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Practical Measurement of X-ray Detection Performance of a Large Solid-Angle Silicon Drift Detector in an Aberration-Corrected STEM

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X-ray analysis especially in (scanning) transmission electron microscopes (S/TEMs) is a very robust approach and has been widely used for ~ 40 years. However, X-ray analysis is not very efficient since availability of signals is very limited due to (1) a small analyzed volume (which is preferred for improved spatial resolution conversely), (2) physically restricted signal-generation and (3) poor signal-detection configurations. The analyzed volume is even more limited when a refined incident probe is used in the latest aberration-corrected STEM. The signal-generation efficiency may not be improved dramatically unless much higher probe currents are applied (which may induce radiation damage) or thicker specimens are analyzed (which obviously degrades spatial resolution). In order to compensate for the poor signal-detection, there have been several attempts to redesign X-ray detector configurations by modifying detector locations [e.g. 1], arraying multiple detectors [2] and employing large solid-angle detectors [3], etc. Recently, the latest aberration-corrected STEM JEOL JEM-ARM200CF equipped with a large solid-angle silicon-drift detector (SDD) Centurio (nominally 0.8 sr) has been installed at Lehigh. In this study, X-ray detection performance was systematically measured in this new instrument and compared with that in VG HB603 aberration-corrected STEM, which is optimized for X-ray analysis.

For measurement of X-ray detection performance, two standard thin specimens were used: one is the NiOx thin film [4] and other is the well-characterized NIST standard reference material (SRM) 2063a [5]. Note that all parameters related to X-ray detection performance in the JEM-ARM200CF were determined at 200 kV with a hard X-ray aperture inserted. Using the NiOx film, the peak-to-background (P/B) ratio in Fiori definition and inverse hole count (IHC) were measured at different process-time setting of the detector and are compared with those in 300 kV HB603 and 200 kV JEM-2200FS aberration-corrected STEMs (Fig. 1). Although the IHC values in the ARM are lower than those in HB603, the P/B ratio in ARM is in the same range both in HB603 and 2200FS. Figure 2 shows holder tilt-angle dependence of the P/B ratio measured in ARM. In this instrument, the Centurio detector is located perpendicular to the rod axis of specimen holder. When the specimen is tilted toward the detector (x positive direction), the P/B ratio decreases slightly. However, the value is drastically dropped if the specimen is tilted against the detector. Conversely, the P/B ratio is not very sensitive to the specimen tilt along y-axis in this tilt-angle range, which can be expected from the detector-column configurations.

Using the SRM2063a specimen, ζ-factors (which are the sensitive factors for quantitative X-ray analysis of thin specimens) were measured [6]. During the ζ-factor determination, thicknesses of the detector window materials can be determined. Based on the thicknesses of the window materials, both ζ-factors in the whole range and the detector efficiency can also be estimated [6], which are plotted against the X-ray energy in Figs. 3 and 4, respectively. It should be mentioned that the decrease of the detector efficiency after ~10 keV is due to the shorter crystal thickness of the SDD. The ζ-factors determined in ARM are compared with those in HB603 in Fig. 3. The ζ-factor values in ARM are ~1/3 in HB603, which indicates that the X-ray collection efficiency is ~3 times higher in ARM than in HB603. From the reported value of the X-ray collection angle in

HB603 [7], the collection angle of the Centurio detector in ARM was estimated as ~0.53 sr by taking into account the difference in the ionization cross-section at different kV.

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Fig. 1

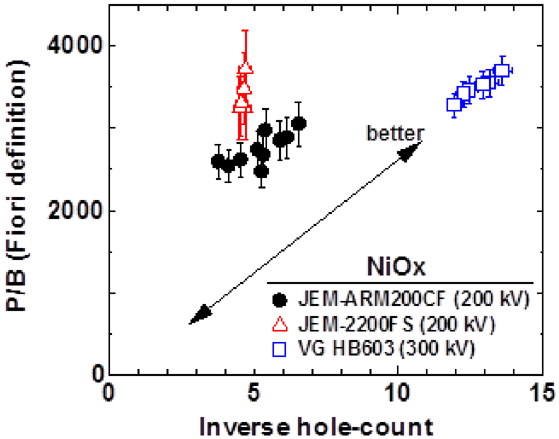

Fig. 2

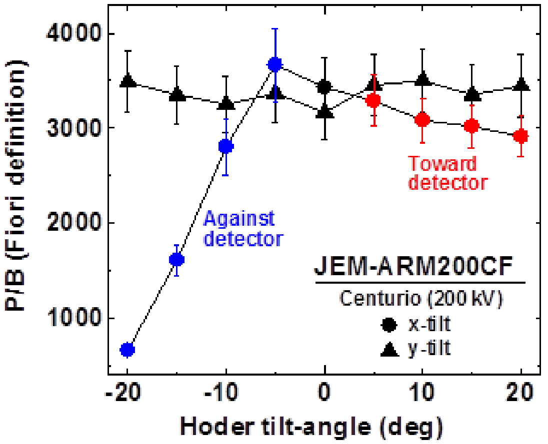

Fig. 3

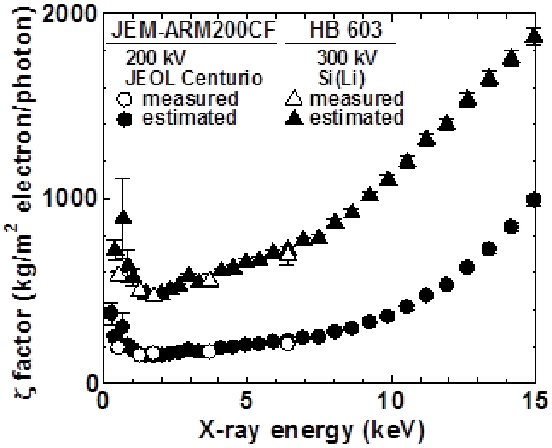

Fig. 4

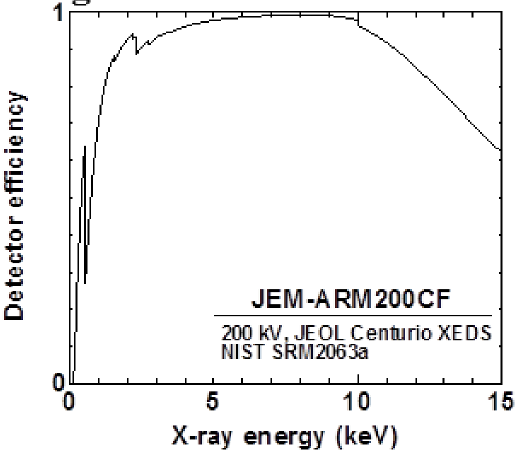

Figure 1: P/B plotted against IHC measured in different instruments, including ARM200CF.

Figure 2: Tilt angle dependence of P/B measured in ARM200CF.

Figure 3: Determined and estimated ζ-factors using the NIST SRM2063a thin film [6].

Figure 4: Detector efficiency of the Centurio determined after the ζ-factor estimation [6].