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Direct Comparison of X-ray Detector Solid Angles in Analytical Electron Microscopes

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One of the most often misquoted parameters of a solid state x-ray detector interfaced to the Analytical Electron Microscope is its collection solid angle. Due to the advances in the design and construction of modern Silicon Drift Detectors (SDD) the collection solid angle which in the past has hovered about 0.1-0.15 sR, is now routinely quoted in the 0.2-0.8 sR range, with the highest reported being just over 3 sR[1-4]. While the definition of solid angle is well established (Figure 1) it is frequently loosely specified by a given manufacturer and is often calculated using idealistic rather than real parameters[5]. This work reports on systematic measurements of the relative solid angle of 10 different detectors interfaced to four different instruments to establish a methodology for absolute comparisons.

The experimental results reported herein were conducted on a series of FEI analytical electron microscopes, operated from 20 to 200 kV. Essential to the measurements was the absolute calibration of the incident beam currents. This was accomplished by building a custom Faraday Cup (FC) measurement system and installing it within the projection chamber one of the instruments (FEI CM200F) which serves as the reference instrument. Installing a similar FC in other instruments evaluated was not practical. Thus in order to normalize beam current measurements from all instruments a specimen stage based Faraday Cup (sFC), was calibrated relative to the more accurate permanent FC so that absolute comparisons between instruments could be accomplished.

Two uniformly thick reference specimens, one each of amorphous Germanium and nanocrystalline Nickel Oxide were used for all measurements. In order to validate direct comparisons, the same area of each specimen was analyzed in all instruments. Prior to any microanalytical measurements, both specimens were plasma cleaned to mitigate any hydrocarbon contamination effects. The NiO specimen was measured while mounted in a double tilt Beryllium gimbal stage, while the amorphous Germanium specimen was measured in a custom built single tilt stage having a 100 µm diameter polymer microloop support ring, extending from a polymer support.

Figure 2 plots the normalize Intensity/sec/nA measured for the Ni K and Ge K line emissions, normalized at 200 kV. Absolute measurement of the specimen thickness (in progress) will allow these data to be eventually converted into absolute cross-section and solid angle measurements.

Figure 3 presents the variation of the Ni K Intensity/sec/nA measured from 4 of the 10 different detector configurations. All spectral profiles shown in this figure were measured from the identical area of the same NiO specimen. This succinctly illustrates the rather large variation in collection solid angle presently available in modern instruments. In this example two of the detectors are Si(Li) systems having ultrathin windows (Moxtek Windows) while the two others are windowless SDD detectors. All of these detectors were specified as having an active area of 30 mm^2 while one was a dual detector configuration which effectively doubles the collection area.

Figure 4, present a compendium of experimental data which compares the relative collection efficiency of the 10 different detector configurations tested to date. All data are normalized to the performance of

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the ANL reference instrument. In excess of a tenfold variation has been measured in the relative solid angles. Additional work is in progress to determine the absolute value of the collection angle of the reference instrument and thus calibrate the solid angle of all system to which it is compared.[6]

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$$\Omega = \frac{S}{R^2} = \frac{2\pi\left(r_a^{\,2} + d^2 - d\sqrt{r_a^{\,2} + d^2}\right)}{r_a^{\,2} + d^2}$$

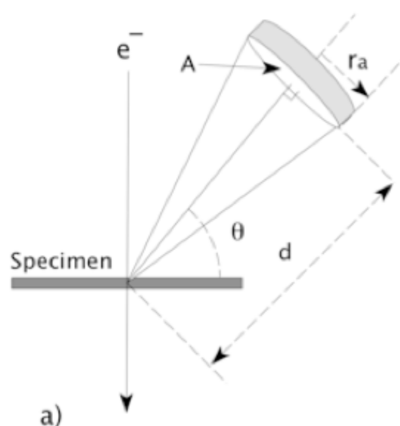

Figure 1.) Collection Solid Angle (Ω) defined in terms of the detector area (A) and radius (r), and its normal distance (d) from the region of interest.

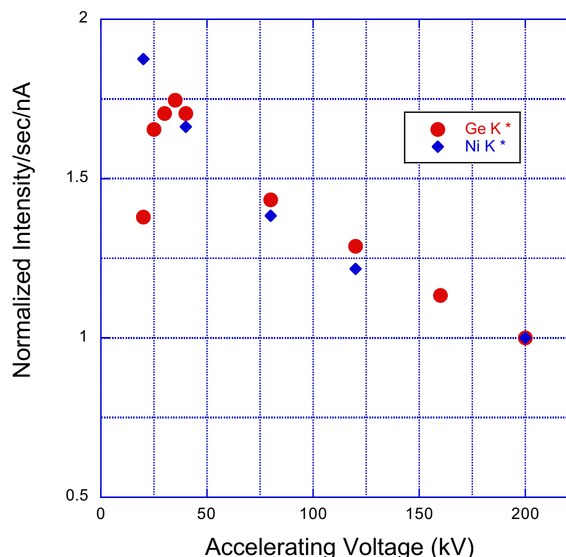

Figure 2.) Experimental variation in Intensity/sec/nA with Accelerating Voltage for the NiK and GeK lines.

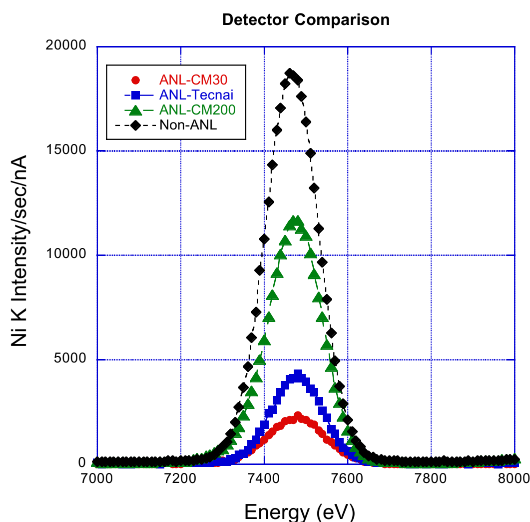

Figure 3. Comparison of the Ni K emission for 4 different 30 mm^2 detectors.

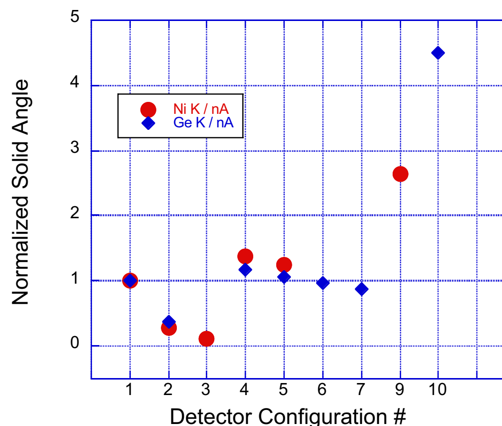

Figure 4. Normalized solid angle for the 10 different detector configurations studied.

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