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Atomic Resolution Imaging and Analysis of Graphene 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 required to reduce the knock-on damage by high speed electrons such as those accelerated at 200 kV and 300 kV. The low accelerating voltages below 80kV are essential to reduce the damage. However, the spatial resolution becomes worse, since the longer wavelength of the electron increases the diffraction limit (D_d) and chromatic aberration (D_c), which are defined as follows.

$D_d = 0.61\ \lambda/\alpha$, λ: wavelength of an electron, α: convergent semi-angle of STEM probe onto a sample.

$D_c = Cc\ (\Delta E/E_0)\alpha$, Cc: chromatic aberration coefficient, ΔE: energy spread of electron, E_0: energy of primary electron.

Owing to the development of higher order aberration correctors and the easy-to-use cold field emission gun that can change the accelerating voltage easily, chromatic aberration becomes smaller due to narrow energy spread (ΔE) of electrons and the allowable convergence angle (α) is improved to be larger, resulting in better diffraction limit. This paper reports the practical experimental conditions, such as convergence angle, electron extracting voltage, for atomic resolution imaging and analysis at low accelerating voltages.

In our experiment, we used an aberration corrected microscope with the fifth-order aberration corrector to reduce aberrations up to 5-th order such as six fold astigmatism (A_5). We reduced the electron extracting voltage from an emitter to obtain electrons with narrow energy spread. Consequently we obtained the enough probe current for the analysis and imaging using large-sized apertures. Figure 1 shows the STEM-ADF images of Si [110] obtained with two energy-spreads (0.4 eV for (a) and 0.3 eV for (b)). The convergence angles for these images were the same (34.6 mrad). The geometrical aberrations up to 5^{th} order were 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 and EELS spectra from a mono-layer graphene. The EELS spectra are of C-K edges from carbon atoms at different positions. The STEM-ADF image shows the clear hexagonal rings of carbon atoms. The image was observed under conditions: $\Delta E = 0.34$ eV, $I_{probe} =$ 30 pA and $\alpha = 34.6$ mrad. When the extraction voltage is lowered, chromatic aberration is reduced while the beam current is decreased. So, the reduction of the probe current was recovered by using the large aperture. The peaks in EELS spectra from different positions of C-K edge (see arrows in Fig. 2(b)) have different shapes and positions. These results were considered to reveal the electronic state of carbon atoms depending on local structure at the edge of graphene.

We have demonstrated atomic resolution imaging and analysis of mono-layer carbon atoms at an accelerating voltage as low as 30 kV. We also suppose that the conditions in our experiments are applicable for other soft materials whose structure is easily destroyed by knock-on damage.

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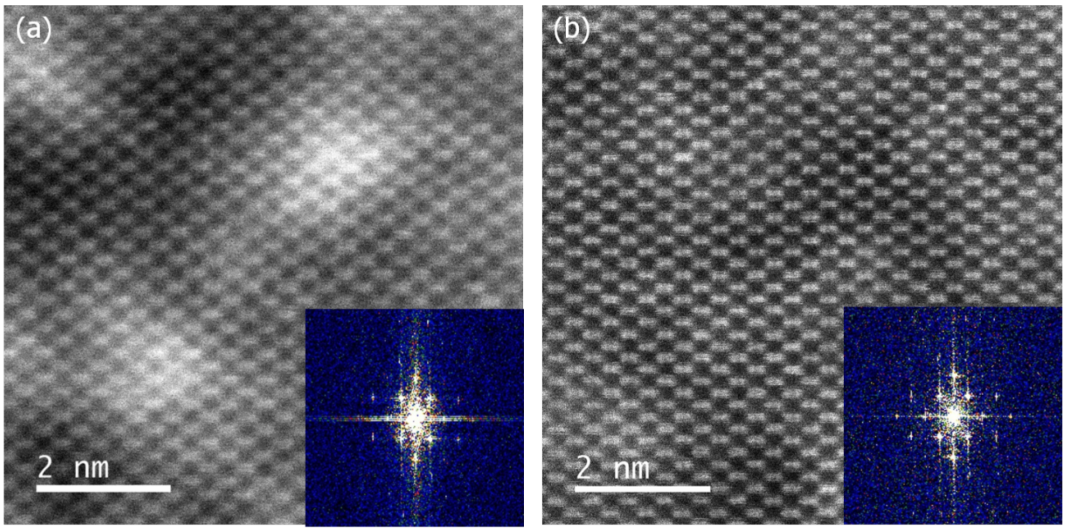

Figure 1. STEM-ADF images of Si [110] with different energy spreads at 30kV (a) 0.4eV, (b) 0.3eV.

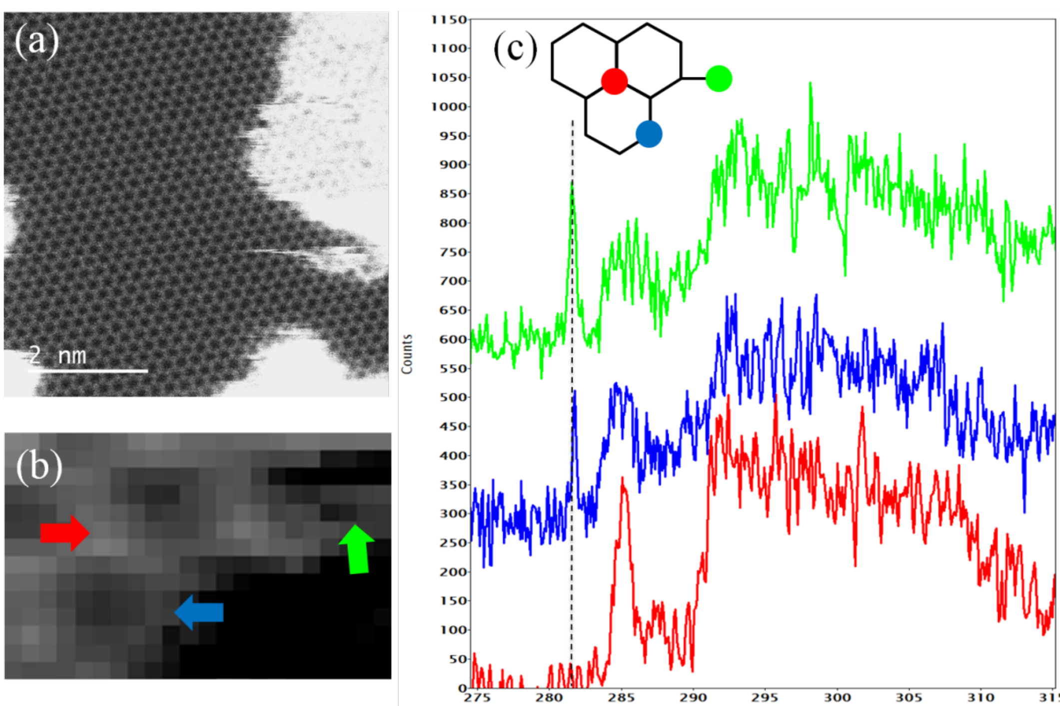

Figure 2. (a) STEM-ADF image of a mono-layer graphene (b) STEM-ADF image of a mono layer graphene edge (c) EELS spectra obtained from carbon atoms at different positions.

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