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Alternative Windows for the Ultrahigh Collection Angle π sr Transmission X-ray Detector

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The development of an ultra high collection angle transmission x-ray energy dispersive spectrometer (TXEDS) was reported last year where it was demonstrated that geometrical collection angles in excess of **π** steradians could be achieved using a novel detection geometry [1-2]. While these TXEDS detectors can provide as much as 300 times improvement in the collection efficiency for microanalysis of nanoparticles compared to some geometries they are inherently more sensitive to window imperfections/impurities.

The first generation of the TXEDS detector employed a12.5 µm thick Be window. This window was sufficient to isolate the detector from the ambient environment, however it had one serious drawback, namely particulate contamination. The nominally 99.9% pure Be sheet was mechanically thinned by rolling between stainless steel platens. This rolling process left small but detectable particles of steel embedded within the surface of the window material. Due to the proximity of the window to electron scattering from thin specimens in the transmission geometry these contaminates sometimes give rise to systems peaks in the resulting spectra. This can be seen in Figure 1, which shows a backscattered electron image of the surface of the Be window as well as two x-ray maps demonstrating the presence of Cr and Fe in the window.

In order to overcome this problem we have reengineered the environmental isolation window to permit its removal and replacement with alternative materials. The traditional sealed isolation window was removed and a new exchangeable cover was designed to fit over the "naked" detector. Pumping of the interspace of the detector is achieved using the conventional vacuum pumping system of the electron microscope. Special to this configuration is the support system for the window, which is necessarily of large diameter (9 mm). Organically grown carbon based fibers ~ 100-150 µm in cross-section are stretched over the diameter of the cover plate and the window material suspended over those fibers [3]. An example of this can bee seen in Figure 2, where a 300nm thick nanocrystalline diamond window is supported over the entire width of the open cover plate. The use of a limited number of fibers mitigates the problems associated with venetian blind configuration, which is typical of the polymer windows supported on Si grid bars, where more than 20% of the active area of the detector is obscured. Figure 3 presents spectra recorded with the original Be window and with the window replaced by a thin Al coated Mylar substitute. The reduction of the Cr K, Fe K, Ni K systems peaks is evident. Diamond windows have also been employed however because they are transparent to visible photons and self-luminescent they must have an Al reflective coating.

References

[1] Zaluzec, N.J. "Innovative Instrumentation for Analysis of Nanoparticles: The π Steradian Detector", *Microscopy-Today*, 17, #4, 56-59 July 2009, doi: 10.1017S1551929509000224

[2] Zaluzec, N.J. "Detector Solid Angle Calculations for X-ray Energy Dispersive Spectrometry" *Journal of* Microsc. Microanal. 15, 93–98, 2009 doi:10.1017/S1431927609090217
[3] patent pending, contact Argonne National Laboratory Technology Transfer Office for details.
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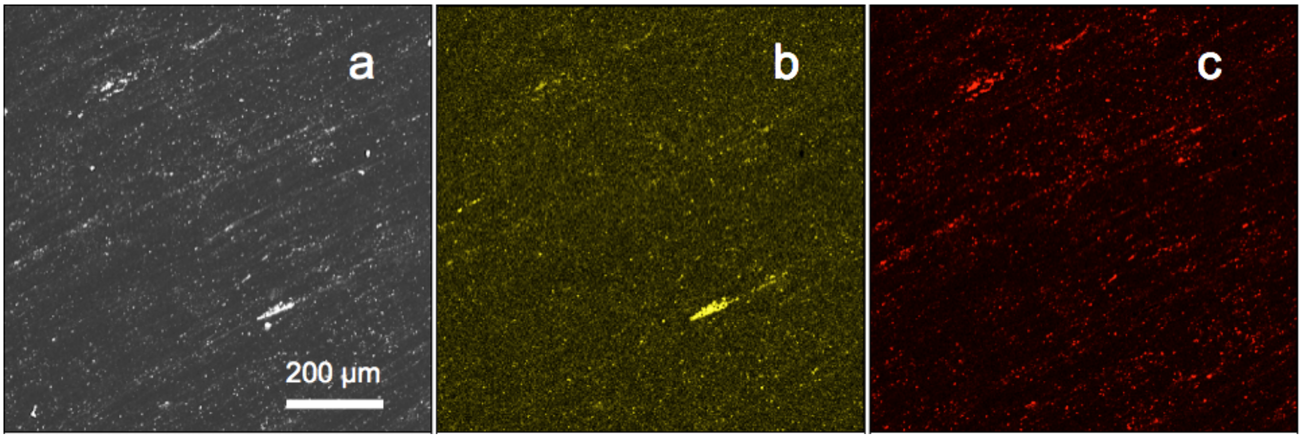

Figure 1 a.) Backscattered electron image employing atomic number contrast of the buried contaminates in the Be window,b.) Cr Kα and c.) Fe Kα x-ray maps of the same region. Fe/Cr contamination is remains from the fabrication/rolling process of the Be sheet.

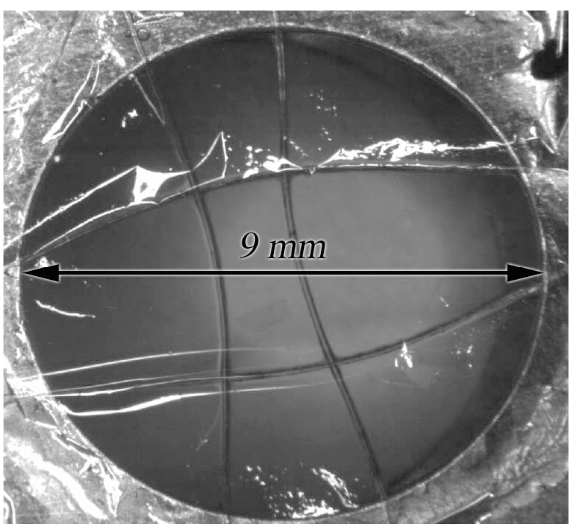

Figure 2. 9 mm diameter / 300 nm thick nanocrystalline diamond window with 150µm support fibers.

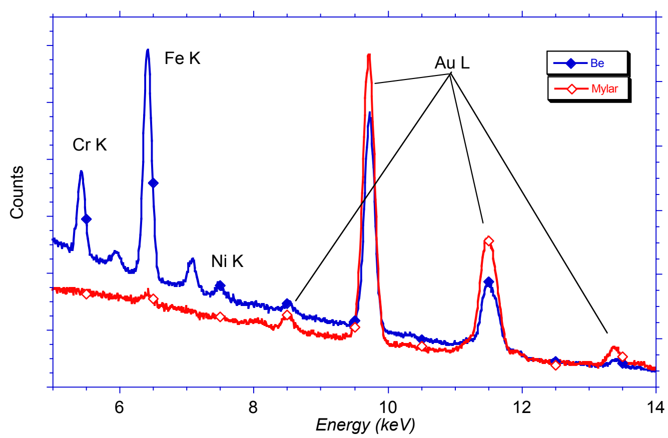

Figure 3. Comparison of Au thin film spectra, using Be window having SS particulates and thin mylar window material. The absence of the Cr, Fe, Ni systems peaks with the hydrocarbon window verifies the source of the system peaks.