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WDS Energy Scans

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Key Words

- High Order Peaks
- MagnaRay
- Overlapped Peaks
- Resolution
- Spectra
- WDS

Abstract

In many samples, peak identifications in EDS spectra can be difficult to interpret, especially for energy peaks with competing elemental lines or with low amplitudes. Manual peak confirmation using WDS energy scans can be very useful to reduce the uncertainty of these locations. In addition, the increased motor speed of the Thermo Scientific MagnaRay permits wide energy range scans in a fraction of the time of conventional WDS spectrometers. These wide energy scans have many benefits, but can also lead to a few complications if the analyst is not aware of the consequences. This paper provides numerous examples of the benefits and potential pitfalls of energy scans with WDS.

Background

The underlying principle behind all WDS analyses is Bragg's Law:

$$n\lambda = 2\,d\,\mathrm{Sin}\theta$$

where

n is the order of the diffraction,
λ is the wavelength of the X-ray, inversely proportional to the energy,
d is the lattice spacing of the diffractor,

and

θ is the diffracting angle.

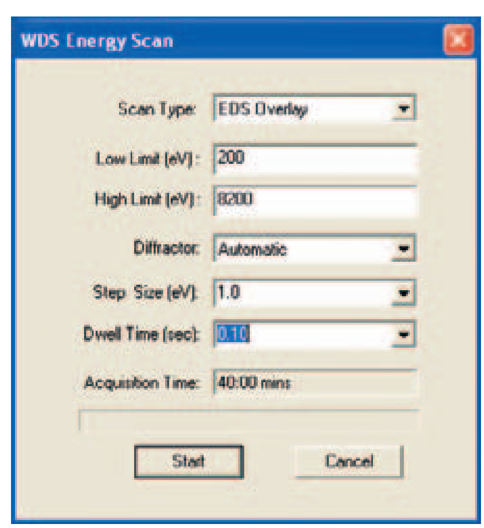
X-ray waves that come from the sample and become incident on an analyzer diffractor only "reflect" from that diffractor and have a high intensity at the detector at specific angles. The dispersion of this angle depends on the focus of the X-ray beam and the homogeneity of the diffractor lattice. The WDS spectral resolution is typical 2-20 x better than the EDS resolution at the same energy. The benefit is the ability to separate closely spaced peaks using WDS that would overlap into a single peak in EDS.

Another result that occurs due to the diffraction is the appearance of energy peaks at integral fractional energies to the primary peak energy. These derive from the "n" factor in Bragg's Law. Because the X-ray energy and n are inversely proportional, Bragg's Law also predicts relatively high X-ray intensities at integral fractions of the incident X-ray energy: E/2, E/3, E/4 etc. These additional peaks, although significantly lower in intensity than the primary peaks, can lead to peak identification issues if their appearance is not predicted and accounted for.

Results

MagnaRay has a single-button function that opens an energy scan dialog, Figure 1. When the dialog appears, a graphical overlay appears on the spectral display showing the energy-range extents of each of the diffractors that are mounted in the spectrometer. Most of the values in the dialog are automatically preset to optimized values based on the current energy-range limits. These values include the diffractor(s) to use and the energy step-size of the scan. Users may choose to override the default values where necessary. The energy range limits can be edited via spectral display slider bars or text input in the dialog. The currently selected diffractors will be indicated in the spectral display by a solid line in the diffractor range limits. If changes in the energy range limits trigger changes in the diffractor selections, the energy step size will automatically update. The only value that is not automatically set is the dwell time for each energy step; that selection is left to the analyst based on the current SEM and X-ray generation conditions.

