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IT-5-P-5868 Trends in X-ray Nano-Analysis by TEM/STEM

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The improvements of X-ray detectors gained momentum in recent years with the introduction of silicon drift detectors (SDD), and the increase in collection solid angle (SA) and speed of acquisition. The ability to produce larger detectors up to 100 mm2 without impairing the energy resolution means that detectors with 1 srad collection angle are now available (1). Since the SDDs are capable of higher throughput they have also improved the minimum detection limit of an element in a given analysis time.
Improvements in TEM/STEM resolution were brought about in recent years by the correction of spherical aberration (Cs) and the introduction of higher brightness sources, both Schottky and cold field-emission sources. This has resulted in sub-Angstrom image resolution for thin specimens, but only sub-nanometer probe sizes at the currents (~1nA) required for acquiring X-ray data with Si(Li) detectors at reasonable signal-to-noise ratios (S/N).
The combination of these developments has resulted in the ability to use lower probe currents to produce good quality analytical data at the atomic level within a few minutes (2). This is crucial for many materials that are damaged by exposure to high-energy electrons at high intensity.
So how should the technique be improved further in the future? On the TEM side, the need to operate at lower kV to minimise specimen damage calls for the use of higher brightness sources with lower energy spread (dE) or for chromatic aberration correction. Cold field-emitters are the brightest sources currently available (reduced brightness Br~1e8 A/m2/sr/V) and, either in combination with a monochromator or Cc corrector, could result in sub-Angstrom resolution at 40 -80kV (see Fig.1). For monochromated (dE=0.1eV) and Cs/Cc corrected instruments with cold field-emitters the probe size (d50) at 10pA is below 0.1nm at 40-50 kV and above.
On the detector side, there is room for improvement of collection angles approaching the theoretical limit of 4π steradians. This would reduce the dose required for good quality nano-analysis (S/N>5) by a factor of 10 or more. Fig.2 shows the relative dose at constant X-ray counts detected as a function of acceleration voltage (kV). As the kV is reduced the X-ray yield increases roughly as inverse square root of kV (3). Consequently, with a 10 sr detector the electron dose could be reduced by a factor of almost 20 by operating at 60 kV and sub-Angstrom probe size, compared to the current best instruments at 200kV with 1 sr detectors.

References

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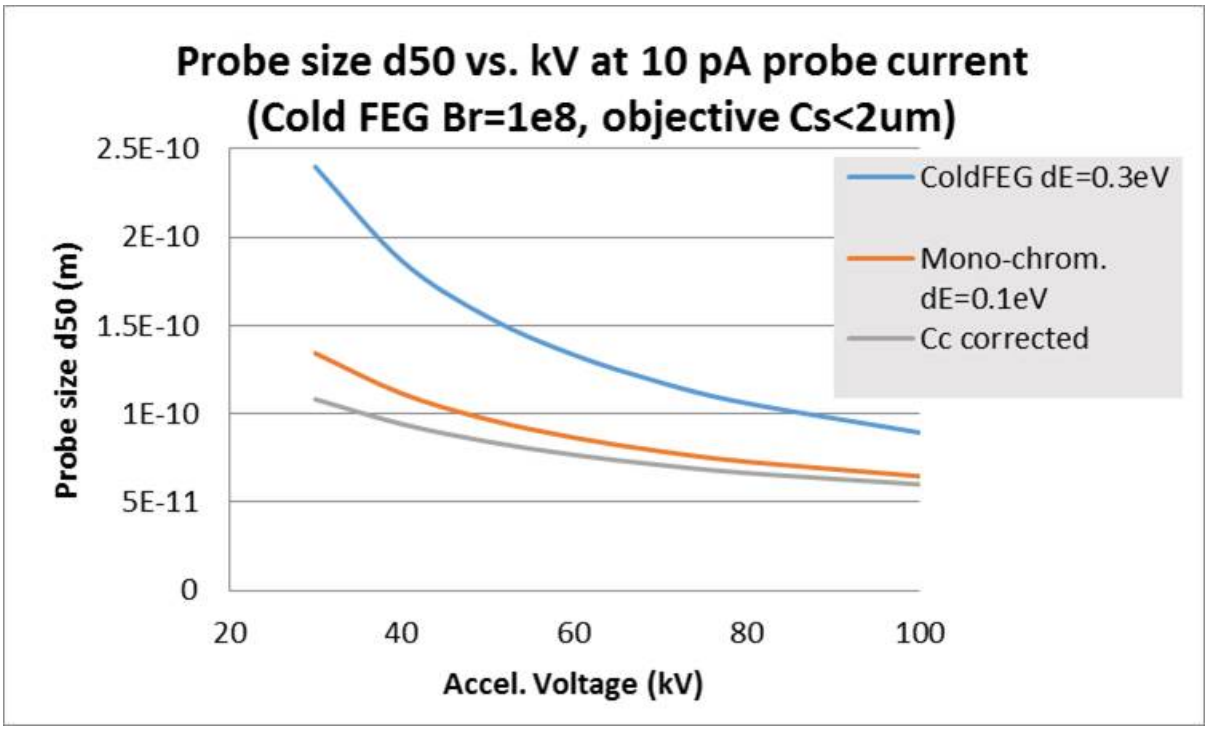

