

Near Shadowless EDS Tomography for Sliced Sample Realized by X-ray Collection with One Large Sized SDD Detector

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Three-dimensional (3D) elemental mapping by energy dispersive X-ray spectroscopy (EDS) is getting popular for characterizations of samples having 3D structures to be solved such as semiconducting devices or blended polymers, since the method enables us to see the atomic species and 3D distribution of sample simultaneously. For the reconstruction of the 3D elemental maps, the EDS tomography is developed combining electron tomography and the two dimensional (2D) elemental mapping by EDS [1]. In the previous X-ray detection system composed of two EDS detectors, the detectors locates symmetrically with respect to the tilt axis of the sample holder. Therefore, some portion of the generated X-rays are blocked by the sample holder or supporting mesh in specific tilting angle range. The shadowing on the configuration may make artifacts in resulting 3D elemental maps. In order to avoid this problem, it is necessary to correct the measured intensity of EDS maps after the acquisition, according to the expected or pre-measured detection efficiency.

Recently, we developed an X-ray detection system with a new EDS-detector configuration for EDS tomography shown in Fig. 1. One detector is located on the tilting axis of sample holder (SDD2), and the other is in the right side of the tilting axis (SDD1). It was expected that SDD2 had no shadowing effect in EDS tomography. Therefore, we tried to use the SDD2 for realizing near shadowless EDS tomography.

The microscope for our experiment was aberration corrected 300 kV TEM (JEM-ARM300F) equipped with two SDDs (SDD1 and SDD2). The solid angles of SDD1 and SDD2, whose detection areas are 158 mm², are 1.106 and 1.108 sr [2]. The sample for the test was selected to be a sliced paint film prepared by microtomy, mounted on 3 mm^φ grid. Figure 2(a) shows an HAADF-STEM image of the sample, which contains the titania, silica, alumina and iron oxide. A tilt series of EDS maps from -60 to +60 degree with a 5 degree increment was acquired with an automated software of tomography (SIF Ltd., Tomography). The size of each EDS map was 256 x 256 pixels. A shot of reconstructed 3D elemental map for the sample is shown in Fig. 2(b). The used 3D reconstructions algorithm for this reconstruction was simultaneous iterative reconstruction technique (SIRT).

The measured total intensity of Ti K α from an EDS map detected by the SDD2 is plotted on tilting angles of the sample in Fig. 3. The intensity from Ti K α keeps at almost constant ($\approx \pm 12\%$) over the sample tilting range. This result shows that the EDS detector located at the new position had almost no blocking of X-ray from the holder and the grid of the sample. It can be said that we had built a near shadowless EDS tomography system, which is realized by using the single EDS detector located on the holder tilting axis. In addition to that we mentioned above, it is worth to mention that the EDS system with two large sized SDDs can collect doubly-high X-ray signal with no shadowing than the case of single SDD, if we prepare rod-shape samples.

References:

- [1] G. Mobus *et al*, *Ultramicroscopy* **96** (2003) p. 433.
 [2] I. Ohnishi *et al*, *Microsc. Microanal.* **22(3)** (2016), p. 318.

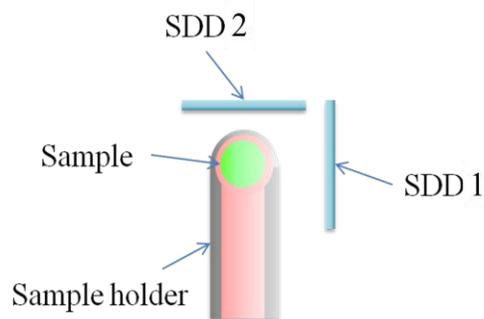


Figure 1. Top view of detector arrangement. One EDS detector located on the tilting axis of sample holder (SDD2) and the other is located at the right side of the holder tilting axis (SDD1).

