Atomic resolution observation and analysis of carbon materials at low acceleration voltages using aberration corrected microscope with cold field emission gun

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For the analysis of carbon materials, such as carbon nanotube and graphene, low acceleration voltage is essentially requested, since these materials are easy to be destroyed through knock-on damage by electrons at high accelerating voltage. The low accelerating voltages below 80 kV are effective to reduce the damage, though the spatial resolution becomes worse, since the longer wavelength of the electrons makes the diffraction limit and chromatic aberration increased. Owing to the development of aberration correctors and the easy-to-use cold field emission gun that serves smaller and chromatic aberration due to narrow energy spread (ΔE) of electrons, the allowable convergence angle (α) was improved to be larger, resulting in better diffraction limit. In this paper, we report the practical experimental conditions, such as convergence angle, electron extracting voltage and etc., for atomic resolution observation and analysis at low accelerating voltages.

In our experiment, we used an aberration corrected microscope with the fifth-order aberration corrector to expand the convergence angle, and we reduced the electron extracting voltage from an emitter to obtain electrons with narrow energy spread. To earn the enough probe current for the analysis and image observation, we used large aperture. Figures 1(a) and (b) show the STEM-ADF images of Si [110] obtained with two different energy spreads (0.4 eV for (a) and 0.3 eV for (b)). The convergence angles for these are same (34.6 mrad). The geometrical aberrations up 5^{th} order was well eliminated with the aberration corrector as the flat region of the Ronchigram was extended to be over 50 mrad. As results, we successfully observe the clear dumbbell structure of Si [110] at 30kV. Figure 2 shows the STEM-ADF image of mono-layer graphene and EELS spectra of the C-K edge at a mono-layer graphene edge. The ADF image shows clear 6-membered ring of mono-layer graphene which was observed with conditions: $\Delta E = 0.34$ eV $I_{probe} = 30$ pA and $\alpha = 34.6$ mrad. The peaks in EELS spectra from different positions of C-K edge (see arrows in Fig. 2(b)) have different shapes. It was considered that they were reflected the electronic state of carbon atoms depending on local structures at the edge of graphene.

We have demonstrated that atomic resolution observation and analysis for mono-layer carbon atoms are possible even at 30kV. Therefore, we think that the conditions in these experiments are applicable for other soft material, which destroys their structure due to knock-on damage.

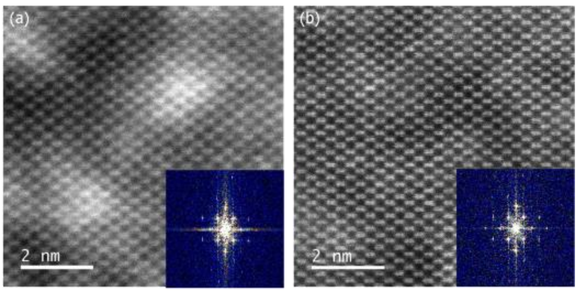

Fig. 1 STEM-ADF image of Si [110] by changing energy spread of electron at 30kV(a) 0.4eV, (b) 0.3eV)

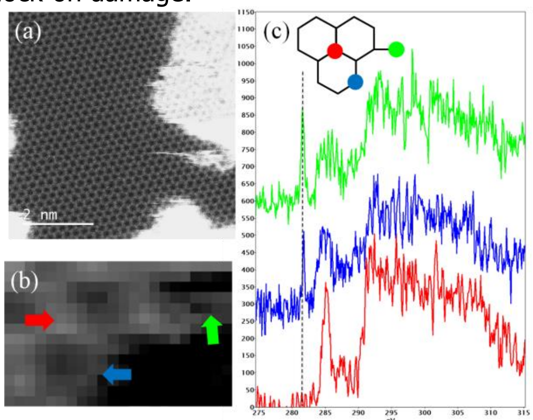

Fig. 2 (a) STEM-ADF image of a mono-layer graphene (b) STEM-ADF image of a mono-layer graphen edge (c) EELS spectrum obtained from each carbon atom