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Quantitative Measurements of the Penumbra of XEDS Systems in an AEM

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One of the most common limiting factors in x-ray energy dispersive spectroscopy (XEDS) is the penumbra of specimen holders which are used in the typical analytical electron microscope (AEM) today. Due to the large number of designs of holders available as well as the plethora geometries which can be realized using multiple x-ray detectors in various configurations of an AEM it is essential to have a quantitative measure/knowledge of the shadowing effect of the holder on the performance of the x-ray detector system. While a penumbra can be calculated [1] based upon system specifications (figure 1), experimental measurement is the more accurate solution. This procedure outlined herein allows one to accurately assess when the maximum collection solid angle of the XEDS detector is realized for a given analytical configuration. and further allows calculation of correction factors for quantitative x-ray analysis.

A uniformly thin, flat amorphous film without obstructions from grid bars or supports along the line of sight path toward the detector, having addition both high (>7 keV) and low (< 1.5 keV) energy lines which can be identified as independent of any instrument system peaks is an ideal performance test specimen [2]. In this protocol an amorphous Ge film was chozen as it does not suffer from the anamolous effects of electron channeling and its simple well resolved lines are not present in any construction materials of either the microscope or the detector. The measurement procedure consists mounting and centering the uniform film in the holder, locating the specimen at the eucentric position of the TEM, and then recording a simple spectral sequence of the emitted intensity as a function of holder tilt at constant beam current and probe size. Figure 2a, plots an example of the integrated intensity of the Ge Kα peak variation versus holder tilt. The plot reveals succinctly where the penumbra of the holder no longer limits the solid angle of the detector and is demarked by a distinct reproducible transition/break point in the signal variation with tilt. In this example, the transition occurred at a holder tilt of +5^o (a positive tilt is toward the detector). Because the detector in this AEM is elevated ($\theta_E \sim 10^o$), signal can be measured even when this customized holder is tilted away (negatively) from the detector normal. Interrogating the data of figure 2 closely we see that the intensity varies nearly linearly with tilt as the penumbra/shadow of the holder transverses the detector, until it reaches a critical angle. At this transition point the holder penumbra no longer limits the detector solid angle, and the intensity varies with tilt with an inverse-cosine response. This inverse cosine is expected as the intensity for a *thin amorphous* film will vary as the projected thickness along the beam direction, which can be appreciated by reference to figures 1b and c. The unobstructed angular range can be made even more obvious by plotting the product of the raw intensity multiplied by the cosine(β). The regime where this intensity product is flat, represents the angular range where the full solid angle of the detector is available to the analyst, yielding the highest performance and sensitivity for the analytical system being characterized. Knowing this tilt sequence profile, the careful analyst can also correct any absolute intensity measurements used in quantification procedures both in the limited and fully unobstructed angular regimes. Finally, using the procedures defined elsewhere together with known values of the incident probe current and Ge layer thicknesses the solid angle of the detector can be measured [3,4].

References:

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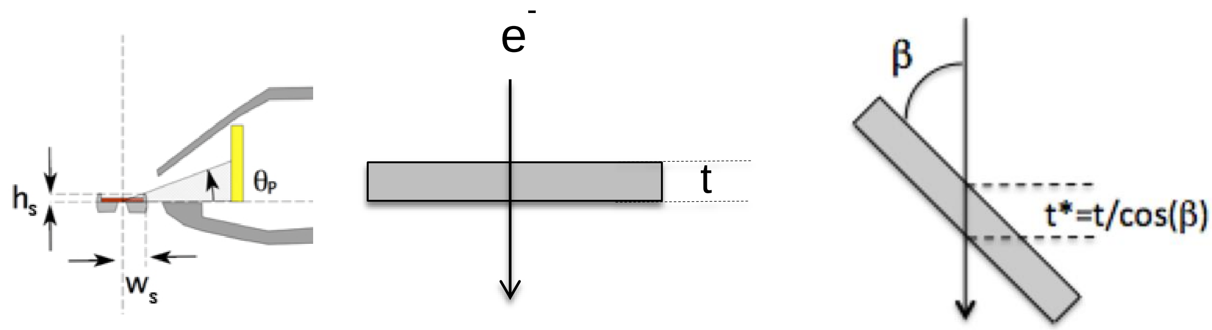

Figure 1. a) The penumbra of the holder is defined by its , width, side wall height as well as the detector elevation angle geometry . b.) Conventional flat specimen of thickness t, c.) tilted specimen with effective thickness t* increased by cos (β).

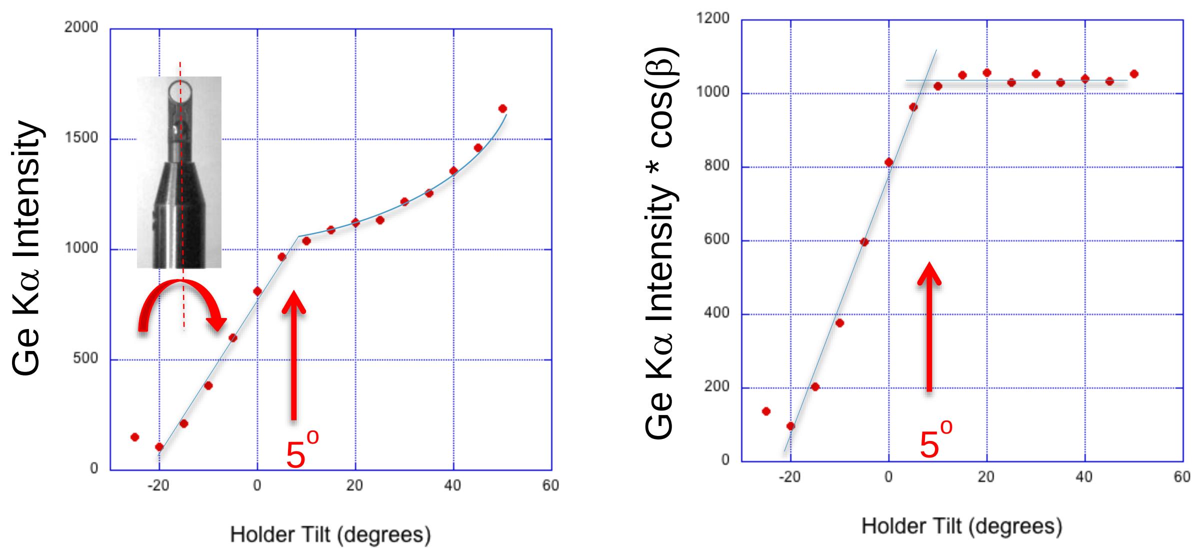

Figure 2. a). Raw data plotting the variation in Ge Kα intensity as a function of holder tilt at constant beam current. Notice the linear regime where the detector shadowing due to the holder penumbra decreases as the tilt is increased, a clear transition at 5° tilt and then an inverse-cosine variation . b.) Data in 2a, multiplied by the cosine of the holder tilt. The flat region represents the angular window in which the full solid angle of the detector is available for measurements. The location of the transition point (5° in this example) will vary with detector elevation angle and holder design. These measurements were made on a FEI Tecnai F20 at 200 kV having an EDAX windowless SDD with θ_E ~ 10° and a customized low penumbra single tilt XEDS holder .

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