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Performance of a Silicon-Drift Detector in 200kV TEM Environments

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Silicon-drift detectors for energy-dispersive spectroscopy have gained increasing acceptance in the field principally because of their outstanding capabilities for high count-rate performance coupled with excellent resolution [1,2]. Improvements in design in the last few years have overcome some early disadvantages of the SDD in comparison to Si(Li) detectors, which have dominated the field for nearly 4 decades. Amongst these are detection efficiency in the low-energy regime, and a drop-off in detection efficiency at higher energies above 10keV. The former disadvantage has been largely overcome by generational design improvements to minimize the effective "dead" layer at the entrance window, so that present detectors provide better performance in the low-energy regime than Si(Li) detectors. In the latter case, higher energy peak detection performance has been improved by increasing the thickness of the detector. This is important primarily for the use of the SDD in a TEM environment, where very high count rates are not encountered due to the thin sample geometry. Also of importance for incorporation of an SDD into a TEM column is the lack of need for liquid nitrogen, since the SDD is chilled by a thermoelectric cooler. This is an advantage for SEM and microprobe operation also, but high-resolution TEMs (particularly aberration-corrected instruments) are more sensitive to vibration than SEMs or microprobes, so the much reduced form factor of an air-cooled SDD relative to a Si(Li) detector with a large LN2 dewar is desirable for use on the TEM. We have recently tested two Bruker Co. SDDs on our 200kV TEMs; the following examples illustrate some of the performance details.

A prototype Bruker 4030 AXS 30mm^2 SDD detector, with a collection angle of ~0.12sr, was installed on a JEOL 2010F TEM at The Dow Chemical Co in 2007. The initial design introduced a beam deflection when the detector was inserted which affected alignment; this problem was corrected in a detector re-design. An improved Bruker SDD with similar detector geometry was installed later on an aberration-corrected (corrector on the probe-forming lenses) JEOL 2200FS instrument at ORNL. Figure 1 shows the latter detector mounted on the column. An example of the light element mapping capability of the SDD, recorded on the Dow 2010F TEM, shows the interface phase between boron carbide and an aluminum boron carbide phase in a ceramic material (Figs. 2a-d). The spectrum image was collected at 1.95nA probe current, with a nominal 0.7nm probe diameter. A primary requirement for the aberration-corrected instrument was that the detector would in no way compromise the imaging operation of the microscope. Figure 3 shows a pair of image Ronchigrams (beam at crossover on amorphous carbon), after a full aberration correction procedure was performed. The Ronchigrams were recorded, respectively, with the detector Out and Off vs with the corrector In and On at maximum cooling level. No differences in Ronchigram character or in the quality of the aberration correction were detectable in these tests, or in several other test conditions using Ronchigrams. In like fashion, Figs. 4a and 4b show HA-ADF images of a strontium titanate crystal in a <100> zone axis orientation, with the SDD Out and Off (4a) vs In and On at maximum cooling (4b). No changes in focus were required between the images, and no effects of the detector condition were seen in the image resolution. These tests confirm that the SDD can be effectively operated in a microscope designed to provide sub-Ångström image resolution, such as the JEOL 2200FS [3].

References

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[2] D.E. Newbury, Proc. Micros & Microanalysis 2006; V12, Suppl 2; Cambridge University Press, p.1380.

[3] This research at the Oak Ridge National Laboratory's High Temperature Materials Laboratory was sponsored by the U. S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Program, and by The Dow Chemical Company.

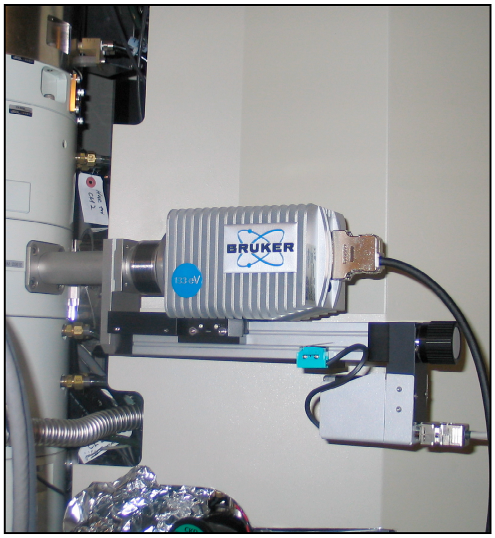


Fig. 1. Bruker-AXS silicon drift detector installed on JEOL 2200FS aberration-corrected STEM/TEM.

