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Practical Measurement of X-ray Detection Performance of Large-Angle Silicon Drift Detectors Toward Quantitative Analysis in the Newly Developed 300 kV Aberration-Corrected Grand ARM

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X-ray analysis in (scanning) transmission electron microscopes (S/TEMs) has been rather limited due to the miserable signal availability in comparison with electron energy-loss spectrometry (EELS). However, X-ray analysis is brought back into the attention because of relatively simple nature and robust aspects, especially after the aberration-corrected STEM instruments become available. Additionally, the recent silicon drift detector (SDD) technology offers more flexible detector-instrument configurations, so that the limited availability of X-ray signals can be improved significantly. The latest aberration-corrected 300-kV JEOL Grand ARM instrument has been developed to achieve the best image resolution and analytical spatial resolution. The Grand ARM is equipped with two large solid angle SDDs: SDD1 is at the traditional geometry (perpendicular to the specimen-holder rod axis) with the take-off angle of 25 degree and SDD2 is placed at the new configuration (along with the holder rod axis) with the take-off angle of 29 degree. The pole-piece is designed to maximize X-ray collection by positioning 2 SDDs as close as possible to the optical axis. In this study, X-ray collection performance of 2 SDD systems was systematically measured and atomic-resolution X-ray analysis was also performed.

For measurement of X-ray detection performance, the NiOx thin film was used [1]. Figure 1 compares two X-ray spectra from the NiOx film measured by the two SDDs in the Grand ARM at 300 kV. The SDD2 with the new geometrical configuration shows better collection efficiency. By comparing X-ray spectra obtained from the previously calibrated HB 603 STEM, the collection angles of two detectors were determined. The conventional geometry SDD1 exhibits 0.593 sr whereas the new configuration SDD2 shows 0.994 sr. When two SDDs are in the Grand ARM, X-ray signals can collected at the solid angle of 0.5π sr, which is equivalent to 25% of the half sphere above the specimen. Using the NiOx film, the peak-to-background (P/B) ratio in Fiori definition and inverse hole count (IHC) of two SDDs were also measured in the Grand ARM and are compared with those in 300 kV HB603 and 200 kV aberration-corrected STEMs (Fig. 2). Although the P/B ratio of the both SDDs in the Grand ARM is comparable to that in other aberration-corrected instruments, the IHC values are superior to those in other instruments. Figure 3 summarizes the holder tilt-angle dependence of the P/B ratio measured in ARM. It should be noted that the positive x-tilt is toward the SDD1 and the positive y-tilt is towards the SDD2. The P/B ratios plotted against the x-tilt and y-tilt are shown in Fig. 3(a) and (b), respectively. Fig. 3(c) shows the P/B as a function of both x and y tilts, which means that the specimen is tilted between the both SDDs in positive direction. The SDD2 exhibits lower P/B values than the SDD1 but less sensitive to the specimen tilt angle. Although the P/B values in the SDD2 is slightly degraded, the SDD2 shows higher solid angle than the SDD1, which implies that this new geometry can improve X-ray collection efficiency.

A set of X-ray maps obtained from $SrTiO_3$ using the Grand ARM is shown in Fig. 4. The maps with 256x256 pixels were acquired only for 2.5 min in total with the probe current of 80 pA.

These atomic-resolution X-ray maps can be acquired with the limited current for very short acquisition time, which can be beneficial for characterization of irradiation sensitive materials.

References

[1] R.F. Egerton & S.C. Cheng, *Ultramicrosc*. **55** (1994), 43.

[2] The author (MW) wishes to acknowledge financial support from the NSF through grants DMR-0804528 and DMR-1040229.

