Type of presentation: Poster

IT-4-P-1607 Application for Low Energy STEM with the In-lens Cold FE-SEM

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Inorganic carbon materials (primary carbon nanotubes and graphene) and organic polymeric materials are being developed more actively. The demands for fine structural, elemental, and chemical characterization of these materials by electron microscopes are rapidly increasing. These requirements have increased the demand to achieve high resolution STEM imaging at low accelerating voltages. It is necessary to determine methods to improve the contrast intensity at low accelerating voltage operation without loss of resolution. In order to respond to such demands, we have developed the Hitachi SU9000 (Figure 1), a cold FE-SEM (CFE-SEM) with an in-lens type of objective lens, capable of high resolution phase contrast STEM imaging. Through using this technique on this microscope, it is possible to routinely achieve lattice resolution of the graphite {002} planes with a spacing of 0.34 nm. In this study, we improve the observation conditions for obtaining enhanced lattice resolution in STEM imaging at an accelerating voltage of 30 kV. Additionally, we have shown the effectiveness of this method for imaging inorganic carbon based materials. Figure 2 shows a simplified lens diagram of the SU9000. By using newly optimized lens parameters and a specialized sample stage which reduces the distance between the objective lens and sample, the Cs was lowered from approximately 2 mm to 1 mm. Figure 3 shows a high resolution BF-STEM image with its inset Fourier transform (FFT) image, observed along the Si<110> zone axis at an accelerating voltage of 30 kV. The sample was prepared using the NB5000 FIB-SEM equipped with a unique micro-sampling system. The specimen was thinned down to approximately 30 nm thickness. (a) is the standard condition (WD: 3 mm,Cs:2 mm) and (b) is the optimized condition (WD: 1.8 mm, Cs: 1 mm). Both (a) and (b) imaged the Si {111} plane, which has a spacing of 0.314 nm, and reflection spots corresponding to 0.314nm were confirmed from FFT images. However, in the optimized condition, not only Si {111} planes (corresponding to 0.314 nm) but also the {002} planes (corresponding to 0.272 nm) are detected from FFT. This confirms that the image resolution is improved by reduction of Cs. Next we applied the optimized condition to a graphene sample. Figure 4 shows a high resolution BF-STEM image with its inset FFT image, the multi-layer graphene membrane was clearly observed at an accelerating voltage of 30 kV. Lattice fringes were easily observed and the reflection spots corresponding to 0.213 nm were successfully confirmed. These results reveal the potential for high contrast visualization without loss of resolution for any carbon-based materials and the latest semiconductor devices with minimal radiation beam damage.

Acknowledgement: The multi-layer graphene membrane specimen was kindly supplied by Dr. Tsuyohiko Fujigaya of the Department of Applied Chemistry Graduate School of Engineering, Kyushu University.

Fig. 1: General view of SU9000 In-lens FE-SEM.

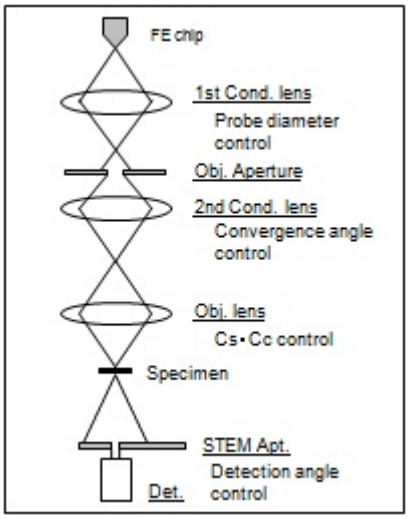

Fig. 2: Configuration of SU9000.

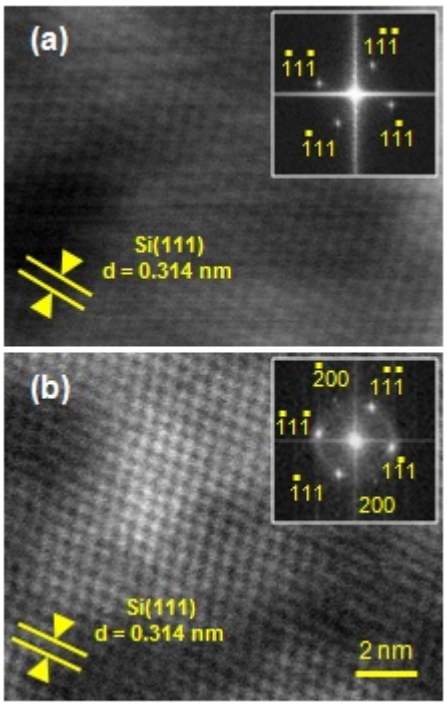

Fig. 3: BF-STEM images and FFT images of Si <011> single crystal. (Accelerating voltage is 30 kV)

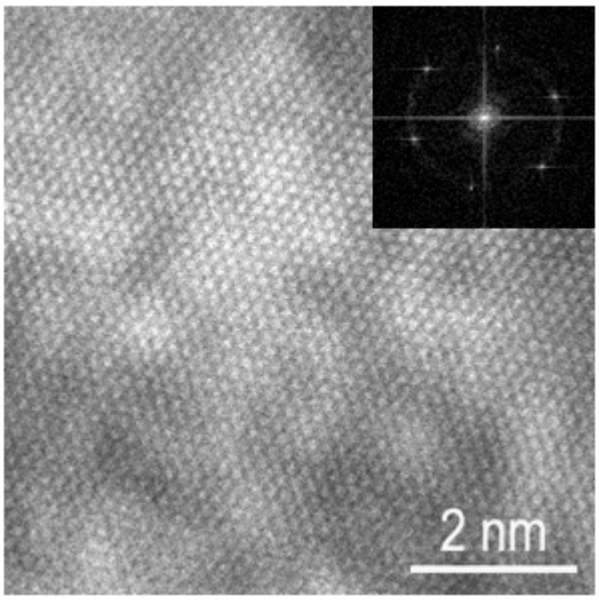

Fig. 4: BF-STEM image and FFT image of graphene. (Accelerating voltage is 30 kV)