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STEM/SEM, Chemical Analysis, Atomic Resolution and Surface Imaging At ≤ 30kV with No Aberration Correction for Nanomaterials on Graphene Support

Takeshi Sunaoshi[1], Kazutoshi Kaji[2], Yoshihisa Orai[3], C.T. Schamp[4] and Edgar Voelkl[4]

[1.] Hitachi High-Technologies Corp., Application Development Department, Ibaraki, Japan.
[2.] Hitachi High-Technologies Corp., Electron Microscope Systems Design 1st Dept., Ibaraki, Japan.
[3.] Hitachi High Technologies Corp., Application Development Dept., Kanagawa, Japan.
[4.] Hitachi High Technologies America, NSD, Clarksburg, Maryland, USA.

For the investigation of nanomaterials and so-called weak-phase objects, going to lower acceleration voltages can be an advantage for several reasons: the knock-on damage [1] decreases significantly and objects displaying little to no contrast at higher voltages (weak phase objects) appear with more contrast due to their increased interaction with the sample.

Full analytical capabilities considered standard for high-voltage STEM/TEMs at 30kV are expensive and typically require monochromators especially for Schottky emitter based instrumentation [1-3] due to the strangle-hold of the chromatic aberrations. In addition, the power supplies for lenses designed for 200/300kV contribute increasingly to the energy width of the electron beam at the low level currents needed for ≤ 30kV instrumentation. Therefore, enhancing an atomic resolution SEM with a cold field emission gun (cFEG) with STEM, EELS and diffraction capabilities provides an excellent platform for combining surface investigations typically for SEMs with high resolution and analysis capabilities of a typical STEM at comparatively low cost.

Graphene as support structure for the analysis of nano-material is well established, but high quality single layer Graphene sheets remain difficult to obtain. Figure 1, left, shows a high-resolution BF STEM image of Graphene with its hexagonal structure resolved. Figure 1, center shows EELS data comparing different areas of the support structure with the data from deposited graphite and diamond nano-particles. The comparison shows a red-shift of the low-loss peaks of graphene with an increasing number of stacked graphene sheets with the biggest red shift occurring for graphite. It appears the red-shift can be used to determine the number of overlaid Graphene sheets. As a comparison, the EELS data (FWHM ≤ 0.4eV) for graphite, diamond and amorphous C were added. Figure 1, right, shows the core loss part of the EELS spectra, consistent with the low-loss data.

Similarly, diffraction data from the same sample and microscope can be used to differentiate the number of layers of Graphene as shown in Figure 2. The data were obtained from a beam with a 9mrad convergence angle and a spot size of 0.5nm diameter. For a large beam diameter of 60nm, the illumination can be adjusted (not shown here) to an almost parallel beam creating a diffraction pattern consistent with the diffraction patterns expected from a TEM (images not show).

The surface of the sample was then imaged via SE at 1kV that allows identification of Au particles deposited on top of the graphene support structure and the result is shown in Figure 3, left. The acceleration voltage was then increased to 30kV and the corresponding SE and BF-STEM images are shown in the middle of Figure 3. The corresponding elemental map via EDX (Oxford Instruments) is shown in Figure 3, right.

Investigations of nano-particles can require a significant number of methods both, EELS and EDX, diffraction, the capability to image at atomic resolution and for differentiating the surface from an STEM image. Such capabilities are available in a single instrument at reasonable cost.

References:

