A Standard Protocol and Specimen for Measuring the Effective Solid Angle of X-ray Detectors in the Analytical Electron Microscope

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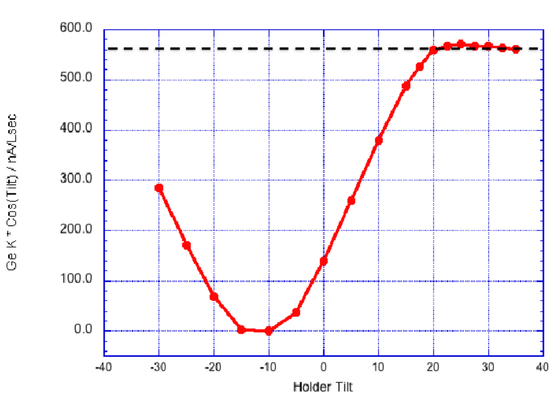
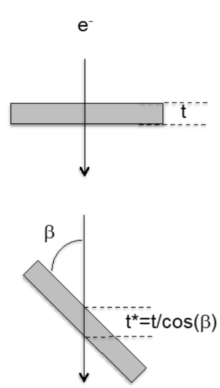
With the rapid evolution of the current generation of solid state x-ray detectors for the analytical electron microscope it is essential that we have a corresponding methodology to test and measure the functionality of these new systems. A simple protocol which employs a uniform thickness TEM film of non-crystalline Ge (20 nm) on SiNx (20nm) has been tested on several dozen instrument and has proven to be a reproducible method to assess: the effective solid angle of the detector system, system peaks/artifacts, as well as the penumbra of the analytical holder. Evaluating all of these in an unbiased and systematic method is particularly important when addressing issues such as comparing performance on different instruments, absolute sensitivity, quantification and absorption corrections for compositional measurements as well as emerging applications, such as 3D elemental tomography where geometrical effects will affect data interpretation.

A uniformly thick amorphous film is the best choice for this measurement as it avoids problems with channeling in crystalline materials which affect the absolute x-ray emission. Ge is an ideal candidate as it can be deposited in an non-crystalline form, is not found in most instruments, and can also be used as an energy calibration specimen. Suitable films can now be procured commercially from EM suppliers.

The protocol for measurement is simple: the Ge test specimen is mounted in the instrument holder and a series of spectra at constant live time, and beam current are recorded as a function of holder tilt. The full peak integrals of the Ge K line are measured and plotted as a function of holder tilt (Figure 1). Since the film is of uniform thickness, the intensity is multiplied by the cosine of the tilt angle which compensates for the change in projected thickness with tilt. A perfect detector/specimen geometry will yield a simple horizontal line as a function of tilt, practically this is never the case. Instead there is a pronounced variation with tilt as shown in Figure 1 as it is inevitable that the holder at some point limits the field of view of the detector. The region of the experimental profile where the intensity variation is constant represents the non-penumbra limited working zone of the instrument. In the example of Figure 1, this occurs only for holder tilt > 20°. All other regions represent penumbra limited configurations of the detector/specimen/microscope geometry and thus have reduced performance. This functional dependence with tilt can be simply understood by reference to Figure 2, which is the calculated performance of an ideal detector whose collection solid angle is limited by the holder penumbra. Once the region of non-holder limited detector performance is identified, the effective solid angle can be calculated knowing the cross-section, specimen thickness, and beam current.





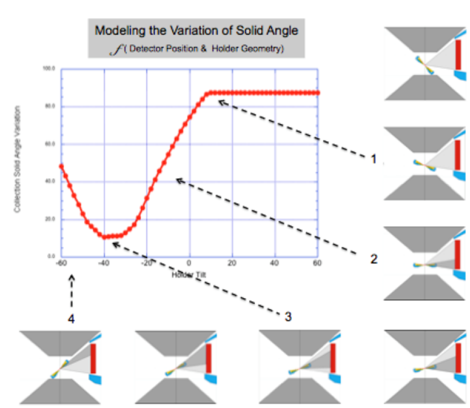


Figure 1. Experimental variation of the GeK with holder tilt. Vertical axis = Ge K integral * Cos (tilt) /nA/LiveSec.

An ideal system has a functional dependence illustrated by the dashed line.

Figure 2. Model calculation showing the penumbra limited variation of effective solid angle

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