Fig. 1: Fig.1 Probe size (d50) at 50% of constant 10pA probe current vs. acceleration voltage for cold FEG source (reduced brightness Br=1e8 A/m2/sr/V) with Cs corrector (Cs<2um), mono-chromated CFEG with dE=0.1eV, Br=3.3e7 and Cc corrected CFEG system with Br=1e8, Cc<0.1mm. [ref. P.Kruit et al. 2006 J. Appl. Phys. 99, 024315]

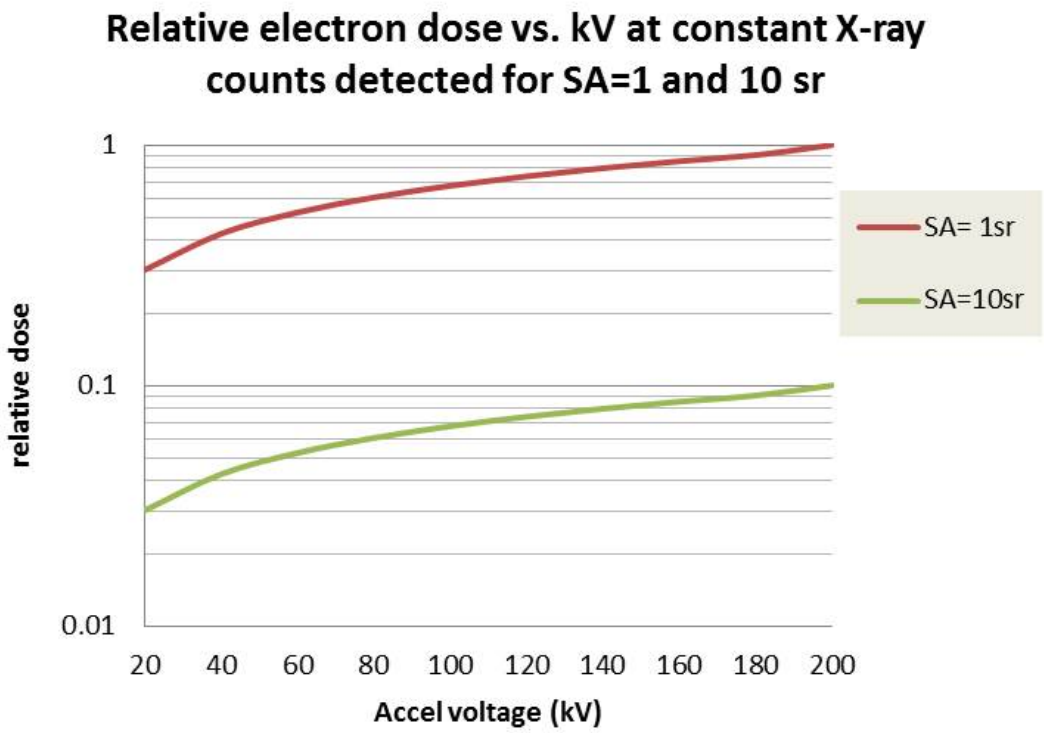

Fig. 2: Fig.2 Relative electron dose vs acceleration voltage at constant number of X-rays detected for X-ray detectors of collection solid angles SA = 1 and 10 sr.