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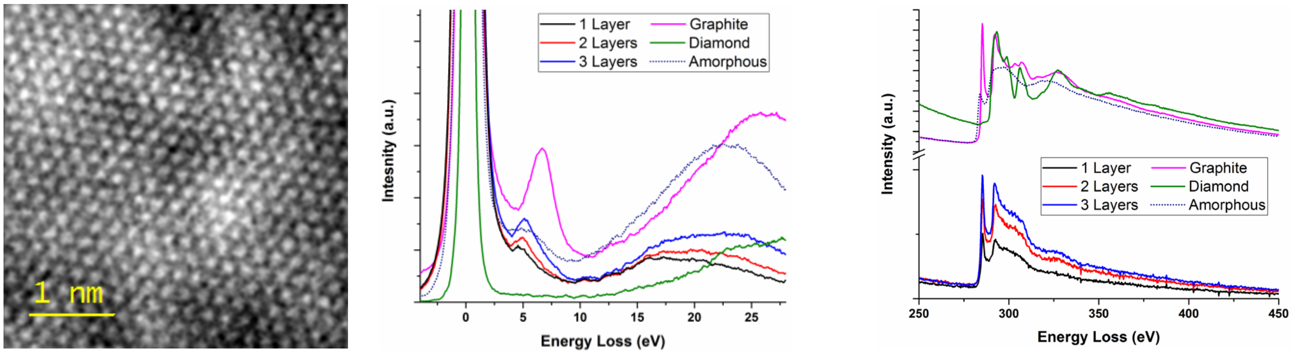

Figure 1. Left: Low-pass filtered, BF STEM image of Graphene at 30kV. Middle: Low loss EELS spectra of 1, 2 and 3 layer graphene, graphite, diamond and amorphous carbon film. The red-shift of graphene with increasing number of layers can be seen. Data were taken at 30keV and a FWHM of <0.4eV for the zero loss peak. Right hand side: Corresponding K-edges from the same EELS data.

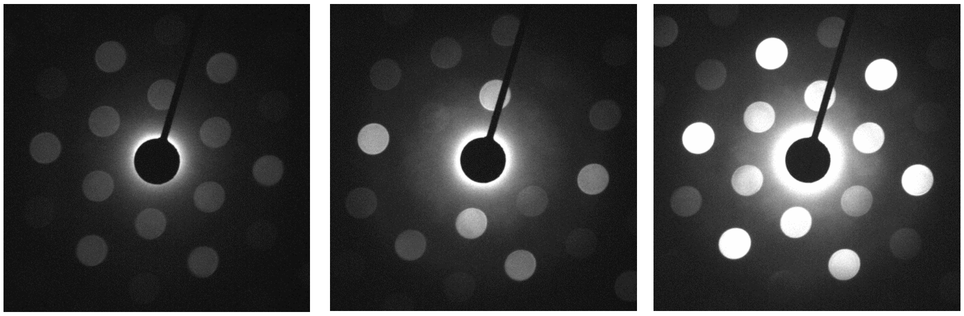

Figure 2. From left to right: diffraction patterns of 1-layer, 2-layer and 3-layer graphene obtained with an electron beam of 0.5nm diameter and a convergence angle of 9mrad (at 30kV).

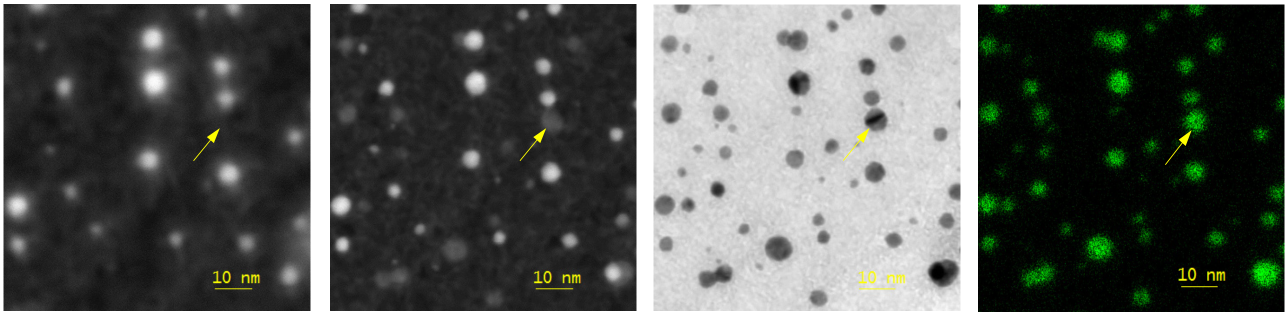

Figure 3. Left: SE imaging the surface of the sample at 1kV shows the location of Au particles on the upper surface of the graphene film. Middle-Left: SE image of same particles at 30kV showing particles on both side of the film. Middle-Right: BF-STEM image at 30kV. Right: elemental EDX map thereof.