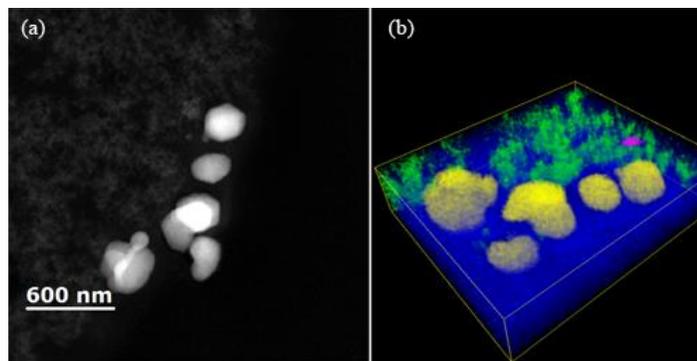


Figure 2. (a) An HAADF image of the paint film, taken by JEM-ARM300F equipped with the EDS detector, whose detector size is 158 mm². (b) The 3D image was reconstructed from the sample shown in (a). Colors indicate atomic species: yellow (Ti), green (Si), magenta (Fe) and blue (C).

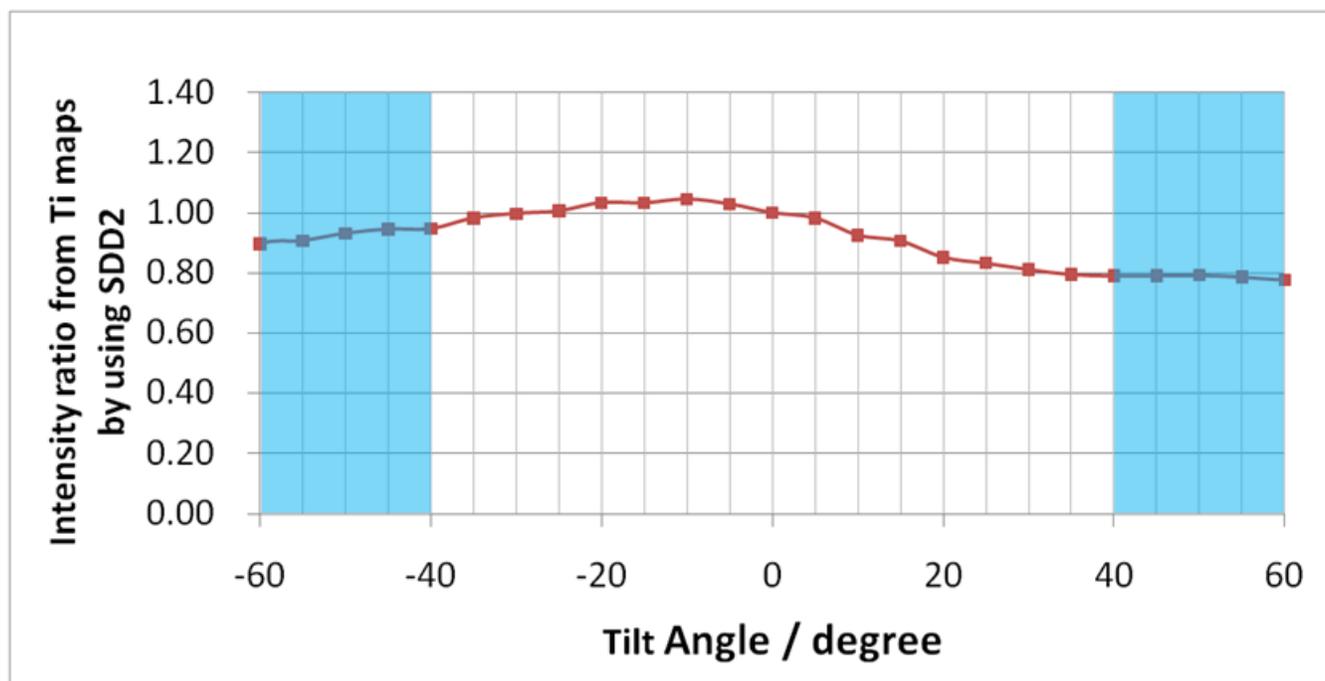


Figure 3. The total intensity ratio of TiK α from a map with respect to one at the tilt 0 degree plotted, depending on tilt angles of the sample (paint film). The data was taken by the single large sized SDD located on the tilting axis sample holder, which was installed in JEM-ARM300F.