

Atomic Resolution Elemental Map of EELS with a Cs Corrected STEM

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Z-contrast imaging by high angle annular dark field (HAADF) technique is a powerful method to determine positions of atoms or atomic columns in an atomic level resolution. Because the Z-contrast imaging method provides atomic number contrast, it cannot specify atomic species. For the determination of element, energy dispersive X-ray spectrometer (EDS) or electron energy loss spectrometry (EELS) is usually used with scanning transmission electron microscope (STEM). EELS is preferable for STEM because of its high-sensitivity of detection and its capability to analyzing the density of states.

Recently, the performance of STEMs is greatly improved by the realization of Cs corrector for the probe forming system. The Cs corrector mainly offers two advantages: a small probe size and high current probe density. As to the former advantage, the smallest probe size was measured to be less than 0.1 nm at 200kV with Cs corrector. This advantage of the small probe size, which serves as a high-resolution capability in imaging, has already been reported by the Pennycook's group [1, 2]. As to the latter advantage, the current probe density is about ten times higher than the one in ordinary STEMs. The higher current probe density enables us to analyze with higher sensitivity reaching an atomic level resolution. Point and/or line analyses with this higher current probe density have already been [3, 4, 5]. The present paper reports atomic resolution elemental maps by the combination of EELS and Cs-corrected STEM.

The sample used in this experiment was Si₃N₄ single crystal. A thin film specimen was prepared by argon ion milling. Image observations and EELS analysis was carried out by a JEM-2100F (JEOL) equipped with a STEM Cs corrector (CEOS GMBH) and a parallel EELS (GATAN Enfina). The probe size was less than 0.1 nm and the probe current was 10pA. EEL spectra were corrected on 50 x 45 pixels and the acquisition time for each spectrum was 0.02 sec. EELS maps were reconstructed from the accumulated spectra on all pixels after the recipe of EELS quantitative analysis such as background subtraction.

Figure 1(a) shows the HAADF image from the specimen. The area framed with a rectangle shows an image obtained from the intensity of EEL spectrum on each pixel whose intensity was one integrated over the entire spectrum. The integrated intensity of each element was obtained after the background was subtracted from the measured spectrum as shown in fig. 1(b). The latter image also shown in an atomic level resolution, and the atomic sites in the latter image well agree with those in the former HAADF image. Figure 2 (a), (b) and (c) show a HAADF STEM image, and the silicon and nitrogen map, respectively. The silicon map clearly shows the higher intensity at a silicon atom site, whereas the nitrogen map shows higher intensity around a silicon atom site. This report clearly demonstrates that the determination of atomic species in each atomic column can be made by using EELS with a Cs corrected STEM.

[1] P.D. Nellist et al., *Science* **305**, 1741 (2004)

[2] E. Abe et al., *Nature* **421**, 347 (2003)

[3] A. R. Lupini and S. J. Pennycook, *Ultramicroscopy* **96**, 313 (2003)

[4] L. J. Allen et al., *Phys. Rev. Lett.* **91**, 105503 (2003)

[5] M. Varela et al., *Phys. Rev. Lett.* **92**, 095502 (2004)