Fig. 1

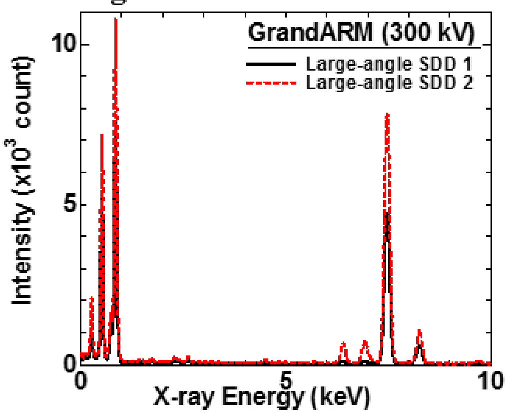

Fig. 2

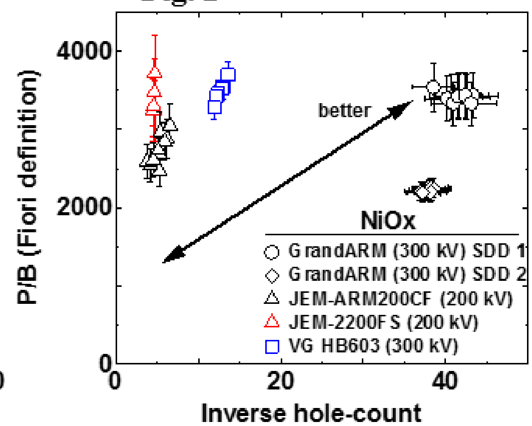

Fig. 3

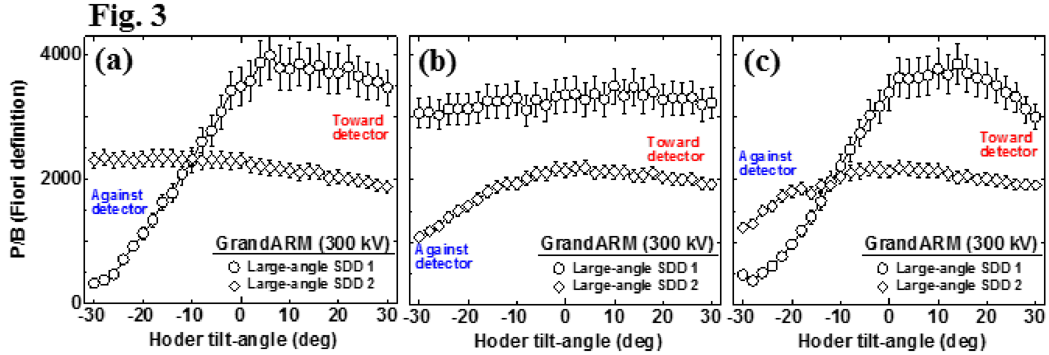

Fig. 4

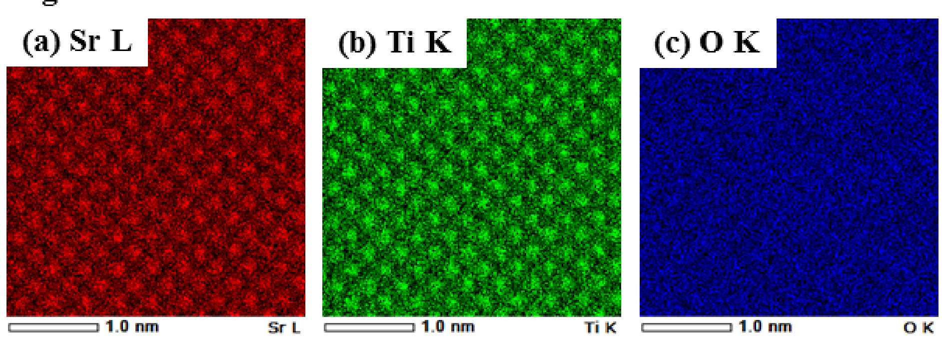

Figure 1: X-ray spectra from NiOx measured by two SDDs in Grand ARM.

Figure 2: P/B plotted against IHC measured in different instruments, including Grand ARM.

Figure 3: Tilt angle dependence of P/B measured in Grand ARM: (a) x-, (b) y- and (c) x/y-tilts.

Figure 4: Atomic-resolution X-ray maps measured from $SrTiO_3$ in Grand ARM.