EDS/WDS Geometry and the Famous Take-off Angle

Introduction

X-ray spectrometry in Scanning Electron Microscopes (SEMs) has routinely been used for decades. EDAX is proud to be a pioneer in this technology with one of the first Energy Dispersive Spectroscopy (EDS) systems designed for SEM applications. The qualitative and quantitative analysis of the measured raw data (the spectra) requires the utilization of complex non-linear models, which were developed over time and further progress is still being made today.

The fundamental physics to consider are the X-ray generation depth and self-absorption of generated X-rays in samples, which both depend on the geometry. Geometry influences the final quantitative matrix-correction used to calculate the concentrations, it is also required for the bremsstrahlung background calculation and line-series overlap deconvolution (the element line-series differ in ratios, which is also due to the absorption situation). Therefore, one can say that the geometrical situation influences, in a crucial way, the entire shape of the spectrum (the measured data), which includes the bremsstrahlung background with absorption jumps, the line ratio of peak series, and the total emitted (and measured) count ratios between different element line-series. The 'understanding' of the measured spectra can then be quite complex and everything that is visible with spectra and what is calculated from spectra evaluation depend on the correct geometry calculations (proper geometry modeling). One example of complexity is a Monacite spectrum (Figure 1). The element line series of La, Ce and Pr are highlighted with different colors, to show their proportions (after deconvolution). Even though this point is very crucial for the evaluation of all the X-ray data, there can still be some confusion with geometry definition and angles.

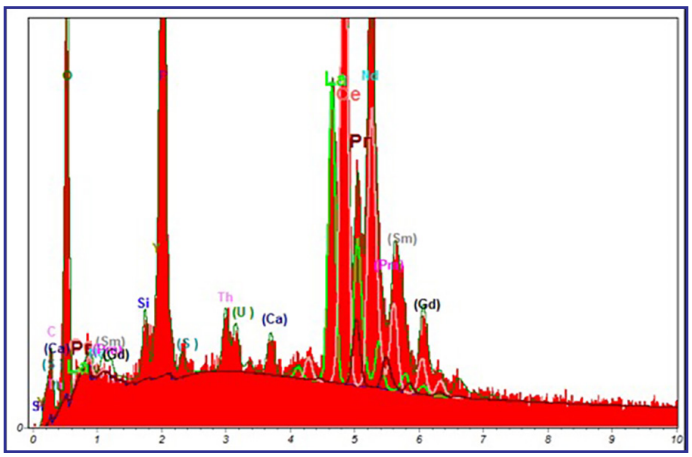

Figure 1. A Monacite spectrum showing the element line series of La, Ce and Pr, displayed after deconvolution.

Early on, the "Take-off" angle was defined and introduced as a basis for X-ray model geometry understanding, with the first Electron Probe Microanalysis (EPMA) systems, the Electron Microprobes. Simply, one can say that this angle is defined to be the angle of (mean) X-ray exit direction from a sample, similar to the way an aircraft or rocket launch is defined. It is the angle between the X-ray direction to the center of the detector and the sample surface. Logically this take-off cannot exceed a maximum of 90°, which is when the X-rays are emitted from a sample perpendicular to the surface (in other words, the detector axis is perpendicular to sample surface). With the basic "Electron Microprobe", there was no ability to tilt the sample. Therefore, the "Take-off angle" (TOA) was defined by the design of the entire instrument. It characterized the absorption situation in samples biunique and was equal to the "Elevation angle" (EA).

As it is now possible to tilt the specimens in SEMs, the TOA is usually calculated as the sum of EA and "Tilt angle" (TA) in simplified cases if the detector azimuth position is zero (perpendicular to the tilt axis). Until this point, the TOA is still representative for the real X-ray excitation and absorption situation. Roughly, this is indeed the case if small tilt angles are used, only small deviations (mistakes) will be in all calculations. But with a more careful look, one can see this is not the fact and big deviations will occur with high tilt samples.

Example 1:

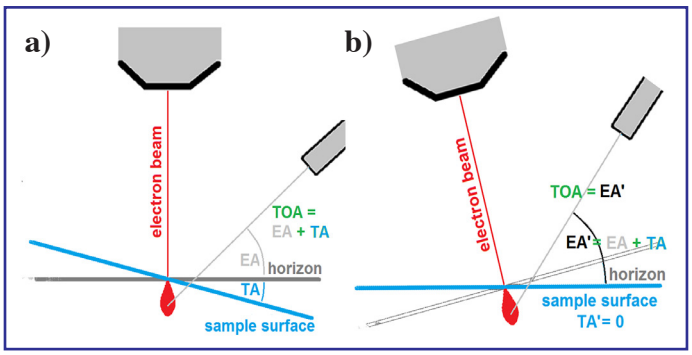

Figure 2. a) Normal setup in a SEM with a tilted sample and b) same picture but twisted, same TOA but in reality the electron column is never tilted.

