

## The Real World Practical Issues and Limitations of Silicon Drift Detectors' on Electron Microscopes.

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As TEMs have started replacing SEMs in advanced materials and process characterization due to shrinking feature size in advanced materials, the need for EDS on TEM has increased significantly. The challenges of EDS on a TEM differ from traditional EDS on SEM issues. While the TEM was historically a research tool - now it is often a production tool, and sometimes even an in-line tool. X-ray generation is limited on TEMs; therefore, increases in detector solid angle are desired in order to capture the maximum data in the shortest timeframe. High end TEM users in materials science, semiconductors and nanotechnology are analyzing samples with smaller and smaller sizes. If sample takes too long to be analyzed, it may drift or be damaged.

This drives the need for faster EDS on a TEM. Also, the nature of the sample – a 3 mm disk, thin enough to be electron transparent, the small amount of material to analyze, and the difficulty of sample prep make it critical to get analysis quickly and cleanly. The interaction volume is very, very small and contamination happens very quickly. Once the sample is contaminated - after as little as a single scan - it has to be removed and cleaned (which may not be enough) or a new sample produced.

Silicon Drift Detector's (SDD) have replaced Silicon Lithium Drift detectors (SiLi) as the preferred Energy Dispersive X-ray Detector on Scanning Electron Microscopes (SEM) and Transmission Electron Microscopes (TEM). The advantages of eliminating liquid Nitrogen cooling and allowing higher count rate capability have proven to be very attractive to users of EDS detectors. SDD's have very small capacitance, allowing low noise at shorter processing times [1]. This means that higher count rates can be tolerated with low dead times. Operation at the higher count rates that SDD's allow does increase the possibility of sum peaks in the stored spectrum, however, and these can easily be mistaken for trace elements that don't exist in the sample. Measures must be taken to minimize the size of these peaks by careful inspection of the incoming data [2]. The SDD has only one fundamental limitation compared to a SiLi detector which is the useful energy range that it can detect efficiently. This is not an issue for SEM's, as a 30keV beam of electrons does not efficiently excite X-rays greater than 15 keV. While higher energy lines are excited on TEM's, usually the lines less than 10 keV are used for analysis.

This change from SEM to TEM analysis has pushed the need for the largest solid angle: SDD's can be manufactured in many sizes and shapes. They can also be made in arrays of several detectors in one package. This gives detector designers more options to create designs that have a larger solid angle.

When applied to TEM/STEM systems, solid angle is of great importance. Usually solid angle is calculated using the simple formula  $\Omega=A/d^2$  with A= the detector area, and d= the distance from the sample to the center of the detector.

As Zaluzec [3] has pointed out, this formula is not accurate for the tight geometries that are used on TEM's, and overestimate the solid angle. There are other factors in the designs that affect the effective solid angle.

Experimental measurement of the solid angle is problematic. While there is a procedure [4] and a sample available for the measurement, there are difficulties to get an accurate value. The sample

thickness is known only to  $\pm 10\%$ , and a beam current must be measured. This can only be done accurately with a faraday cup. Most TEM users do not have a Faraday cup available, and substituting screen current measurements lead to further inaccuracies.

If a window is used on the detector housing, it usually has a support structure which absorbs X-rays which would otherwise impinge on the detector. The most universally used of these structures is said to pass 80% of the X-rays. But when the distance from the sample to the detector becomes small, there is a larger shadowing effect due to the thickness of the support material. Figure 1 shows a typical window support structure. Figure 2 shows the shadow pattern on the detector front face when the sample to detector distance is on the order of 10mm. The white areas are illuminated by X-rays, while the black areas of the detector are not illuminated. The shadowed portion of the detector is about 40 to 45% of the detector area, reducing the effective solid angle by that amount.

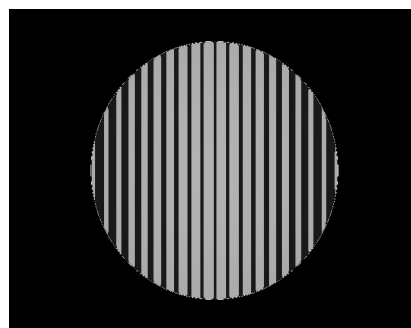
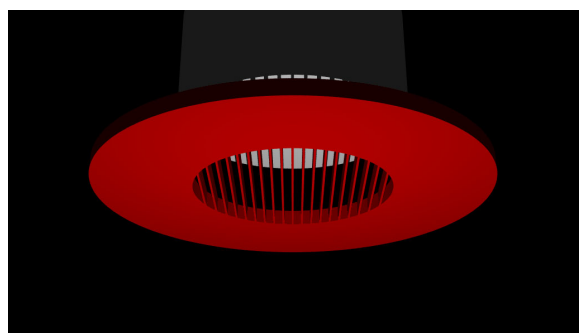
Windowless SDD systems are very practical due to the higher temperature operation of the device. This eliminates the window support structure shadowing as a reducer of solid angle. Windowless detectors can realize the full area of the detector and thus achieve the highest possible solid angle. They should be the preferred device for TEM/STEM systems.

Large, oval shaped detectors have become available [5]. This allows a more favorable geometry with greatly increased solid angle. When these devices are used without a window and its support, 1 sr solid angle detectors are now possible. This new detector geometry provides fivefold increase in count rate over previous SDDs used on TEMs, and opens doors to atomic level X-ray mapping. Additionally, this new geometry will allow simultaneous EDS/EELS collection, which had been previously held back by the slower speed of EDS at 0.3 sr.

Also as part of the move to faster and more powerful EDS on a TEM, detectors for the TEM are moving to a windowless design- both to increase the solid angle and to enhance the light element sensitivity.

#### References:

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**Figure 1.** Frontal view rendering of a Si window support. **Figure 2.** Shadow Pattern of X-rays on a detector.