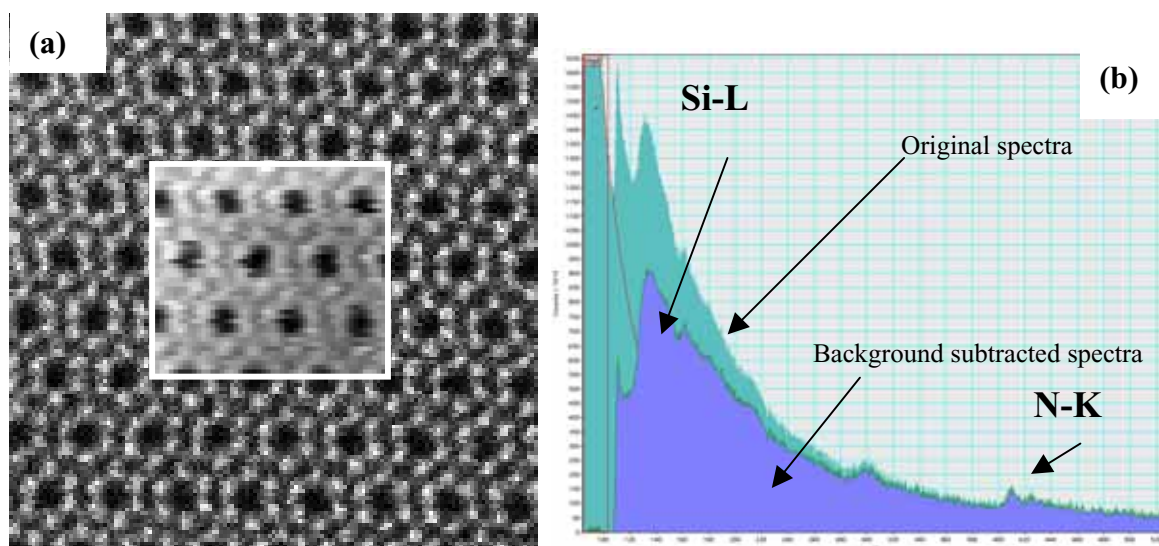


Fig. 1 STEM HAADF image and EEL spectrum of Si_3N_4

- (a) High-resolution HAADF image with an EEL spectrum image (inside a white rectangle area), whose intensity was obtained by integration over the entire spectrum.
 (b) EEL spectrum.

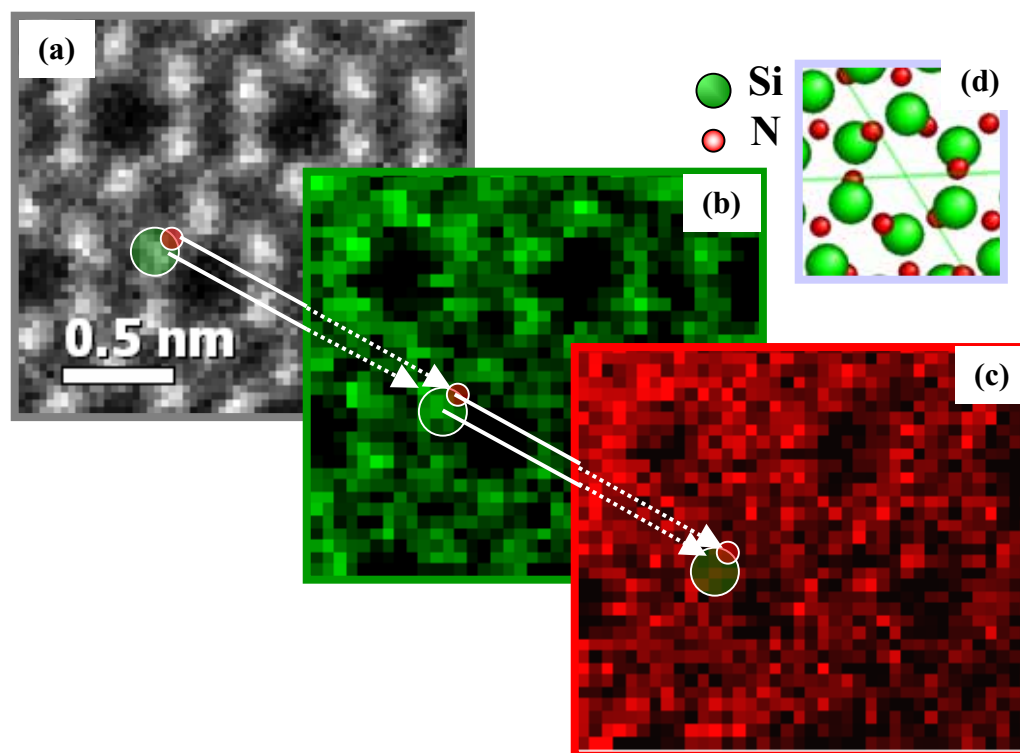


Fig. 2 STEM HAADF image and EEL spectrum maps of Si_3N_4

- (a) STEM HAADF image.
 (b) Si-L map
 (c) N-K map
 (d) Crystal structure model of $\text{Si}_3\text{N}_4(001)$