

Structural and elemental analysis under the sub-Angstrom resolution with Cs-corrected STEM

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Recently, scanning transmission electron microscopy (STEM) has been progressed owing to a stable field emission gun (FEG) [1] and sophisticated spherical aberration corrector (Cs-corrector) [2], which is now becoming widely spread as a commercial instrument. In the STEM, a high angle annular dark-field (HAADF) image is formed with incoherently scattered electrons [3] and it provides a contrast sensitive to the chemistry, which is known as Z-contrast; therefore an image interpretation is easier than in conventional bright-field high resolution transmission electron microscopy (HRTEM). The Cs-corrector for probe forming lens system enables to probe a specimen sub-Angstrom size, resulting in the sub-Angstrom resolution imaging. And also, a higher intensity of the probe owing to the Cs-corrected STEM provides an elemental analysis at atomic scale resolution with good signal-to-noise ratio.

In this paper, high-resolution structural and elemental analysis were performed on Fe₂O₃ particles and a SrTiO₃ perovskite single crystal, with a Cs-corrected STEM. The instrument used for this experiment was the JEM-ARM200F (Atomic Resolution analytical Microscope, 200 kV FEG TEM/STEM) equipped with Cs-corrector for probe forming lens system. The microscope was equipped with the analytical instruments, such as a STEM bright-field (BF) detector, a dark-field (DF) detector, an EDS detector and an electron energy-loss spectrometer (EELS, Gatan Enfina).

Figure 1 shows a comparison of BF (a) and DF (HAADF) (b) images of SrTiO₃ oriented [100] and atomic model (c) of the perovskite structure. The probe diameter was 0.08 nm and both BF and HAADF images were acquired simultaneously. The inset on (b) shows atomic model at the same scale and the same orientation. The BF image shows dark contrast at atom positions of Sr and Ti-O, however, it is difficult to identify the elements from the image contrast directly. The HAADF image shows bright contrast at atom positions, and heavier atom of Sr were shown in brighter contrast. It is easy to determine the positions of Sr atoms from the contrast directly. Figure 2 shows HAADF images (a) & (b), and elemental maps (d) – (f) of SrTiO₃. The conditions for this experiment were as follows: probe diameter : 0.15 nm, beam current : 150 pA, dwell time : 0.03 sec/pix and frame acquisition time : 3.5 min. The HAADF image (b) and EELS spectra were acquired simultaneously. Elemental maps were reconstructed from an EELS Spectrum Imaging (SI) data cube. The maps show that the detected elemental signals show a good S/N ratio and Sr, Ti-O and O positions (atom columns) are clearly resolved at atomic resolution. Figure 2(c) shows a color-reconstructed elemental map, where each element of the atomic column is displayed in a different color. It is quite easily understandable.

According to this experiment, it is ascertained that the Cs-corrected STEM is able to realize imaging and elemental analysis at atomic resolution.

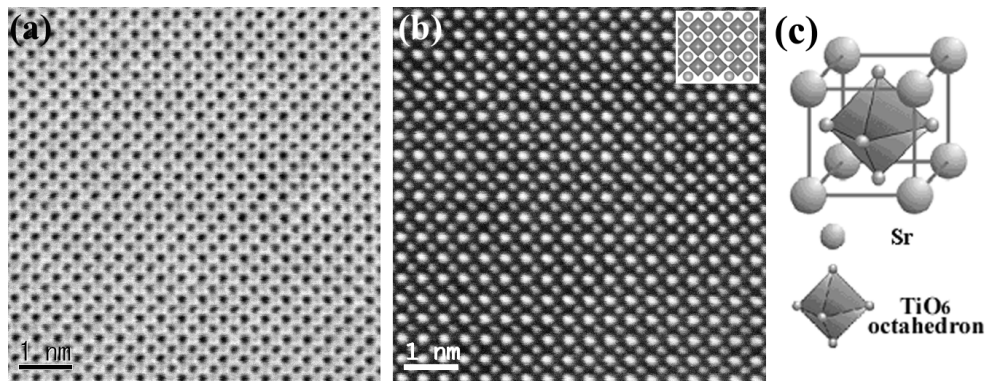


Figure 1. A comparison of STEM BF-image (a) and DF-image (b) and atomic model (c) of $SrTiO_3$. Both images were acquired simultaneously. Probe diameter was 0.08 nm. The inset on (b) shows atomic model at the same scale and orientation as the STEM images.

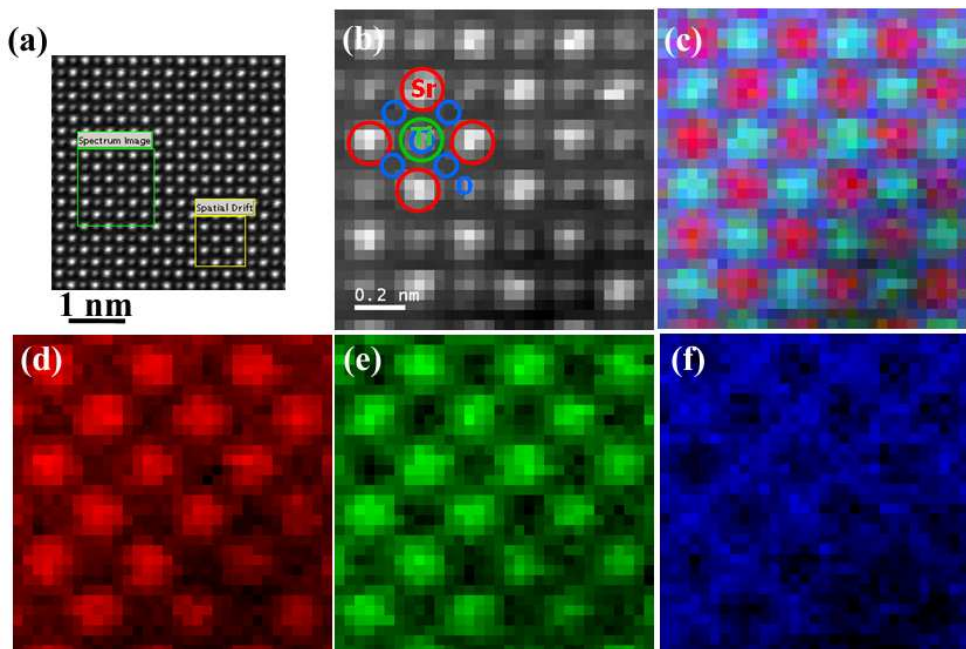


Figure 2. STEM HAADF images and elemental maps of $SrTiO_3$. Probe diameter : 0.15 nm, beam current : 150 pA, dwell time : 0.03 sec/pix.

- (a) HAADF image showing an acquisition area for Spectrum Imaging (SI) (green square) and an area for specimen drift monitor (yellow square).
- (b) Enlarged HAADF image at the same magnification as elemental maps. The atom positions are indicated in the figure.
- (c) Color reconstructed elemental map. Red corresponds to Sr column, light blue (mix of oxygen (blue) and titanium (green)) to $Ti-O$ and blue to O .
- (d) Sr map reconstructed from an EELS Spectrum Imaging (SI) data cube.
- (e) Ti map from the cube.
- (f) O map from the cube.

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