In Figure 2a, the normal setup in a SEM (two-dimensional, simplified for the case of "Azimuth angle" (AA) is zero). The TOA is the sum of EA and TA.

It is assumed that another SEM setup with a bigger " elevation angle", just EA = EA + TA, then one can twist Figure 2a with the "tilt angle" and with a second setup the TOA will be the same in case the sample is not tilted (Figure 2b). Both setups have identical TOA independent of the tilt of the electron beam. But as one can see very easily, the absorption situation in the sample would only be the same, if the electron column is also tilted. But this is not the reality.

We can learn from this example that the TOA only represents one part of the absorption situation, the trajectory orientation of the emitted X-rays vs. the sample surface.

A second part depends on generated X-ray depth distribution (generation distance from sample surface). This part depends on the electron beam incidence angle in relation to the sample surface. And it is assumed in Figure 2b to be the same as in Figure 2a. But in reality, with the Figure 2b setup and no tilt (same TOA), the electron column stays perpendicular to the horizon and therefore the real depth distribution is deeper than Figure 2b suggests. Finally, despite both cases having the same TOA, the geometry for the entire X-ray excitation and the absorption physics are actually different!

Example 2:

One can imagine that with the usual high-tilt case used in Electron Backscatter Diffraction (EBSD), the TOA based models run out of order completely, because the TOA model does not consider the depth distribution, which is much reduced with a large slope sample. A usual EBSD tilt situation is with TA = 75°. If assuming a normal EA = 35°, then the TOA calculates 75° + 35° = 110°. By definition, the TOA is never > 90°, so it can be determined that the final TOA = 70°. So, does it mean in cases with an EA = 35°, that the TA = 75° has the same X-ray characteristics as a TA = 35°? It is easy to understand that this is not true. With the TA = 75°, the excitation of X-rays is much closer to the sample surface than with the other case, but both have the same TOA. The final emission of X-rays is much higher in the "high-tilt", 75° case, than with the 35° condition, even though the absorption path orientation is the same (represented by TOA). It is because the generation depth is closer to the surface with a 75° tilt.

The TOA depicts only one aspect of the final X-ray emission situation, the absorption of X-rays, which are generated in the depth. The second aspect, the actual depth distance from surface, cannot be considered by TOA. This is another example showing that a TOA based metric is not a complete base model for X-ray physics in a sample and therefore it is not suited for correction models with tilted samples.

Consideration of two independent angle parameters EA and TA is necessary, to get the complete picture of the geometrical situation with X-ray emission from a sample (with AA being zero). TOA is a derived value, one can calculate the TOA from EA and TA. But it is not possible to get the complete geometrical picture from one angle parameter (TOA) alone.

EDAX is using another geometrical model in place of the classical TOA geometry base. First with TEAM™ EDS spectra evaluation, which is properly, geometrically defined with taking tilted samples into consideration: "Improved EDS Performance at EBSD Geometry" [1]. There was the statement: "An EDS quantification model, termed eZAF, which incorporates a blend of the most advanced models with recent and more precise atomic mass absorption coefficients, was used to quantify…"

The vast majority of the high-tilt situation improvement with EDS spectra evaluation was that eZAF replaced spectra evaluation algorithm without using a TOA founded geometry calculation, compared to legacy software models.

EDAX spectra evaluation algorithms have been using the following geometrical formula since the TEAM™ EDS software was introduced:

Absorption path of X-rays = depth of X-ray production in material * geo

(Equation 1)

The 'depth of X-ray production in material' is the depth distribution of generated X-rays inside the sample material with electrons perpendicular to the horizontal plane surface incidence.

$$geo = \frac{\cos(TA)}{\sin(TA) * \cos(EA) * \cos(AA) + \cos(TA) * \sin(EA)}$$

(Equation 2)

The parameters are: TA is the tilt angle; EA is the elevation angle and AA is the azimuth angle (angle between the perpendicular of the specimen stage tilt axis and the real detector position).

For no tilt cases, the formula transforms into a very simplified version, also in a 3-dimensional view:

$$geo = \frac{\cos(TA)}{\sin(TA) * \cos(EA) * \cos(AA) + \cos(TA) * \sin(EA)}$$

(Equation 3)

And this is exactly the case for old Microprobes, where no tilt was possible normally, one could use the TOA = EA.

Equation 3 also represents cases with AA = 90°, if the detector is in a position parallel to the tilt axis. Then any tilt is meaningless (if a simplified assumption is made that the X-ray emission is from a point-source and there is no divergence; in reality, this is only if the detector area is very small and the detector is far away from the sample).

