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Design of an Analytical TEM/STEM with 0.3 srad EDX detection

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Although EDX has been a standard technique in AEM for a long time, there is still room for performance enhancement. The reason for this is to some extent due to the opposing requirements for analytical and imaging performance: the first requiring space for detectors close to the specimen, the second requiring the minimum gap between the objective pole pieces, unless aberration correctors are used.

The motivation for this work was to enhance the EDX performance – both in quality and in count rate - of the FEI Tecnai TEM/STEM further without degrading imaging resolution. Previous work [1] had established that improvements in the spectrum quality, i.e. high peak-to-background (P/B) ratio and hole count, as well as low spurious peak levels, could be achieved by raising the elevation angle of the EDX detector. To improve the count rate (at a given beam current) in EDX spectra, that is, to increase the collection solid angle of the detector, the pole pieces or the detector have to be modified. We made the choice of redesigning the pole pieces to fit slightly modified Si(Li) detectors, with a target of 3 times higher count rate (~0.3 srad collection angle) and 1 mm spherical aberration coefficient of the objective lens.

The resulting instrument (Tecnai X-Twin) has the detector geometry shown schematically in Fig. 1. The Si (Li) detector, designed by Edax Inc., has a 30mm^2 crystal at less than 10 mm from the specimen, resulting in a collection angle of 0.3 srad and an elevation angle of 25 degrees. The lower pole piece is covered by a beryllium shield to reduce the back-scattered electrons and spurious X-rays reaching the detector. The gap and bore dimensions have been adjusted to give the required 1 mm spherical aberration coefficient. The electron optical parameters are listed in Table 1. The design allows for tilt angles up to 30° with a low-background, double-tilt holder, and up to 70° with a tomography holder.

The instrument has been tested at 200kV, and the results show the TEM information limit to be below 0.13 nm and HR STEM imaging resolution at about 0.16 nm, corresponding to theoretical prediction. The EDX performance is compared in Fig. 2 with that of a Tecnai S-Twin instrument (0.13 srad detector, 20 deg. elevation angle) for the same specimen: the count rate per nA of probe current is increased by 3 – 4 times. This increase is due to the factor of 2.5 larger solid angle and the higher elevation angle of the detector. The quality of the spectrum in terms of P/B ratio [2] is high (>4000 for Ni-K), the hole count ratio is low (<0.2%) and the spurious peak level is below 0.5%, as illustrated in Fig. 3 for a standard NiOx specimen on Mo grid.

References

[1] H.S. von Harrach et al., *Micron* **34** (2003) 185.
[2] C. Fiori et al. *Microbeam Analysis-1982* (1982) 55.

TABLE 1 Optical parameters of X- Twin lens and guaranteed resolution

X-TWIN parameters	
STEM Cs	1.0 mm
TEM Cs	1.3 mm
TEM Cc	1.3 mm
STEM resolution	0.18 nm
TEM point resolution	0.25 nm
TEM information limit	0.14 nm

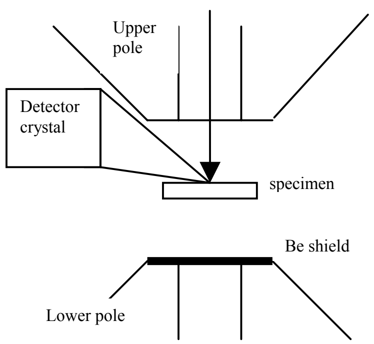

Fig. 1 Schematic diagram of X-Twin geometry: detector collection angle = 0.3 srad, elevation angle= 25 deg.

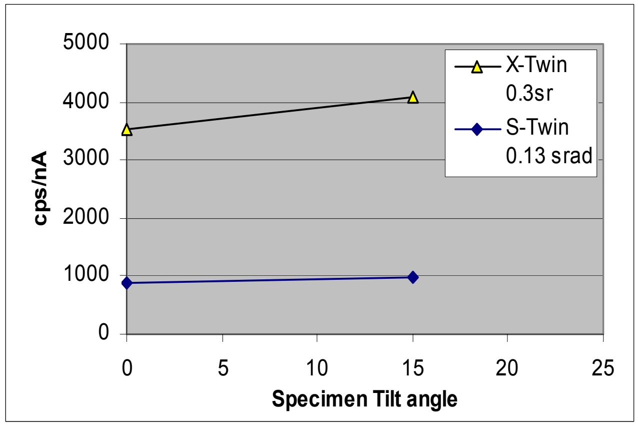

Fig. 2 Count rate comparison between Tecnai S-Twin (0.13 srad) and X-Twin (0.3 srad); LBDT holder.

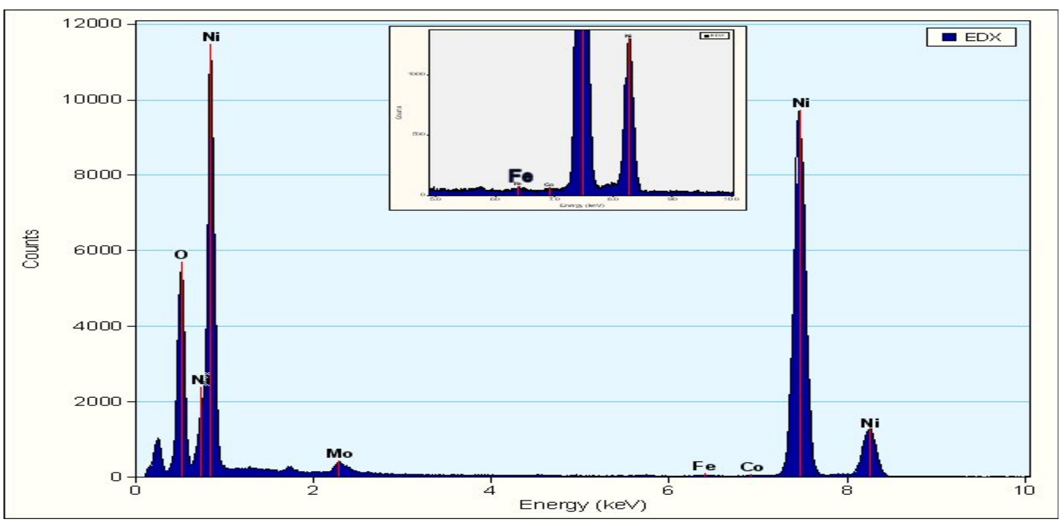

Fig. 3 NiOx spectrum (200kV X-Twin, 0 deg. specimen tilt). Insert: enlarged region at 5-10keV, Fe-K peak < 0.3% of Ni-Kα peak

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