Application of 80-200 kV aberration corrected dedicated STEM with cold FEG

This content has been downloaded from IOPscience. Please scroll down to see the full text.
(http://iopscience.iop.org/1742-6596/241/1/012011)

View the table of contents for this issue, or go to the journal homepage for more

Download details:

IP Address: 54.191.40.80
This content was downloaded on 17/08/2017 at 01:49

Please note that terms and conditions apply.

You may also be interested in:

High-resolution SEM observation at the atomic level using a dedicated STEM with aberration correction
M Konno, Y Suzuki, H Inada et al.

Configuring a 300kV cold field-emission gun for optimum analytical performance
I M Ross and T Walther

Application of Low Energy STEM with the In-lens Cold FE-SEM
Y Orai, T Sunaoshi, S Okada et al.

Ultra-high resolution electron microscopy
Mark P Oxley, Andrew R Lupini and Stephen J Pennycook

Experimental setup for energy-filtered scanning confocal electron microscopy (EFSCEM) in a double aberration-corrected transmission electron microscope
P Wang, G Behan, A I Kirkland et al.

Present status and future prospects of spherical aberration corrected TEM/STEM for study of nanomaterials
Nobuo Tanaka

Comparison of the contrast in conventional and lattice resolved ADF STEM images of InGaAs/GaAs structures using different camera lengths
Y Qiu, L Lari, I M Ross et al.

Towards quantitative analysis of core-shell catalyst nano-particles by aberration corrected high angle annular dark field STEM and EDX
H E, P D Nellist, S Lozano-Perez et al.
Application of 80-200 kV Aberration Corrected Dedicated STEM With Cold FEG

M Konno, Y Suzuki, H Inada and K Nakamura

1Naka application Center, Naka Division, Nanotechnology Products Business Group, Hitachi High-Technologies, 11-1, Ishikawa-cho, Hitachinaka-shi, Ibaraki-ken, 312-0057, Japan
2Advanced Microscope System Design 2nd Department, Naka Division, Nanotechnology Products Business Group, Hitachi High-Technologies, 882, Ichige, Hitachinaka-shi, Ibaraki-ken, 312-8504, Japan

E-mail: konno-mitsuru@naka.hitachi-hitec.com

Abstract. We have developed new STEM instrumentation with a cold field emission source (Hitachi HD-2700) in order to perform structural characterization and elemental mapping at the atomic level. The instrument utilises the CEOS GmbH (Germany, managing director: Dr. Max Haider) aberration corrector. The accelerating voltage range is between 80 kV and 200 kV. The cold field emission source proves to be the ideal emitter for analytical transmission electron microscopes due to its high brightness, high current density and small energy spread. In this study, we have examined low accelerating voltage conditions for obtaining high image contrast and high performance elemental analysis (in which FWHM of zero loss peaks are 0.3 eV for acquisition time of 1 second and 0.34 eV for acquisition time of 40 second by accelerating voltage of 80 kV, respectively). We have observed high contrast bright field STEM images of graphene carbon at an accelerating voltage of 80 kV, in which lattice fringes can be clearly seen.

1. Introduction

Electron energy loss spectroscopy (EELS) is a powerful technique for qualitative and quantitative elemental analysis. The spectrum shape also contains information on the chemical bonding state, commonly referred to as energy loss near edge structure (ELNES). Recently, the demands for ELNES analysis have increased as material scientists investigate novel materials and devices on the nanoscale. For this reason cold field emission guns [1] or monochromators [2] have been utilized in transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) and the achievable energy resolution has reached around 0.2 eV. Monochromated electron sources, however, have a few weaknesses, such as low probe current and enlarged probe size. In comparison, a cold field emitter has high current density while maintaining a small energy spread, without the drawbacks described above, and therefore maybe the ideal source for an analytical TEM or STEM. Furthermore, spherical aberration (Cs) correctors are now commercially available devices. It is indeed possible to make up for some of the weaknesses of a monochromator with the advantages of a Cs corrector in STEM (with the

1  konno-mitsuru@naka.hitachi-hitec.com
Cs corrector improving probe current and reducing probe size) but it is clearly preferable have an initial source with higher current and lower probe size. Additionally, according to optical principles, the contrast of the elastically scattered electron in aberration corrected STEM is less than that of STEM without Cs corrector. This clearly has an effect on imaging of light elemental materials such as nm-sized carbon particles and some polymers and it is necessary to determine methods to improve the contrast such as lower voltage operation. In this study, we have investigated low accelerating voltage conditions for obtaining high performance imaging and EELS using a STEM instrument equipped with a with Cold FE gun and Cs corrector.

2. Experiment
In order to respond to such demands, we have developed a dedicated STEM instrument with a newly-designed ultrahigh vacuum Cold FE gun (Hitachi HD-2700), in which the CEOS GmbH (Germany, Managing Director: Dr. Max Haider) Cs corrector has been utilized and optimized [3]. The convergence angle of the aberration corrected STEM is around 40mrad[4] at 200kV and 25mrad at 80kV, and the accelerating voltage range is between 80 kV and 200 kV. Source brightness are $1 \times 10^9$ A/cm²·Sr at 200 kV, $6 \times 10^8$ A/cm²·Sr at 120 kV and $4 \times 10^8$ A/cm²·Sr at 80 kV. The instrument is equipped with an EELS spectrometer (Gatan, ENFINA, 16 bit 100×1340 pixel CCD) and a CCD camera (Gatan, ORIUS, 14 bit 2.6k×2.6k) for the capture of diffraction and Ronchigram patterns. Additionally, the column unit is fitted with a magnetic shielding cover (which showed significant improvement in EELS stability due to the shielding of electromagnetic interference). Here, we have measured image resolution, energy spread and beam stability using EELS at accelerating voltages of 80 kV, 120 kV and 200 kV. We have also compared the EELS spectrum of graphene carbon, known for its susceptibility to radiation damage, at accelerating voltages 200 kV and 80 kV.

