Development of Monochromatic Analytical Electron Microscope Equipped with Higher-Order Aberration Corrector

Masaki Mukai, Eiji Okunishi, Akiho Nakamura, Takaki Ishikawa and Hidetaka Sawada

JEOL Ltd., 3-1-2 Musashino, Akishima, Tokyo 196-8558, Japan * Corresponding author: mmukai@jeol.co.jp

Monochromatic and aberration corrected electron microscope was developed in order to study an electronic structure of various materials, combining with a scanning transmission electron microscope (STEM) and an electron energy-loss spectroscopy (EELS) at atomic scale. [1] This microscope is equipped with a double Wien-filter monochromator, and obtained energy resolutions are selectable from 0.25 eV to 0.02 eV keeping the atom-sized electron probe on the specimen plane by the change of the width of the slit located between two filters. Since this microscope is equipped with the conventional third-order aberration corrector, the residual dominant geometric aberration was six-fold astigmatism. On the other hand, the higher-order aberration corrector was developed, which enable us to correct fifth-order geometric aberrations including six-fold astigmatism and to expand a uniform phase area of a Ronchigram in STEM to about 60 mrad in half angle even at lower accelerating voltages. [2-3]

We have developed a new monochromated analytical electron microscope equipped with the higher-order aberration corrector. Figures 1(a) and (b) show comparison of the Ronchigrams at 200 kV, when we used the higher-order and conventional third-order aberration correctors. Due to the correction of six-fold astigmatism by the higher-order corrector, the uniform phase area in the Ronchigram was extended to be 60 mrad as shown in Fig. 1(a). Figures 2(a), (b) and (c) show the raw high-angle annular dark-field (HAADF)-STEM images and their power spectra for sample of Si <110[×], obtained with monochromatic electron probes. The convergent semi-angles of the probe were ranged from 19.8 mrad to 47.3 mrad, and energy spread of that was 0.25 eV. The {440} reflections, which implies lattice spacing of 96 pm⁻¹, are present in the all power spectra of the recorded images, owing to correction of six-fold astigmatism by the higher-order aberration corrector suggests the possibility of the optical sectioning at sub-nanometer depth resolution in STEM. [4]

Figures 3(c)-(f) show the simultaneously acquired elemental maps of SrTiO₃ (100) by EELS and energy dispersive X-ray spectroscopy (EDS) at 200 kV, where the probe current and the dwell time per a pixel were 120 pA and 0.01 seconds. The obtained elemental maps of EELS and EDS exhibit atomic resolutions. Total solid angle for the EDS maps using two 100 mm² sized silicon drift detectors (SDD) was 1.75 sr. and their take-off angles of the SDD detectors were 21.5° and 18.3°. These results show high capability of the atomic resolution elemental analyses by EELS and/or EDS with the monochromatic electron microscope equipped with higher-order aberration corrector.

References

[1] M. Mukai, et al.: Microscopy, **64[3]**, (2015), 151-158.

- [2] H. Mueller, et al.: Microsc. Microanal., 12 (2006), 442-455.
- [3] T. Ishikawa, et al.: Microsc. Microanal., 13 (supple.3), (2015) 1599-1600
- [4] S. Pennycook : Ultramicroscopy, 180 (2017), 22-33



Figure 1. Comparison of the Ronchigrams at 200 kV, when we used (a) higher-order and (b) conventional third-order aberration correctors. The uniform phase areas in the Ronchigrams were (a) 60 mrad and 36 mrad in semi angle. The coefficients of six-fold astigmatism were (a) 122 μ m and (b) 1500 μ m.



Figure 2. Comparison of raw HAADF-STEM images and their power spectra of Si (110) at 200 kV on the convergent semi-angles of (a) 19.8 mrad, (b) 33.6 mrad and (c) 47.3 mrad. The energy spread of the monochromatic probe was 0.25 eV, and the probe currents were (a) 8 pA, (b) 21 pA and (c) 41 pA.



Figure3. Results of the simultaneous acquisition of atomic resolution HAADF images and elemental maps by EELS and EDS of $SrTiO_3 (100)$ at 200 kV, obtained with an electron probe of 120 pA probe current. (a) Raw HAADF-STEM image, (b) Raw HAADF-STEM image of the mapping area, the elemental maps of (c) Ti and (d) O obtained by EELS, (e) Sr and (f) Ti obtained by EDS and the spectra of (g) EDS and (h) EELS with an acquisition time of 0.01 seconds