An Integrated Multiple Silicon Drift Detector System for Transmission Electron Microscopes

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Abstract. A new EDX system, consisting of multiple SDDs has been developed for an FEI 200kV TEM/STEM in which the SDDs, designed by PN Sensor with a total collection angle approaching 1 srad has been obtained by placing 4 SDD's symmetrically around the electron beam axis in the objective lens chamber. The massive increase in solid angle of collection compared to previous designs in S/TEMs leads to a huge reduction in the time for EDX mapping. First results from the detector are reported.

1. Introduction

Silicon Drift Detectors (SDD) are rapidly replacing Si(Li) detectors for EDX microanalysis in SEM, but have yet to have an impact in the S/TEM world. Main reason for this difference is the low X-ray count rate created by thin S/TEM samples compared to the bulk samples in SEM. These low count rates make EDX mapping a very slow process in S/TEM. However, the introduction of probe correctors and more recently the introduction of higher brightness electron sources has led to significantly increased beam currents in small electron probes and, potentially, to higher EDX count rates. Since a key advantage of the SDD is the high count rate capability, the throughput improvement compared to the Si(Li) detectors will be considerable in these new instruments. In addition, since many materials are easily damaged by fast electrons it is essential to improve the collection efficiency of the X-rays generated, in order to reduce the dose on the sample.

2. EDX detector design

For the above reasons a new EDX system (patents pending), consisting of multiple SDDs has been developed for an FEI 200kV TEM/STEM in which four SDDs, designed by PN Sensor with a total active area of 120 mm^2, are placed symmetrically around the electron beam axis in the objective lens chamber. The design is shown schematically in figure 1.

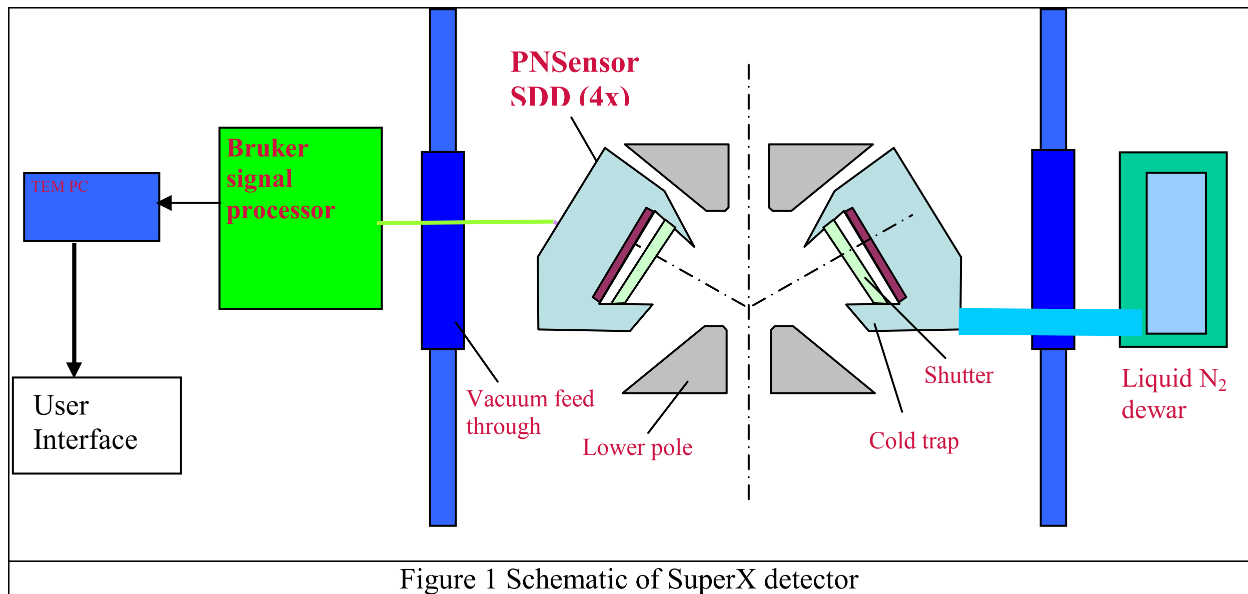

Figure 1 Schematic of SuperX detector

The detectors are integrated into the cold-trap for best performance. The windowless design allows for a better sensitivity for light-element detection than conventional thin-window detectors. Except during analysis, shutters are closed in front of the detectors to prevent contamination. The signals from the SDDs are processed by an ultra fast four-channel pulse processor produced by Bruker AXS, with pixel dwell times down to 10 µs and output count rates up to 60 kcps per channel.

3. Performance

The detector has been characterised using a nickel oxide standard sample of 50 nm thickness on a Mo grid.

Figure 2 shows a spectrum of the NiOx sample at 200kV. The spectrum shows a high peak-to-background ratio, exceeding a Fiori number of 5000, and the absence of spurious peaks. The Ni-L and O-K peaks are 1.3 and 2.5 times higher, respectively, than for a Si (Li) thin window detector.

