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Resolution enhancement at a large convergence angle by a delta corrector with a CFEG in a low-accelerating-voltage STEM

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ABSTRACT

Resolution reduction by a diffraction limit becomes severe with an increase in the wavelength of an electron at a relatively low accelerating voltage. For maintaining atomic resolution at a low accelerating voltage, a larger convergence angle with aberration correction is required. The developed aberration corrector, which compensates for higher-order aberration, can expand the uniform phase angle. Sub-angstrom imaging of a Ge [1 1 2] specimen with a narrow energy spread obtained by a cold field emission gun at 60 kV was performed using the aberration corrector. We achieved a resolution of 82 pm for a Ge–Ge dumbbell structure image by high angle annular dark-field imaging.

1. Introduction

Observation at a low acceleration voltage is advantageous because of minimal damage to the specimens and a larger scattering cross-section resulting in a higher signal count for analysis in an electron microscope. The demand for an atomic-resolution instrument operating at a lower voltage is considerably increasing (Suenaga et al., 2009; Krivanek et al., 2010; Lee et al., 2012). However, it is difficult to perform high-resolution imaging because, at a low acceleration voltage, the longer wavelength of the electrons used for imaging increases the diffraction limit, so that the resolution worsens. The defocus spread (df_{Cc}) due to chromatic aberration with an energy deviation of dE is defined as $df_{Cc} = Cc{\cdot}dE/U$, and a blur due to the chromatic aberration d_{Cc} is expressed as $d_{Cc} = Cc{\cdot}dE/U{\cdot}\alpha$, where U is the accelerating voltage and α is the convergence semi-angle. The blur causes a severe resolution reduction in a low-voltage microscope, because ratio dE/U increases with a decrease in U, which in turn causes d_{Cc} to increase. For maintaining atomic resolution at a low accelerating voltage, a larger convergence angle with aberration correction and a narrow energy spread of the source to reduce the blur are required.

We have developed an electron microscope having higher-order aberration correctors, which are called delta correctors (Sawada et al., 2009a). The delta corrector system with three dodecapoles can compensate for a 3rd order spherical aberration and a six-fold astigmatism. The developed microscope achieved high resolution at an accelerating voltage lower than 60 kV, by using a narrow energy spread obtained by a cold field emission gun (CFEG) (Kohno et al., 2010). The atomic-resolution imaging of a Si [1 1 0] specimen and a gold polycrystalline particle at 30 kV and 60 kV has been demonstrated by Sasaki et al. (2010). The probe sizes at the low accelerating voltage have been evaluated using the experimental data of a dark-field image obtained using a scanning transmission electron microscope (STEM) by comparing with the simulations (Sasaki et al., 2012). By using this instrument, the chemical information at atomic resolution is obtained by using electron energy loss spectroscopy (EELS) in the STEM (Suenaga et al., 2009; Suenaga and Koshino, 2010; Suenaga et al., 2011).

In this article, sub-angstrom imaging using the developed microscope is demonstrated at a low acceleration voltage. To obtain a smaller probe to attain higher resolution, we optimized the convergence angle for STEM imaging and the Gaussian probe size for the specimen. For sub-angstrom-resolution imaging, we have used a germanium (Ge) crystalline specimen because the image in the [1 1 2] orientation shows a pair of two adjacent Ge atomic rows (dumbbell) separated by 82 pm, according to the geometrical configuration (O'Keefe et al., 2005).

2. Experimental set-up

An operating accelerating voltage of 60 kV was used in the experimental test. We used a delta corrector equipped with a triple dodecapole and two sets of transfer lenses, and Ronchigrams were recorded for the evaluation of the spherical aberration corrector

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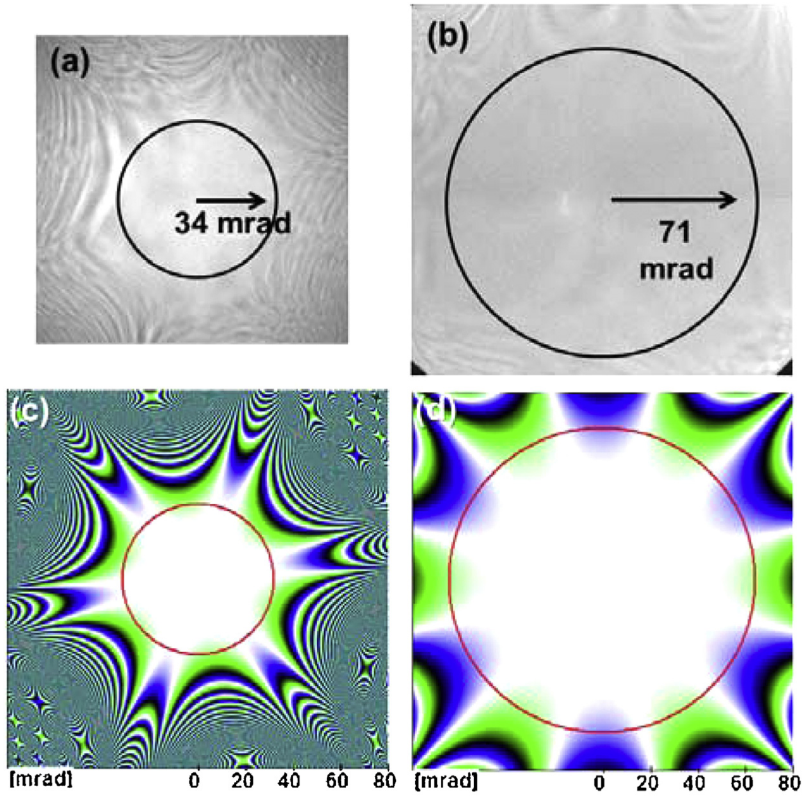

