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High-Resolution Secondary Electron Imaging of a FIB Prepared Si Sample with an Aberration Corrected Electron Microscope.

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Demands on structural characterization at atomic level not only in the bulk but also on the surface of a sample using electron microscopes are rapidly increasing in the field of material science and engineering. For a better understanding of the surface morphology as well as atomic structure it is necessary to exploit methods that can improve the resolution of surface structures. In order to respond to such demands, we have developed a dedicated STEM instrument, Hitachi HD-2700 [1], incorporated with a high efficiency secondary electron detector. The instrument is equipped with a CEOS GmbH aberration corrector and can routinely achieve 1A spatial resolution in secondary-electron (SE) imaging and STEM imaging [2]. Here we report an application of the method on a focused-ion-beam (FIB) prepared Si sample.Our samples have a pillar geometry and are covered with a Ga-ion damage-layer (FIG.1) induced during FIB milling with a NB5000 FIB-SEM equipped with micro-sampling system [3]. We measure the thickness of the damaged layer caused by fabrication at the accelerating voltage of the FIB (2-40kV) using HD2700C in the SE mode operated at 200 kV. We take advantage of our unique FIB-STEM compatible sample holder that allows a 360° sample rotation inside the FIB system and the microscope [4]. FIGs.2 are the cross-sectional TEM images of the interface between the FIB damaged surface amorphous layer and Si crystalline bulk, in which the samples were thinned at different acceleration voltages. The thickness of the surface amorphous Si layer was measured to be 28 nm at 40kV(a), 8 nm at 10kV(b) and 3nm at 2kV(c), respectively. FIG.3(a-c) shows the corresponding SE images observed along the Si[011] zone axis. Not surprisingly, the SE image of the sample thinned at 2 kV demonstrates the best result and that at 40 kV yields very poor signal noise ratio. FIG.4 is a magnified SE image of FIG.3a, with the corresponding Fourier diffractogram showing the achievable frequencies of the 004 reflection (silicon dumbbell) and the 333 and 115 reflection (105 pm). These results emphasize the possibility of SE images by higher energy electron to realize the surface chemical characteristics of materials. Although SE image at atomic resolution of light element such as Si is exciting, the fact that it can reveal structure underneath of a 2-3nm amorphous Si is unexpected. Further studies are needed to fully understand the underlying mechanism of SE imaging.

References

[1] Inada, H., Wu, L., Wall, J., Su, D., & Zhu, Y., *J. of Electron Microsc.* 58, 111-122 (2009)
[2] Zhu, Y., Inada, H., Nakamura, K. & Wall, J., *Nature Mater.* 8, 808-812 (2009)
[3] Ohnishi,T. and Ishitani,T., JP Patent 2774884 and US patent 5270552
[4] Kamino, T., Yaguchi, T., Konno, M., Ohnishi, T and Ishitani, T., *J.Electron Microsc.* 53, 583-588T (2004).

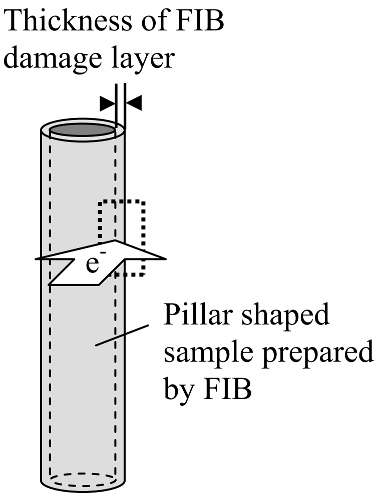


FIG . 1 Measurement of the thickness of FIB damage layer.

