1326
doi:10.1017/S1431927613008623

Microsc. Microanal. 19 (Suppl 2), 2013
© Microscopy Society of America 2013

Application of Low Energy Broad Ion Beam Milling to Improve the Quality of FIB Prepared TEM Samples

Anahita Pakzad[1], Stephen Mick[1], Catherine Vartuli[2], Jayhoon Chung[3] and Guoda Lian[2].

[1.] Gatan Inc. R&D Headquarters, Pleasanton, CA, USA.
[2.] Kilby Imaging Lab, Texas Instruments, Dallas, Texas, USA.
[3.] ATD, Texas Instruments, Dallas, Texas, USA.

Focused ion beam (FIB) is one of the most common techniques for TEM sample preparation, which is especially viable when specific sample orientation or site specific positional accuracy is required. On the other hand, amorphization, redeposition, implantation, vacancies, sample alteration due to beam heating, surface roughness and selective abrasion are some of various damages caused by this method [1].

Amongst all these problems, FIB induced surface amorphization is the most extensive and limiting. The reason is two fold. Firstly, the amorphous material reduces the signal to noise ratio [2]. Secondly, in order to avoid geometrical blurring for high resolution transmission electron microcopy (HRTEM) applications, the thickness of the sample must be reduced by the square root of the feature size [3]. A 25 KeV Ga+ FIB column can generate ~20 nm per specimen side in damage for a Si sample [4] and this could be specifically limiting in the case of modern Si based semiconductor devices with less than quarter micron features.

While the depth of FIB induced amorphous layer depends on beam energy, beam angle and the material being milled, several efforts have been dedicated to reduce or eliminate this damage in TEM samples:

- Gas-assisted etching: although it enhances the milling rate, it increases the roughness of crystalline-amorphous interface, which leads to further damaging of TEM images [5].
- Low energy FIB: although reduction of beam energy reduces the damage depth, etching rate and positional resolution are also reduced at these energies.
- Wet etching: although, when suitable solution is used, significant improvements have been observed in single layered materials [3], this method cannot be used for multi layer semiconductor devices
- Argon ion milling: This is the most promising method for multi-layered materials, where the original FIB damage layer is replaced by newly formed Ar+ induced damage layer [3, 6]. The thickness of this new layer can be minimized through optimization of milling parameters (broad ion beam energy, incident angle and milling temperature).

In the current study, the quality of FIB prepared TEM samples was improved and the FIB induced amorphous layer thickness was reduced by the use of low energy (<300 eV), broad beam ion milling in Gatan's Precision Ion Polishing System II (PIPS II). Multiple new features in PIPS II such as focused ion beams at low energies, X-Y alignment stage, optical camera along with Digital Micrograph imaging software and stationary milling mode, make this possible.

Several FIB prepared specimens made of different materials were collected. In the case of lift-out samples, they were mounted on either Cu or Mo OmniProbe grids. Specimens were imaged before any cleaning in the PIPS II, as to have a reference of the sample condition after preparation in FIB. Samples

https://doi.org/10.1017/S1431927613008623 Published online by Cambridge University Press

were then polished using low energy Ar ion guns in stages with sequential observation in a TEM. Argon Beams were set to mill at incident angles <5° from either top or bottom. Samples were cooled during polishing to reduce heat induced damage. Samples were positioned using the PIPS II X-Y stage with respect to each gun and the sample was stationary during milling. Milling the samples in Stationary Mode and focused ion guns at low energies made it possible to polish the FIB sample efficiently at <300 eV, without redeposition of the holder material (Al) or the grid they were mounted on (Cu or Mo). Results are shown in figures bellow.

References:

[1] J. Ayache *et al* in "Sample Preparation Handbook for Transmission Electron Microscopy: Methodology", (Springer, New York) p. 126.
[2] M.J. Suss, E Mueller and R. Wepf, Ultramicroscopy **111** (2011), p. 1224.
[3] N.I. Kato, Journal of Electron Microscopy **53** (2004), p. 451.
[4] L.A. Giannuzzi and F.A. Stevie, Micron, **30**, (1999) p. 197.
[5] K Gamo, Materials Research Society Symposium Proceedings (1993) p. 577.
[6] A Barna, Journal of Computer Assisted Microscopy, **9**, (1997), p. 101.

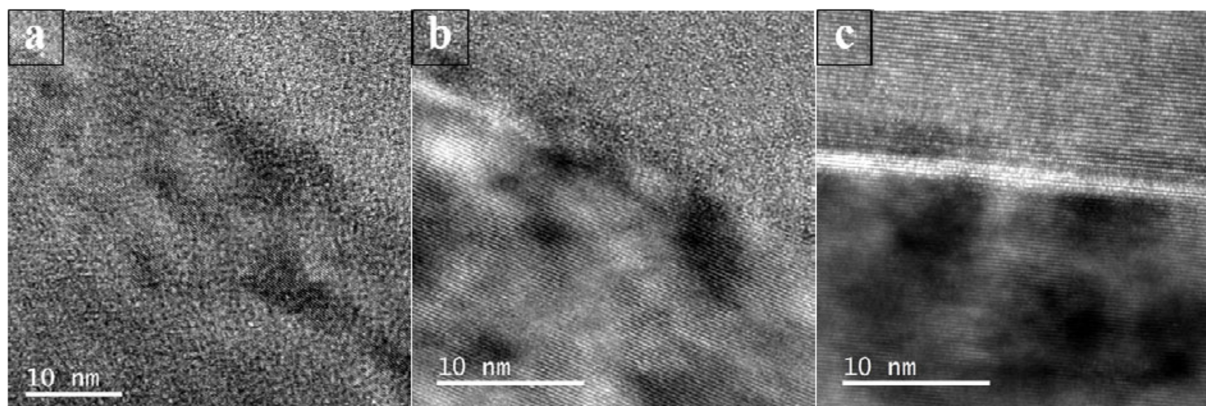

Figure 1. TEM micrographs of a multi layer sample (a) as prepared in FIB, (b) after polishing in PIPS II for 1 min, (c) after polishing in PIPS II for additional 40 s; due to FIB induced amorphization, the second layer in the middle is not visible in (a) and (b), but after sufficient Ar ion polishing it is clearly visible in (c).

https://doi.org/10.1017/S1431927613008623 Published online by Cambridge University Press