crystalline and amorphous peak) intensity ratio will be performed based on [7] and compared with the TEM measurements from the sidewall damage specimens.

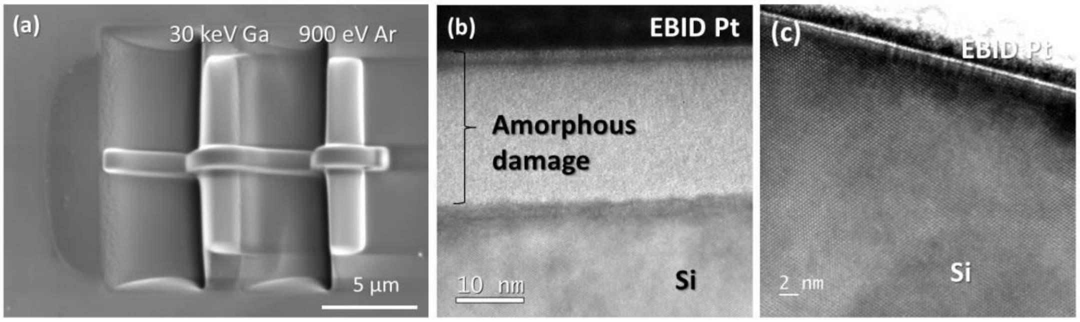

Figure 1. Scanning electron microscope image (**a**) of Si specimen during electron beam-induced deposition (EBID) of Pt after exposure to 30 keV Ga and ex situ to 900 eV Ar. High resolution TEM images acquired from identified areas in (**a**) show amorphous sidewall damage after 30 keV Ga FIB milling (**b**) and amorphous layer removal after 30 keV Ga FIB milling followed by 900 eV Ar CIB milling (**c**).

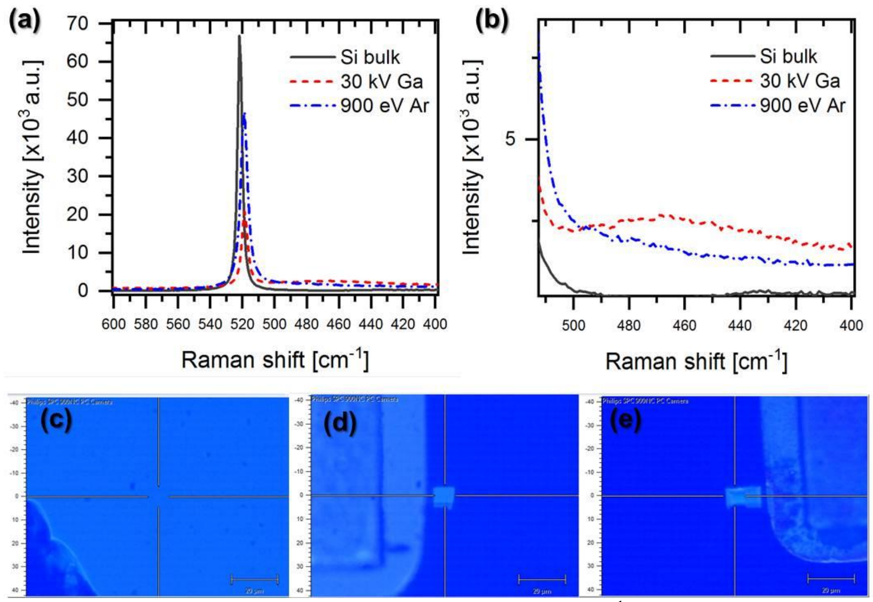

Figure 2. Raman spectra at wide-ranging shift (**a**) and at 550 to 400 cm^{-1} shift (**b**) comparing signals acquired from a bulk Si wafer (**c**), specimen after 30 keV Ga FIB milling (**d**) and specimen after 900 eV Ar CIB milling (**e**). A broadband peak at 470 cm^{-1}, which is related to amorphous damage in Si, is present in (**b**) for the 30 keV Ga FIB prepared specimen.

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