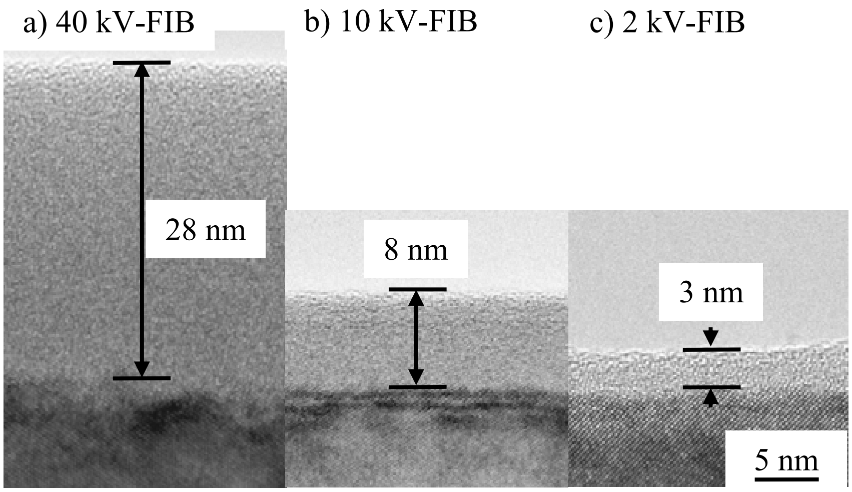


FIG . 2 Cross sectional TEM images of the FIB damaged amorphous layer of samples thinned by a 40 kV (a), 10 kV (b) , and 2 kV (c) FIB beam.

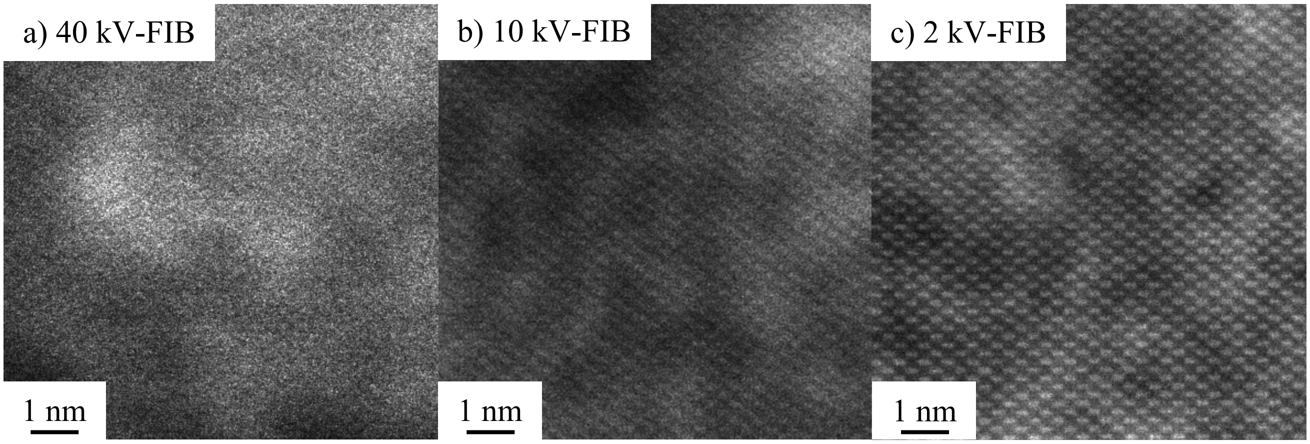
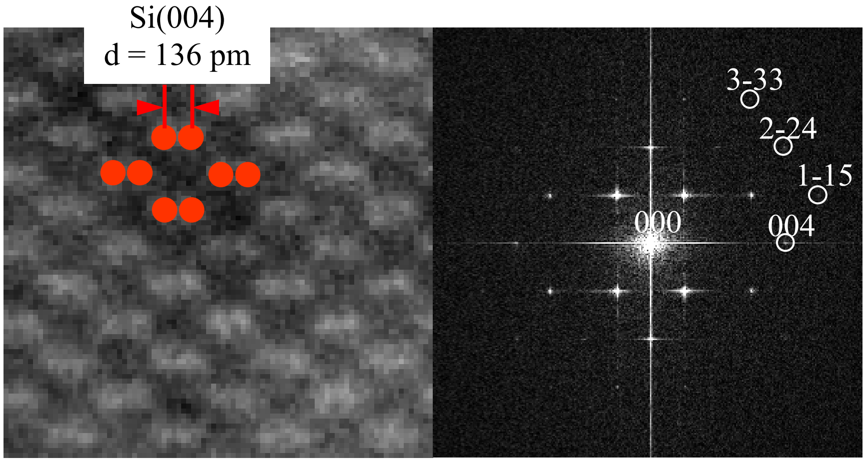


FIG . 3 Secondary electron (SE) images taken with HD2700C at 200 kV along the Si[011] zone axis of a Si samples prepared using a FIB beam at 40 kV (a), 10 kV (b) and 2 kV (c).



Si(004) d = 136 pm
Si(2-24) d = 111 pm
Si(3-33) d = 105 pm
Si(1-15) d = 105 pm

FIG . 4 A magnified SE image observed along the Si[011] zone with the corresponding FFT.