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A New Wavelength Spectrometer for SEM Analysts

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Wavelength Dispersive Spectroscopy (WDS) is a very powerful technique for many analyses using electron beam instruments. The two most popular and effective analyses are peak detection for low amplitude or energy-overlap peaks and quantitative composition analyses.

The highest precision data comes from instruments that are dedicated to these measurements, i. e., microprobes. These "SEM on steroids" are much more than high beam current instruments; they are purpose-built systems designed for electron beam stability measured in weeks. Predominantly, this means that lower amplitude peaks and lower composition materials can be analyzed with a greater certainty. The disadvantages are that the instrument is typically more costly than an analytical SEM, and the level of user expertise necessary to operate is quite high. Typically the expertise is needed in the area of adjusting the electronics settings of the spectrometer for the optimum detection of x-rays.

The traditional x-ray sensor of choice for WDS spectrometers is a proportional counter, typically a flow counter. The amplification from different x-ray energies can be compensated by adjusting the bias voltage on the counter, however, compensating for the flow rate due to atmospheric pressure changes is much more difficult. The variation in this amplification requires that the detection electronics be adjusted or measured for each use of the spectrometer. This means that the settings are not "set and forget" and required much attention by the analyst.

WDS spectrometers have been attached to existing analytical SEM + EDS instruments and have produced some spectacular results. However, the analysts should not expect that the precision of the data should always meet that of a dedicated microprobe because the beam stability of typical SEM is measured in hours, not weeks. Any imprecision in the beam current directly affects the precision of the x-ray detection. In addition, the skill necessary to optimally operate a WDS microanalysis system is so high that a very experienced operator is needed to collect high quality data at a high level of confidence. With analyst's time being diluted with using a multitude of techniques in the lab, this level of expertise is difficult to maintain in many labs.

So the question becomes, "What WDS capabilities do I need on a common SEM + EDS system to obtain high confidence results?" Quantitative compositional analyses, elemental mapping and linescan analyses, and peak detection are just as important in the SEM as in the microprobe, but the expected detection limits of the data should be relaxed. Since most SEM operators investigate samples with unknown elements, an additional result from a WDS spectrometer that could benefit the EDS operator would be peak identification confirmation, especially for overlapped energy peaks. This is a known issue with EDS spectral analyses that has been described in great detail [1]. The high-spectral resolution capabilities of WDS are a primary application that can be applied to this most difficult analysis.

The typical methods to confirm elemental peak identification are peak-to-background measurements and peak energy scans. The former method is preferred because it is faster to measure only the peak and background energies. The total movement time, although less than the energy scan, can still take a significant time to perform the analysis, so it is only performed when deemed necessary by the operator. This operation requires manual identification by the operator of a problem situation and a

manual selection of the P/B acquisition for each element to be confirmed which requires much expertise on the part of the analyst.

With these limitations of WDS spectrometer designs in mind, a new spectrometer has been produced which has the performance capabilities that SEM operators need to obtain results with the highest confidence possible. The two major design considerations were in the speed of crystal and detector positioning and amplification stability. The final design has direct-drive high-speed motors and encoders for the Theta and 2-Theta axes. For vacuum integrity, these assemblies are mounted externally from the vacuum system and the motion is transferred via ferro-fluidic mechanical feedthrus. These feedthrus permit a much higher rotation speed than other vacuum feedthru technologies while maintaining the same high vacuum environment. The second design consideration was to reduce the amount of expertise that the operator needs to routinely operate the spectrometer at the highest efficiency possible. This required the substitution of the flow proportional counter with a sealed unit. The advantage is that the electronics settings are continuously adjusted to optimized values with no input from the user.

Some microanalytical results of these design considerations are shown in the following figures. In Figure 1, a summary of the WDS P/B measurements are shown which confirms both the existence and the absence of potential peak overlaps. No user intervention was necessary except enabling the software setting to activate the automatic WDS confirmation. The total time needed to perform the 4 P/B measurements was less than 70 seconds. Figure 2 shows a plot of the quantitative composition measurements from 16 distinct particles of 160A stainless steel reference material. The standards for these analyses were collected two days prior to the quantitative analyses. The precision and accuracy of the quantitative measures are superior to typical WDS standards analyses in an SEM.

In the past, WDS was an accessory to an SEM that has provided some unique capabilities. The latest generation of spectrometer provides necessary capabilities to the microanalysis systems that increase the SEM analyst's confidence in the results using very little user expertise.

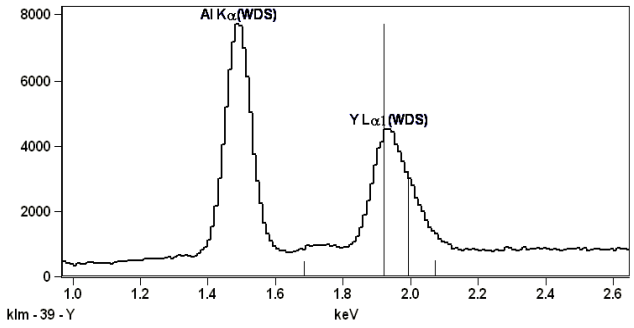

Figure 1. Display of automatic WDS peak identification confirmation routine for turbine sample.

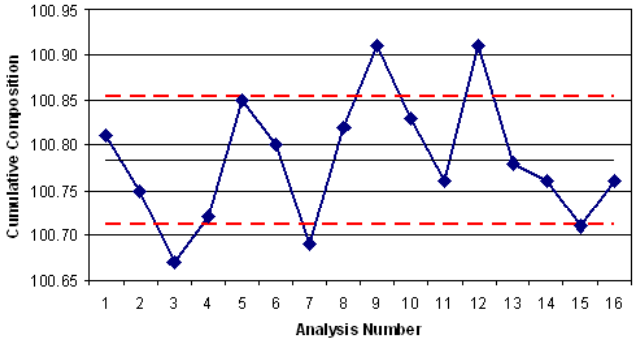

Figure 2. Plot of cumulative composition for 16 points of a 6-standard WDS analysis.

References

[1] D. E. Newbury, Microsc Microanal 11(Suppl 2), 2005, pp 1286-7; Microscopy and Microanalysis (2005), 11:6, pp 545-561; Scanning (2007), 29:4, pp 137-151.

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