

Performance Evaluation of Dual Bruker XFlash6 | 100 EDS Detector Integrated in FEI Themis With Analytical Objective Pole Piece

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Advances in high brightness electron sources and aberration correction of electron probe result very flexible probe sizes and currents. Introduction of multiple windowless SDD detectors [1] increased the collection efficiency greatly (from about 0.15 to 0.7 sr) and light element sensitivity. Furthermore using different combination of probe sizes and beam currents, analytical electron microscopy using EDX spectrometry (EDXS) can be realized from nm scale to Ångstrom scale [1]. It is therefore obvious that EDXS analysis of various materials will definitely benefit from increase in count rate performance and collection efficiency resulting shorter experiment times, decreasing the total dose used and therefore limiting the extent of the sample damage.

In this report, we demonstrate the performance of Dual Bruker XFlash6 | 100 SDD detectors mounted on FEI Themis TEM column. These SDDs have active area 100mm² and no window is present in the detector assembly. Windowless design ensures light element sensitivity and careful positioning of the detector increases the collection efficiency. In order to accommodate the detectors the objective pole piece of the Themis was modified to special geometry (herein after called as X-Twin). Dual detectors are symmetrically positioned with respect to Alpha tilt axis therefore ideal for EDX tomography data acquisition. For the characterization of the different EDX systems with respect to energy resolution, signal-to-noise-ratio, stray radiation, collection angle and relative detector efficiency a NiO_x specimen was analyzed as described in Ref [2].

In Fig. 1(a) EDX spectrum of a NiO_x specimen are shown with the Dual-X detector recorded at 200 kV. A beam current of 1 nA was used, a specimen area of 0.4 μm² was illuminated and an acquisition time of 100s was chosen. It is observed on Si₃N₄ sample that the Dual-X detector with its bigger collection angle has higher collection efficiency and acquires 50000 X-ray counts per second per nA beam current (Fig 1(b)). Tilt dependent peak to background ratio of the EDX system was measured for different tilt angles on the same spot for same beam current and hence. We ensure similar X-ray collection performance for each tilt angle. As tilt angle increases, absorption in the specimen and shading from the holder gradually reduces and peak to background ration increases as it can be seen in the Fig. 1.(c).

HRSTEM image of <110> oriented Silicon sample obtained on X-Twin lens equipped with Probe Cs-corrector at 200kV can be seen in Fig.2 (a). Spots corresponding to 78 pm inter-planar distance is visible in the FFT of the image, in Fig. 2(a)

FIN Capacitor sample doped with Arsenic was used to test the performance of the Dual-X detector. The multiple frame EDX map acquired with 700 pA beam current for 220 seconds clearly shows the Arsenic doping on Si. Furthermore, Arsenic diffusion on to silicon oxide layer was also found as can be seen in Fig .2 (b). It is clear from the short acquisition time that this X-ray detecting system is a very efficient and therefore EDX maps with minimal sample damage can be acquired. [3]

References:

- [1] P Schlossmacher et al, *Microscopy Today* **18** (2010), p.14
- [2] RF Egerton and SC Cheng, *Ultramicroscopy* **55** (1994), p. 43
- [3] The authors acknowledge the support from Bruker Nano GmbH

