Background Analysis and Modeling

The background or continuum radiation is often overlooked or even ignored in Energy Dispersive Spectroscopy (EDS) analysis, mostly because it is assumed that it contains no relevant information. However, by a more or less detailed analysis of the background signal, significant information can be extracted and the background itself can be a very beneficial troubleshooting tool.

The emitted background is generated by electrons changing velocity as they move through the sample. The most easily accessible piece of information hidden in the background is the Duane-Hunt limit [1]. Basically, the energy of the incoming electron determines the highest energy X-ray that can be emitted, meaning that the tail of the background tells us what the landing energy of the incoming electron was. Figure 1 shows the change in landing energy depending on the charge build-up on a quartz sample. Under high vacuum conditions, the electrons have about 3.5 keV of landing energy, at 2 Pa about 5 keV and at 30 Pa there is no charge build-up resulting in a landing energy of 15 keV, corresponding to the acceleration voltage of 15 kV. So, if there's any question about whether a sample is charging or not, it is a good idea to look at the high energy background to see where it tails off.

To extract more information, it is often necessary to model the background. With the release of APEX™ 1.5, it is now possible to select between two different background methods, namely Statistical Non-linear Iterative Peak clipping (SNIP) [2] and Bremsstrahlung [3]. SNIP is basically a mathematical filtering/fitting approach to estimate the background signal while Bremsstrahlung is based on a full physical model of the sample and setup to calculate the background. An example of the two background methods can be seen in Figure 2.

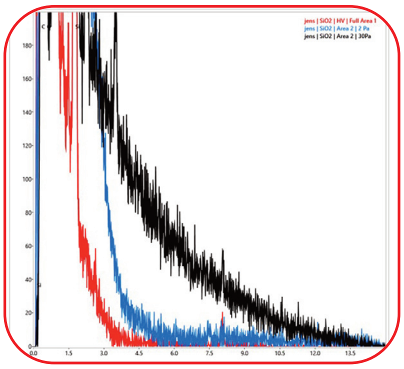

Figure 1. Quartz spectra at 15 kV showing variations in landing energy depending on vacuum conditions high vacuum (red), 2 Pa (blue), 30 Pa (black).

The main advantages of the SNIP approach are that it does not require any user input, gives consistent results and will give a good visual fit regardless of geometry and sample surface. This means that it works very well for qualitative analysis of non-ideal samples such as fracture surfaces and particles, the downside is that any 'red-flags' will be hidden since SNIP is a filtering approach.

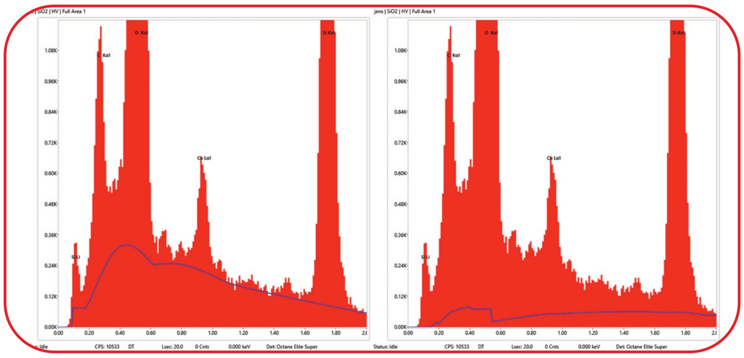

Figure 2. Example of SNIP method (left) and Bremsstrahlung model (right) on the same spectrum.

Bremsstrahlung on the other hand takes the entire geometry and composition of the sample into account in order to calculate the theoretical background. This means that if a "bad" fit is seen with bremsstrahlung, it is a good indication that the assumptions of the model are not met and since the assumptions are similar to that of the quantification model, it means that quantification results will most likely be incorrect. The input parameters to the Bremsstrahlung model include the sample composition and sample geometry and one of the often-seen issues is an over/under-estimate of the background in the low energy region. Figure 3 shows an example of this, where the low energy signal in increased due to tilting the sample towards the detector, which leads to an underestimate of the background since it was calculated for a flat sample.

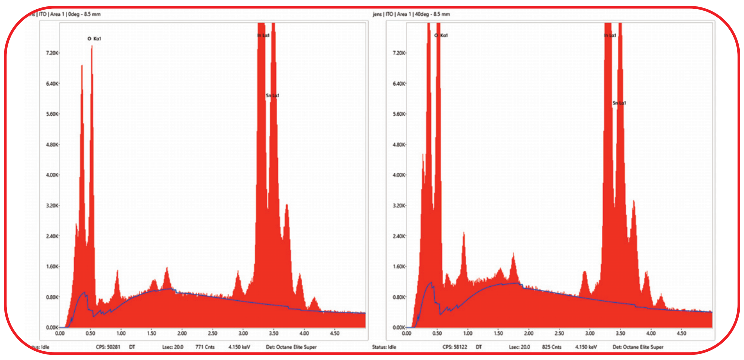

Figure 3. Spectra from an ITO sample where the left spectrum was collected with the stage flat while the stage was tilted towards the detector in the right spectrum. The background model was calculated for a flat sample in both spectra.

If the stage is flat but an underestimate of the background is seen using a Bremsstrahlung model, this indicates that the sample is not flat but tilted towards the detector. On the other hand, if the background is overestimated it indicates that the sample is tilted away from the detector or shadowed. Note that the sample self-absorption also influences the background calculation, so it is important to identify all elements present before looking at background deviations.

Bibliography

[1] W. Duane and F. L. Hunt, "On X-Ray Wave-Lengths," Physical Review, vol. 6,pp. 166-172, 1915.

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[3] F. Eggert, Microchim Acta, vol. 155, pp. 129-136, 2006.