However, not everything has been taken into consideration. The formulas used are only proper in cases where the geometry is exactly in the designed ideal analytical position, the sample "working distance" (WD) is exactly at the point where the detector axis is striking the electron beam at sample surface. In practical cases this is often not true, actual WDs are not exactly at the intersection point:

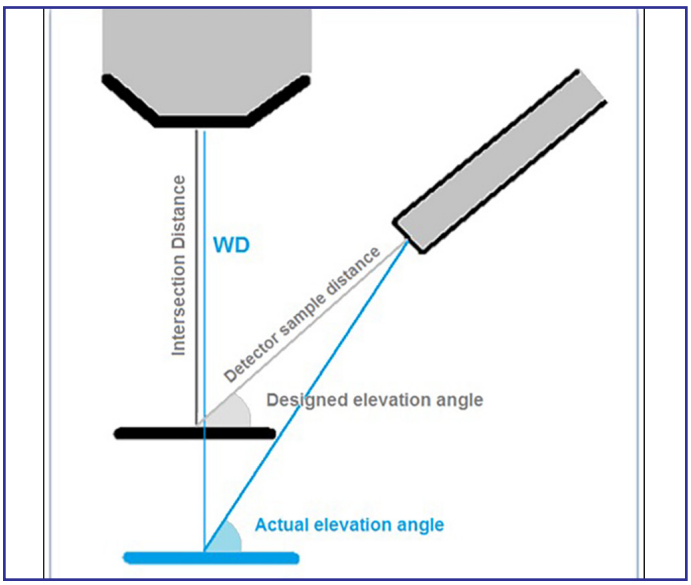

Figure 3. Shows the effects when the WD is not exactly in the optimal position.

"The correct detector elevation is important for accurate quantification as the matrix correction has a strong dependence on this parameter." [2]

Therefore, it is necessary to distinguish an "actual elevation angle" (or "effective elevation angle") from the nominal angle, the "designed elevation angle". Equations (2) and (3) used EA nominally, as if the operator really measured exactly at intersection point WD what is usually the ideal WD. If there are deviations, then the absorption in the sample differs. The "actual elevation angle" is required to calculate the base geometry values:

"Typically, the optimal sample position is located on the electron-beam axis at an optimal working distance. At this distance, the effective-elevation angle equals the nominal elevation angle. …

The difference between the effective elevation defined by the actual working distance and the nominal elevation as defined by the intersection of the detector axis and the optic axis. The effective evaluation angle can be calculated from the actual working distance given the optimal working distance, the nominal elevation angle, and the nominal sample-to-detector distance." [3]

The EDAX spectra evaluation models and algorithms follow this citation. The "actual elevation" angle is always used, which is normally different from the "nominal elevation". But Figure 3 also shows two other effects in cases where the WD is not exactly in the optimal position:

1. There is a deviation between the detector axis and incoming X-rays, a "detector input deviation angle".
2. The "detector sample distance" is changing, different from the nominal.

Indeed, the elevation angle changes and the effects of no perpendicular X-ray entrance into the detectors do not produce huge absorption deviations in many analytical cases, compared to other error components. But it matters more the closer the detectors are to sample. In general, and more in cases where detectors are close to the sample, the change of "detector sample distance" is affecting the solid angle (the total fraction of all emitted X-rays compared to all the directions emission). In many cases, this change can be in the order of magnitude of several %. In all cases, it is required to calculate and carefully consider if an absolute view is performed with X-ray spectrometry, e.g. with eZAF results that are not normalized based on reference measurements. It is also very important for full "standards-based quantification", in this case even with normalized results. The solid angle change does not affect the PeBaZAF model results, which are not normalized, because characteristic radiation and bremsstrahlung have practically the same change in solid angle, and the effect cancels out.

Conclusion

The "take-off angle" parameter is no longer a good metric for X-ray data evaluation in the SEM. More appropriate geometry models have been used by EDAX since the release of TEAM™ EDS software. Consequently, in its new software platform (APEX™), EDAX will expose the parameters which are actually used, and which describe the geometry situation better and uniquely: the "actual elevation angle" and "tilt". The TOA value being displayed was a concession on a decade long legacy geometry model, people were used to it, but it is not state-of-the-art. The experiences with the high-tilt EBSD parallel measurements, have shown that the old quantitative model, where TOA is used for base geometric consideration, would fail and it is required to be replaced by the geometry view, as the new eZAF [1] was developed for this with the first internal working name of "HiTiltZAF".

References

1 Nowell M M, Anderhalt R, Nylese T, Eggert F, de Kloe R, Schleifer M, Wright S I (**2011**) Microsc. Microanal. **17** (Suppl 2) 398

2 Goldstein J at al. (**2018**) Scanning Electron Microscopy and X-Ray Microanalysis, 4th Edition, Springer, New York, p242

3 Goldstein J at al. (**2018**) Scanning Electron Microscopy and X-Ray Microanalysis, Edition, Springer, New York, p240ff