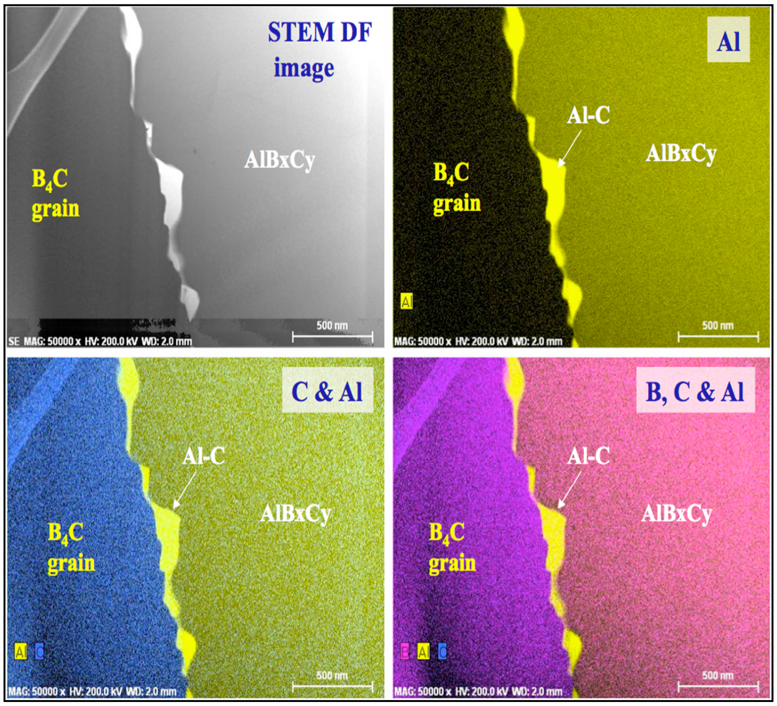
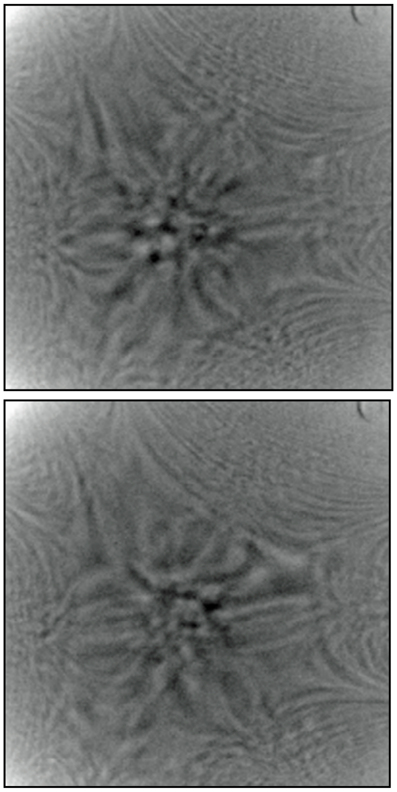


Fig. 2. a) STEM ADF image of interface in an Al-B-C ceramic, showing a thin Al-rich phase in high contrast between a B_4C grain and an AlBxCy grain; b-d) element maps as indicated.



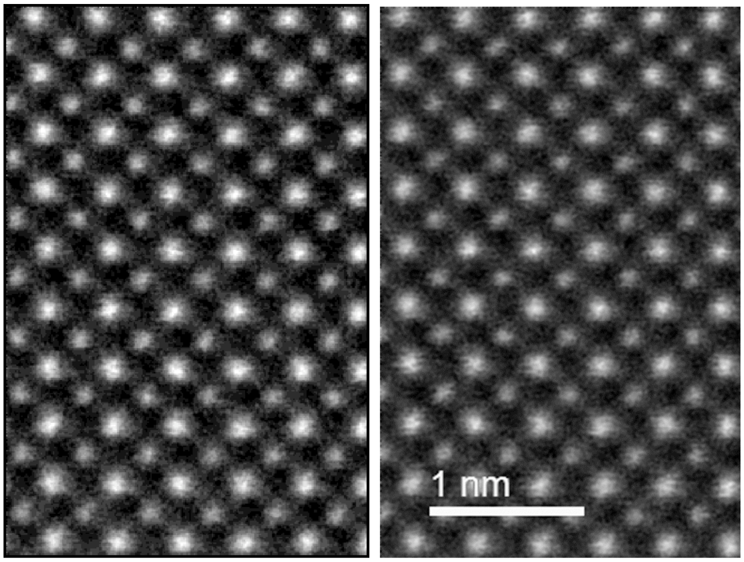


Fig. 3. Ronchigrams showing no discernible change in corrector alignment between SDD Out and Off (top) vs In and MaxCool (bottom).

Fig. 4. HA-ADF images of SrTiO3 in <100> zone axis orientation, showing equivalent high resolution with SDD Out and Off (left) vs In and MaxCool (right).