doi:10.1017/S1431927614004541
Microsc. Microanal. 20 (Suppl 3), 2014
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XEDS in the AEM: Has Everything Thing That Can be Invented, Been Invented?

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There is a fairly old apocryphal quotation stating : "*Everything that can be invented has been invented.*" [1] attributed at one time to Charles H. Duell of the US Patent office in 1899. We all realize this is an absurd statement, and it is well documented that Duell actually said something quite different and urged the US Congress to encourage the advancement of science as well as to improve the patent system [2,3]. This does however bring to focus a salient point concerning x-ray spectroscopy, as it relates to analytical electron microscopy. For many years instrument developers have concentrated upon advances in electron-optics, with justifiable and demonstrated improvements in imaging and probe forming systems. Aberration correctors, high sensitivity imaging detectors and electron spectrometry systems abound [4] , however, there are only a few seminal events which mark evolutionary advances in x-ray spectrometry. Arguably we can list the foremost of these as: the invention of electron microprobe analysis (EMPA) by Castaing [5], the invention of the Lithium Drifted Silicon (Si(Li) solid state detector [6], the development of the first AEM (EMMA-4) [7] and the development of the Silicon Drift Detector (SDD)[8]. While these are all key events which mark the onset of major advances in our ability to perform microanalysis, by comparison they represent only a fraction of the investment in resources compared to the electron optical developments. A very large fraction of the instrumentation found in characterization facilities worldwide has ancilliary x-ray analysis subsystems. The majority of these ancilliary x-ray spectrometry systems have been interfaced to an electron optical column in the simplest possible configuration and although they are at times considered routine, this does not mean that we have achieved all that these resources can provide, nor does it mean that we have addressed fully all their methodological intricacies and/or potentials.

If one were to compile a list of areas which are obvious candidate areas deserving attention at a minimum it would include the following:

- geometrical collection efficiency, optimized use of detector arrays [9-11] (fig. 1),
- high-energy efficiency, using thick, multi-layer detectors and/or alternative materials (fig. 2),
- spectral artifacts and energy resolution under conditions of high count rate,
- radiation resistance of detectors to damage by energetic electrons,
- sensitivity (peak/background) variation created by new geometries (fig. 3) [11-13],
- new modalities for channeling dominated, time resolved and position tagged spectroscopy [14],
- quantification / absorption issues created by new geometries (fig. 4).

Addressing these will improve the quality, speed, accuracy and ultimate sensitivity of XEDS in the AEM. While for thin specimens ($t/\lambda < 0.4$) electron energy loss spectroscopy is in many situations a more signal rich spectroscopy, for thicker specimens ($t/\lambda > 1$) the microanalytical advantages will in many cases shift toward x-ray spectroscopy.

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[15] This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357 at the Electron Microscopy Center of Argonne National Laboratory.

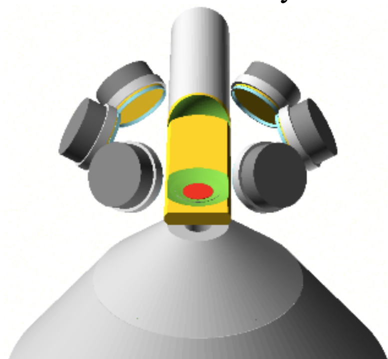

Figure 1.) Improving geometrical collection efficiency (Ω) using an array of detectors surrounding the specimen. $\Omega_{total} = \sum \Omega_i$

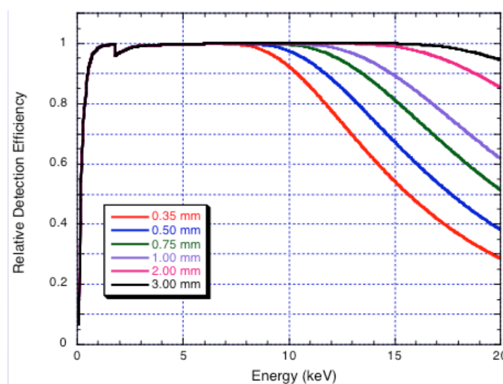

Figure 2.) Comparison of the detection efficiency as a function of x-ray energy and detector thickness for the new generation of SDDs note reduced high energy detection efficiency above ~ 8 keV for thinner detectors which are now common (~ 0.45 mm).

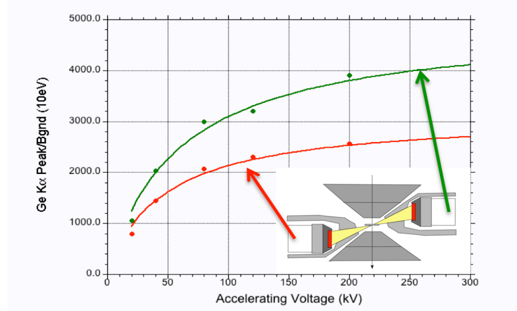

Figure 3) Variation in the experimental P/B ratio of a a thin Ge film as a function of accelerating voltage in an FEI CM200 AEM for a detector observing the electron entrance and exit surface of a specimen.

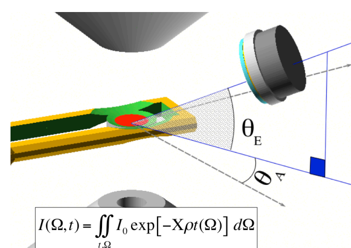

Figure 4). New large solid angle detectors require reformulation of the absorption correction to account for variable take-off angles and multiple detector geometries which will have large values of $d\Theta_E$ and $d\Theta_A$.