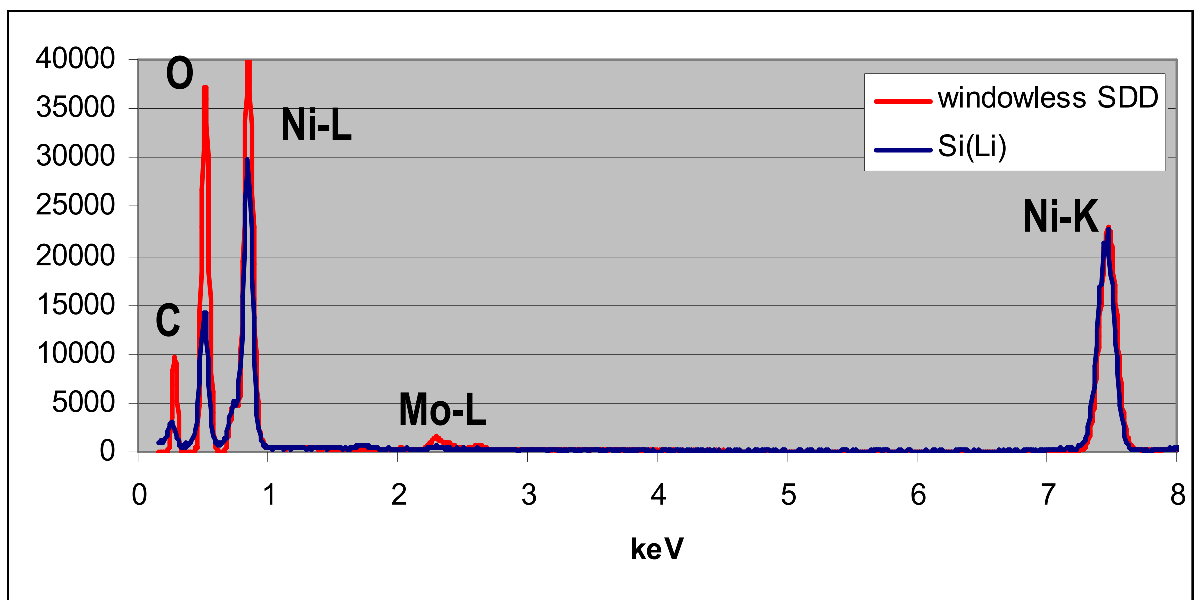

Figure 2 SuperX and Si(Li) detector spectra of NiOx specimen on carbon film and Mo grid at 200kV under identical conditions; spectra normalised to Ni-Kα peak

The input and output count rates were measured for the detectors. Fig.3 shows output count rates up to 350 kcps. The dead time is below 60% up to 240 kcps output count rate. The energy resolution is below 135 eV up to 240 kcps output count rate.

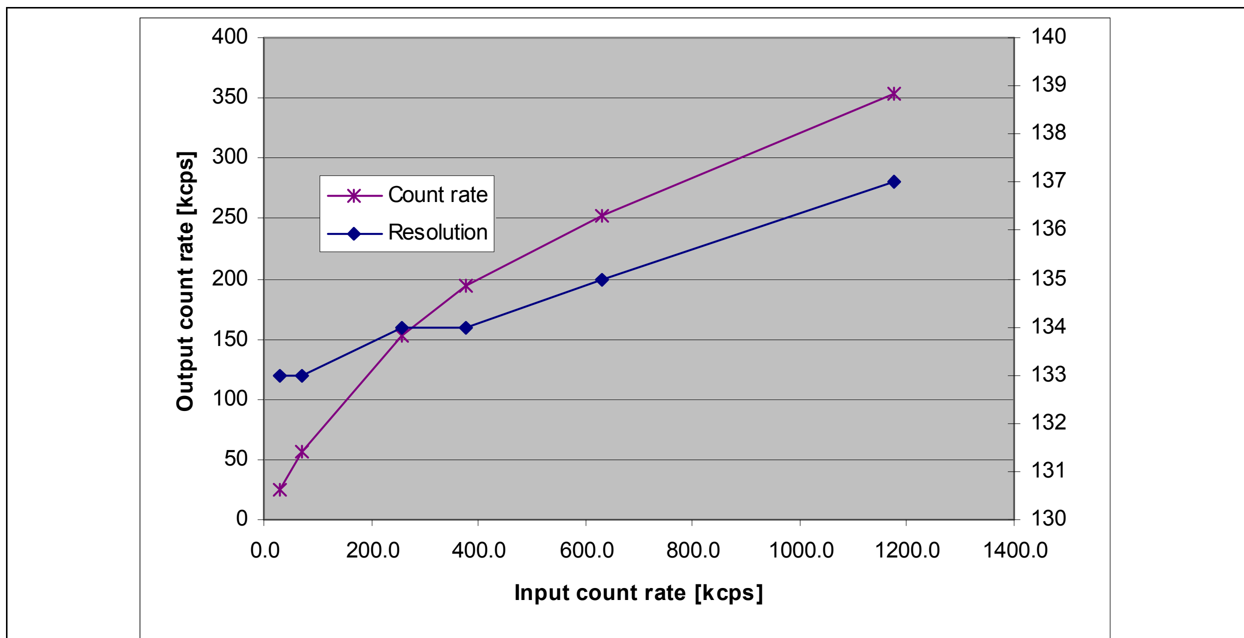

Figure 3 Input-Output count rate and energy resolution (RH scale) vs. input count rate