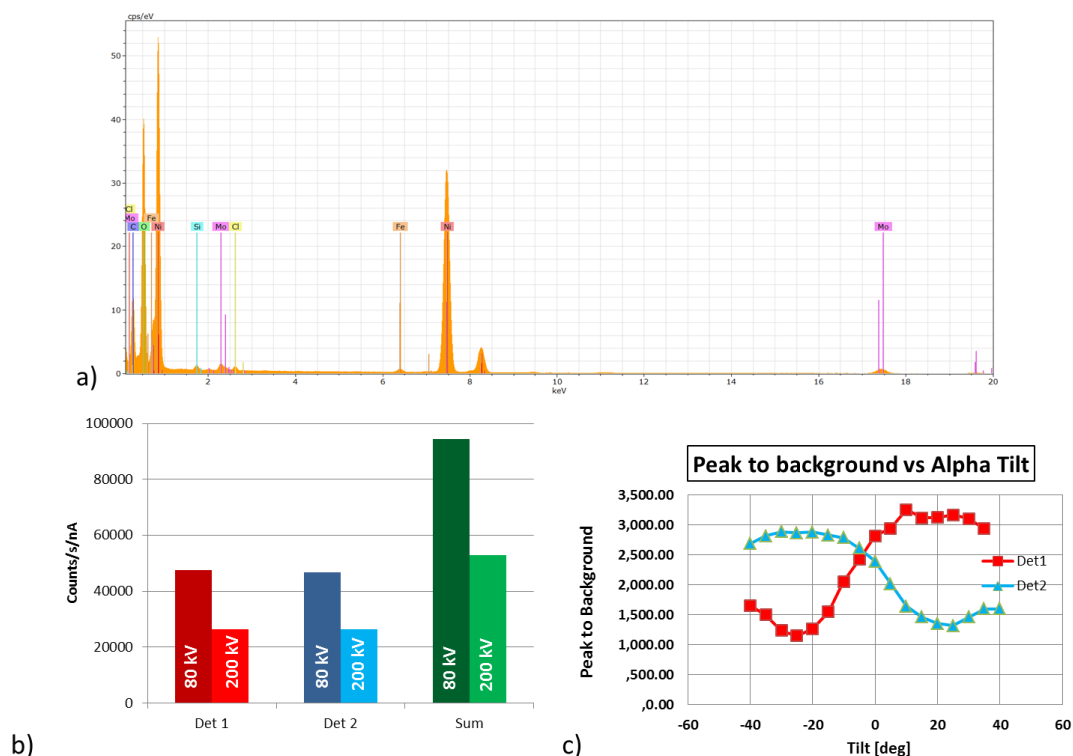


Figure 1. a) Typical EDX spectrum obtained on NiO_x. in addition Ni and O peaks, Cl is the contaminant on NiO_x, Mo is from holder clip, Fe is from the pole piece and C is due to slight contamination b) Total counts obtained on Si₃N₄ sample per nA per seconds c) Plot of peak to background ratio for symmetrical Alpha tilt.

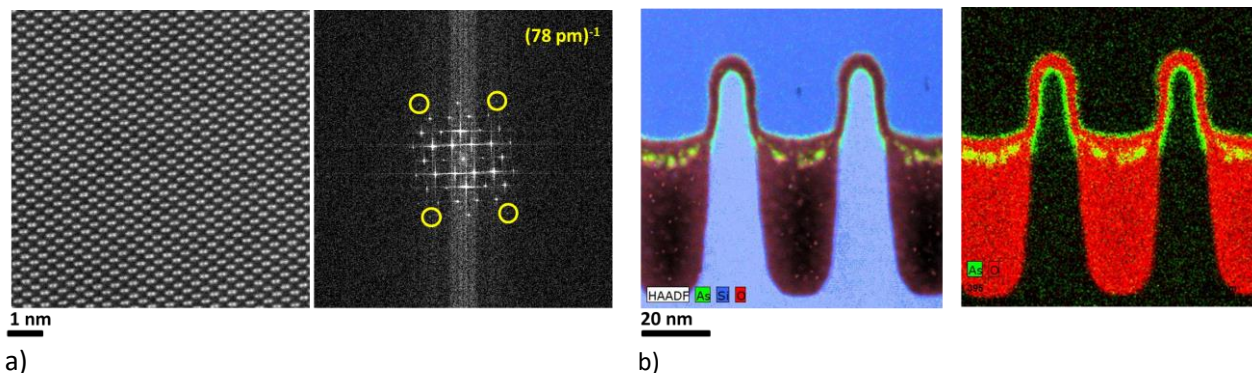


Figure 2. a) Probe Cs-corrected HAADF-STEM image of Si[110] and its FFT taken at 200 kV b) 512 x 512 pixels EDXS elemental maps acquisition of Fin Capacitor sample taken at 200kV with 700 pA beam current. Total acquisition time was 220 seconds.