Fig. 1. Experimental Ronchigrams obtained using the (a) double-hexapole-type and (b) delta correctors at 60 kV. Calculated phase with (c) A_6 = 2 mm and fifth-order spherical aberration = 1.5 mm and (d) A_6 = 0.05 mm at 60 kV. White to black corresponds to π, and white to white corresponds to 2π. Circles describe the $\pi/4$ limit.

(Cs corrector) (Sawada et al., 2009a). To compare the aberration correction capability of the double-hexapole-type corrector and the delta corrector, Cs compensation using a double-hexapole system was tested. The contrast flat area of the Ronchigram reached 34 mrad with the correction using two hexapole fields, as shown in Fig. 1(a). The coherently converged angle was limited by the six-fold astigmatism (A_6). Next, the delta corrector was used to correct the geometric aberrations, as shown in Fig. 1(b). For the delta optical system, the contrast flat area of the Ronchigram increased to 71 mrad. A_6 was measured to be less than 0.05 mm from the experimental Ronchigram (Sawada et al., 2008). Fig. 1(c) and (d) shows the calculated phase with A_6 = 2 mm and 0.05 mm, respectively. The values of the $\pi/4$ limit, where the phase shift from the center becomes $\pi/4$ because of aberration, are 32 mrad (151 pm)$^{-1}$ and 65 mrad (75 pm)$^{-1}$, respectively. With the delta corrector, a convergence angle of over 40 mrad is adaptable for high-resolution imaging using the STEM.

The microscope was equipped with a CFEG having a tungsten [3 1 0] tip to obtain a small energy spread of the source. The energy spread is varied by changing the extractor voltage. The full width at half maximum (FWHM) of the electron energy was measured at several settings, as shown in Fig. 2. In present observation, the energy spread of the source has an FWHM of 0.36 eV, after setting the probe current.

3. Simulations

The probe size was calculated as a function of the convergence semi-angle at 60 kV. The simulation was based on the incoherent superposition method, using experimental optical parameters. The chromatic aberration was 0.84 mm, and the energy spread of the CFEG was 0.36 eV in this calculation. The Gaussian probe size of the specimen was set to be 14 pm. Here, D59 denotes the diameter of the electron beam that includes 59% of the total beam current (Haider et al., 2000). Notations D50, D75, and D90 are equivalent terms. The FWHM of the calculated probe monotonically decreases with an increase in the convergence semi-angle because of a smaller diffraction limit at a higher angle. On the other hand, D50, D59, D75, and D90 increase at angles over 40–50 mrad due to the blur caused by the chromatic aberration $d_{Cc} = Cc{\cdot}dE/U{\cdot}\alpha$, even though the probe-forming system has a small energy spread by CFEG and a small amount of a chromatic aberration coefficient. D50 has a minimum value at a semi-angle between 35 mrad and 45 mrad, whereas the value of D59 is minimum at a semi-angle between 30 mrad and 40 mrad. When a small D59 value is obtained, the signal-to-noise (S/N) ratio of the image is better because the tail of the probe is weak. On the other hand, a higher angle of over

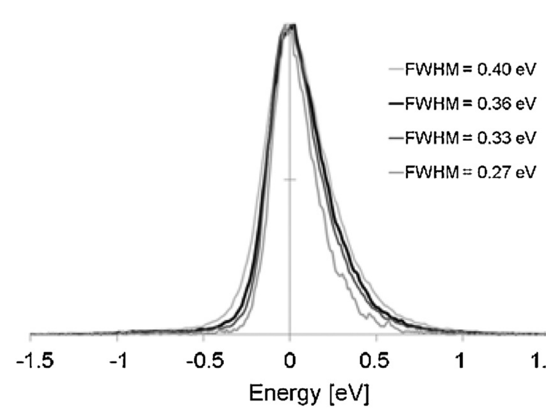

Fig. 2. Measured energy spreads of the CFEG at 60 kV by varying the extractor voltage. Note that the exposure time = 0.1 s, and dispersion = 0.01 eV/channel using the Gatan Quantum ER with an entrance aperture of 1.5 mm.

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