4. Fast mapping

The mapping speed is illustrated in figure 4. A 300 x 50 pixel map was acquired on a Bi2212 superconductor specimen at 200kV. Using a pixel dwell time of 50µs per pixel, the map was acquired in 1.5 minutes and shows clearly the Bi, Sr and Ca lattice planes. This acquisition time is estimated to be 30 times faster than with conventional EDX detectors. Note the missing planes of Ca atoms indicated by arrows in Fig.4. At these defects the BiO spacing is reduced (see model of unit cell in Fig.4), but the spacing of SrO planes is unchanged at about 0.8 nm.

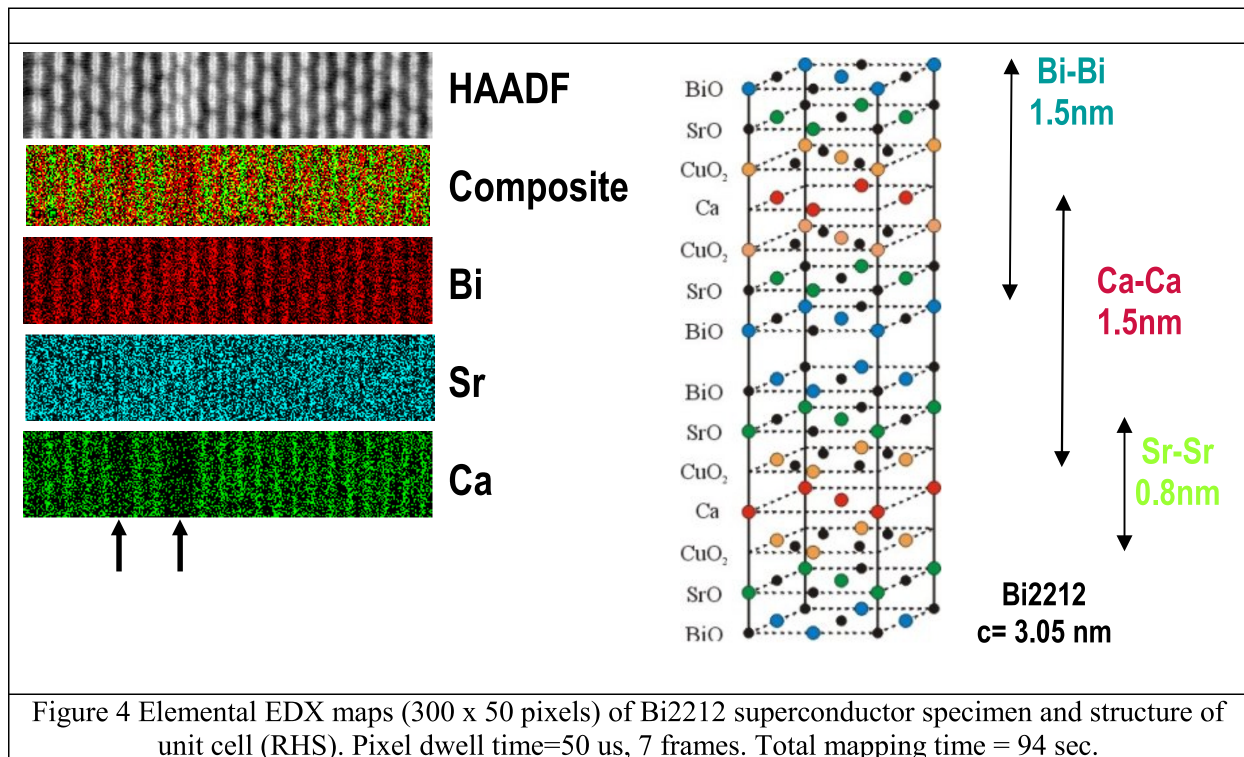

Figure 4 Elemental EDX maps (300 x 50 pixels) of Bi2212 superconductor specimen and structure of unit cell (RHS). Pixel dwell time=50 us, 7 frames. Total mapping time = 94 sec.

5. Conclusions

The SuperX detector design results in a massive increase in solid angle of collection to 0.9 sr compared to 0.1 – 0.3 sr of previous designs in S/TEMs, and hence, a huge increase in count rate for a given beam current. When combined with a high-brightness electron beam a large reduction in the time for EDX mapping from hours to minutes is achieved. Sub-nanometer resolution is achieved in less than 2 minutes' acquisition of X-ray spectrum images.

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