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# Everything You Always Wanted to Know about XES …..

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Next to secondary and back-scattered electron imaging, X-ray Emission Spectroscopy (XES) in electron optical instruments is one of the most widely used characterization methodologies in both the materials and life science community. The most common implementation of XES in the electron microscope involves the interfacing of a solid state x-ray energy dispersive spectrometer (XEDS) to an instrument and subsequent detection and analysis of both characteristic and continuum x-ray radiation generated by the electron-solid interaction. It is rare that a general purpose electron microscope (either SEM or TEM) is procured with this versatile microanalytical accessory particularly in a User Facility. Over the last 30+ years the performance of these solid state detectors has gradually improved, and their application has abounded in all areas of research. Although XEDS usage is considered turn key operation in many situations, its application to characterization in the SEM/TEM will be limited by three factors which, in demanding applications, dictates the utility of XEDS for practical problem solving. These factors are: spectral resolution, high count rate performance, and analytical sensitivity. In the case of the latter, the development of the ultra high energy resolution micro-calorimeter detectors and/or CCD based wavelength dispersive spectrometers provides the potential of high energy resolution spectroscopy with a demonstrated 20 fold improvement in energy resolution. However, this is achieved at the expense of count-rate ($R$~ 500 c/s) and solid angle ($\Omega$ < .01 sr) performance. Under conditions where either count rate or solid angle are the more important criteria, which is the more common situation, then the Si(Li) (lithium drifted silicon) or the SDD (silicon drift detector) ($R$ > 10 kcps, $\Omega$ >0.7 sr) become more suitable.

Understanding how these detectors operate in detail is not essential to their usage, however, a basic knowledge on their construction and response characteristics goes a very long way in aiding in the optimization of an experiment. For example, in figure 1, we plot the efficiency of typical Si(Li) or SDD systems as a function of x-ray energy for various detector parameters. At the low energy end (0-2 keV) these detectors are limited by the choice of environmental protective and/or entrance windows, while at the high energy regime they are limited by the type and thickness of the detector [1]. Efficiency of detection although important is not the only consideration when configuring an experiment. This is illustrated in figure 2, where partial spectra is shown of the Ni Kα emission obtained from the same area of the same NiO specimen taken from 4 different instruments under identical conditions [2]. The variation in detected intensity varies nearly 10 fold and is related to the geometrical collection solid angle of the detector. When neither time or beam current is important then any one of these instruments would be suitable to analyze the specimen. However, when an analysis is data limited, then choosing the optimal position of the detector and/or geometry, should it be adjustable, is paramount. An example from hyperspectral imaging, which employs optimized detectors is shown in Figure 3, here the Ga, N, and In elemental distributions are mapped at high spatial resolution [3]. The collection time for this type of data can be as long as 8-24 hours using a poor collection efficiency detector, with a high collection efficiency detector this can be reduced to 30 minutes. Given the complexity introduced to an experiment by drift, contamination and stability clearly optimized collection is essential for hyperspectral data sets.

Once we know the limitations of the data acquisition system and how to identify various artifacts due to

the instrument, its surroundings, as well as those created by the incident electron beam, the next step is quantification. A brief discussion of these topics is beyond inclusion in the body of this short paper, but will be included in the platform presentation [4].

*References:*

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[3] I.H. Wildeson etal. *Journal of Applied Physics* **108**, 044303 (2010)
[4] Research 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.

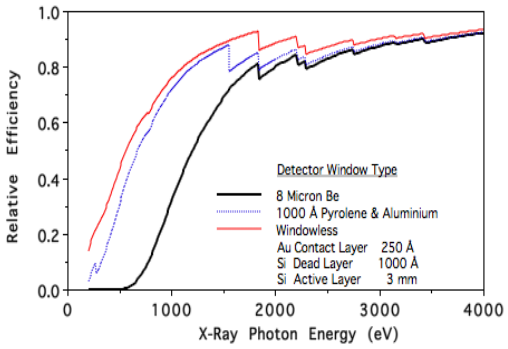


Figure 1a.) Detection efficiency as a function of incident x-ray energy for typical Si(Li) and SDD systems for low energy x-rays (0-4 keV). Note the dependence on the detector window.

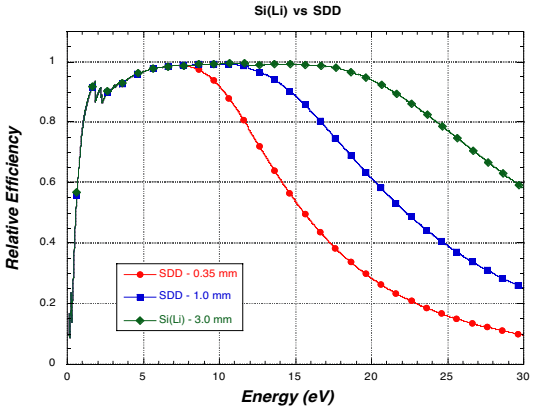


Figure 1b. ) X-ray detection efficiency as a function of incident x-ray energy for typical Si(Li) and SDD systems for higher energy x-rays. Note dependence of the detector thickness

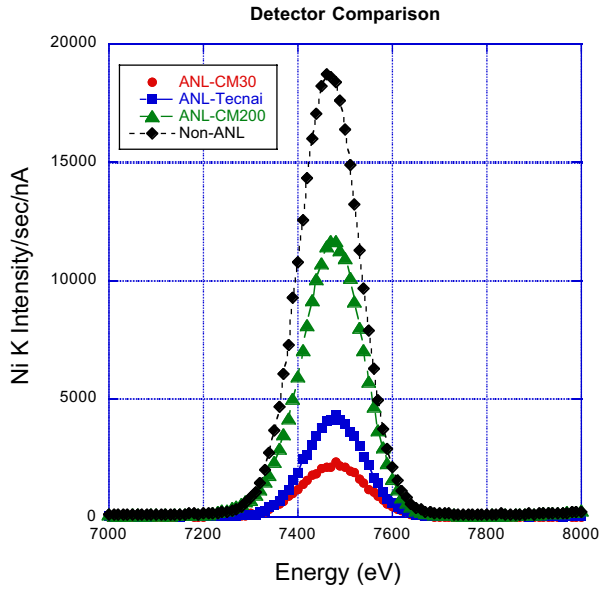


Figure 2. Comparison of the Ni Kα emission for 4 different instruments all under identical conditions. The variation is nearly 10 fold and depends upon the collection solid angle.

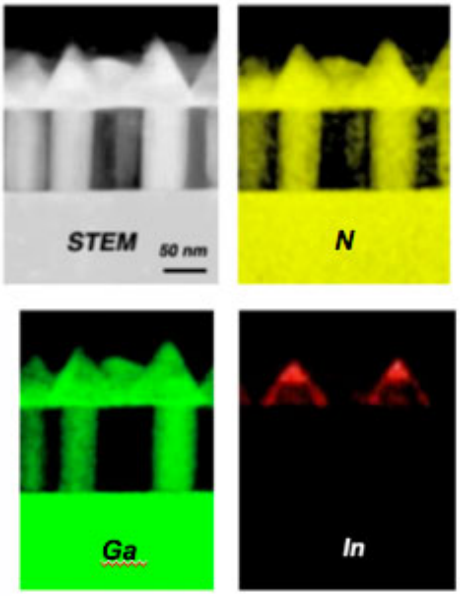


Figure 3. Portions of a Hyperspectral Image showing the elemental distribution of Ga, N, and In, in engineered LED's.

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