3. Results and discussion
Figure.1 shows high resolution Annular Dark Field (ADF) STEM images observed along the Si[110] zone axis at accelerating voltages of 80 kV(a), 120 kV(c) and 200 kV(e), and the Fourier transform (b, d, f) of each ADF STEM image. Not surprisingly the 200 kV condition demonstrates the best result, but at 80 kV it is still possible to slightly-separate the silicon dumbbell structure. The Fourier transform image shows the 004 reflection spot of the silicon dumbbell and the 2-24 reflection spot, corresponding to 0.111 nm at 80 kV. The result at 80 kV reveals the potential for atomic visualization in carbon with minimal radiation beam damage. The Fourier transform image at 120 kV shows the 3-33 and 2-24 reflection spot, corresponding to 0.105 nm. In the 200 kV condition sub-angstrom resolution in the Fourier transform image is detected, with the 4-44 reflection spot (corresponding to 0.078 nm) and 3-35 reflection spot (corresponding to 0.083 nm). This result expands the possibility for structural characterization at the atomic level.

![Figure 1. ADF-STEM images observed along the Si[110] zone axis with accelerating voltage of 80 kV, 120 kV and 200 kV, and the corresponding Fourier transform of each image.](image)
Next, we observed an EELS energy time trace of the zero loss peak under the normal analysis condition. The beam current was measured, using a Faraday cup in the specimen holder, to be 29 pA in a 0.1 nm diameter probe at 200 kV, 27 pA in a 0.2 nm diameter probe at 120 kV and 5 pA in a 0.2 nm diameter probe at 80 kV. Figure 2 shows the EELS energy time trace for zero loss peak at 200 kV (a), 120 kV (b) and 80 kV (c) for 40 seconds duration. The time trace images give 800 EELS spectra, each of which is acquired with an exposure time of 50 ms (total exposure time is 40 seconds). The minimal fluctuation of the bright line, which is the zero loss peak, demonstrates the stability of the beam energy. Table 1 indicates the FWHM of fluctuation and stability by statistical processing (Stability = FWHM of fluctuation / Beam energy) of the zero loss peak in each line of Figure 2. The FWHM of fluctuation and stability is 146 meV, 0.73 ppm at 200 kV, 133 meV, 1.11 ppm at 120 kV and 121 meV, 1.51 ppm at 80 kV, respectively. The FWHM of zero loss peaks are 384 meV at 200 kV, 320 meV at 120 kV and 300 meV at 80 kV for an acquisition time of 1 second, which shows the inherent performance of the Cold FE gun (These spectra was smoothed) (Figure 3). Here, the 200 kV condition has the best result for stability. However, 80 kV shows the best result for FWHM.

### Table 1. Fluctuation of zero loss peak and stability of beam energy.

<table>
<thead>
<tr>
<th>Beam energy (keV)</th>
<th>Fluctuation (meV)</th>
<th>Stability (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>121</td>
<td>1.15</td>
</tr>
<tr>
<td>120</td>
<td>133</td>
<td>1.11</td>
</tr>
<tr>
<td>200</td>
<td>146</td>
<td>0.73</td>
</tr>
</tbody>
</table>

**Figure 2.** EELS energy time trace of zero loss spectra at 200 kV (a), 120 kV (b) and 80 kV (c) for 40 seconds duration.

**Figure 3.** The FWHM of zero loss peaks at 200 kV (a), 120 kV (b) and 80 kV (c) for 1s duration.

**Figure 4.** The FWHM of zero loss peaks at 80 kV for 1s (a), 20s (b) and 40s (c) duration.
Next, we have measured the FWHM over longer acquisition times. Figure 4 shows the FWHM of the zero loss peak at 80 kV for 1 second (a), 20 second (b) and 40 second (c) duration (These spectra was smoothed). FWHM of the zero loss peaks are 0.30 eV for acquisition time of 1 second, 0.327 eV for 20 second and 0.340 eV for 40 second, respectively. The result reveals the high stability and low energy spread performance of HD-2700 system. Figure 5 shows the Bright Field (BF) STEM image of graphene carbon sheet at an accelerating voltage of 80 kV, in which lattice fringes can be clearly seen. The atom to atom space of graphene carbon is precisely 0.142 nm. This result emphasizes that high resolution imaging and analysis are possible with minimal damage through the use of aberration-corrected lower-energy operation. Figure 6 shows the EELS spectrum of graphene carbon at accelerating voltages of 80 kV(a) and 200 kV(b). The spectrum at 80 kV clearly shows the characteristic five peaks showing bonding information. In comparison, the spectrum at 200 kV is rounded and shows a low peak to background ratio, which is attributed to radiation damage by the 200 kV electron beam.

**Figure 5.** BF-STEM image of graphene carbon sheet.

**Figure 6.** Comparison of EELS spectra accelerating voltage of 80 kV (a) and 200 kV (b).

4. Summary

We have shown high-resolution ADF STEM images and EELS data of graphene carbon using aberration corrected dedicated STEM. Sub-angstrom information, corresponding to 0.078 nm at 200 kV and a narrow energy spread of 0.3 eV in the FWHM of the zero loss peak at 80 kV is obtained under routine analysis conditions. We have also shown stability improvements in the instrumentation which will be a key parameter for chemical bonding state analysis in EELS at the atomic level.

References