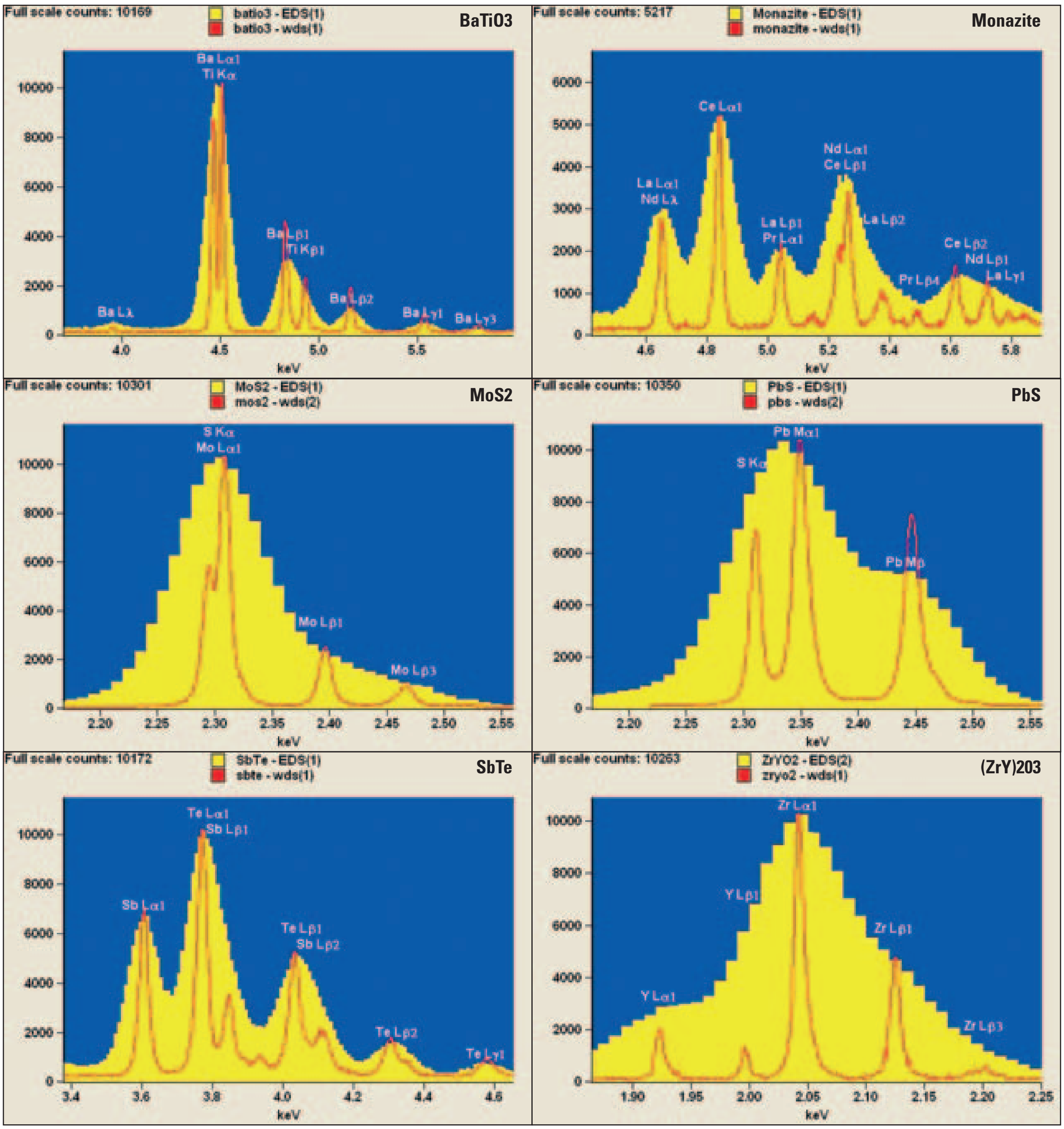
When the value selections are satisfactory, clicking the OK button starts the WDS energy scan with the original EDS spectrum automatically overlaid as a background spectrum. A progress bar is displayed to indicate the amount of time left for the acquisition. As the WDS spectrum develops, both spectra are automatically scaled to display both spectra in an optimum fashion. When the acquisition completes, all of the spectral data from all of the diffractors are automatically saved into a single industry standard EMSA format file.



The following sections provide examples illustrating the benefits of WDS energy scans for peak separations, and complications due to higher-order diffraction.

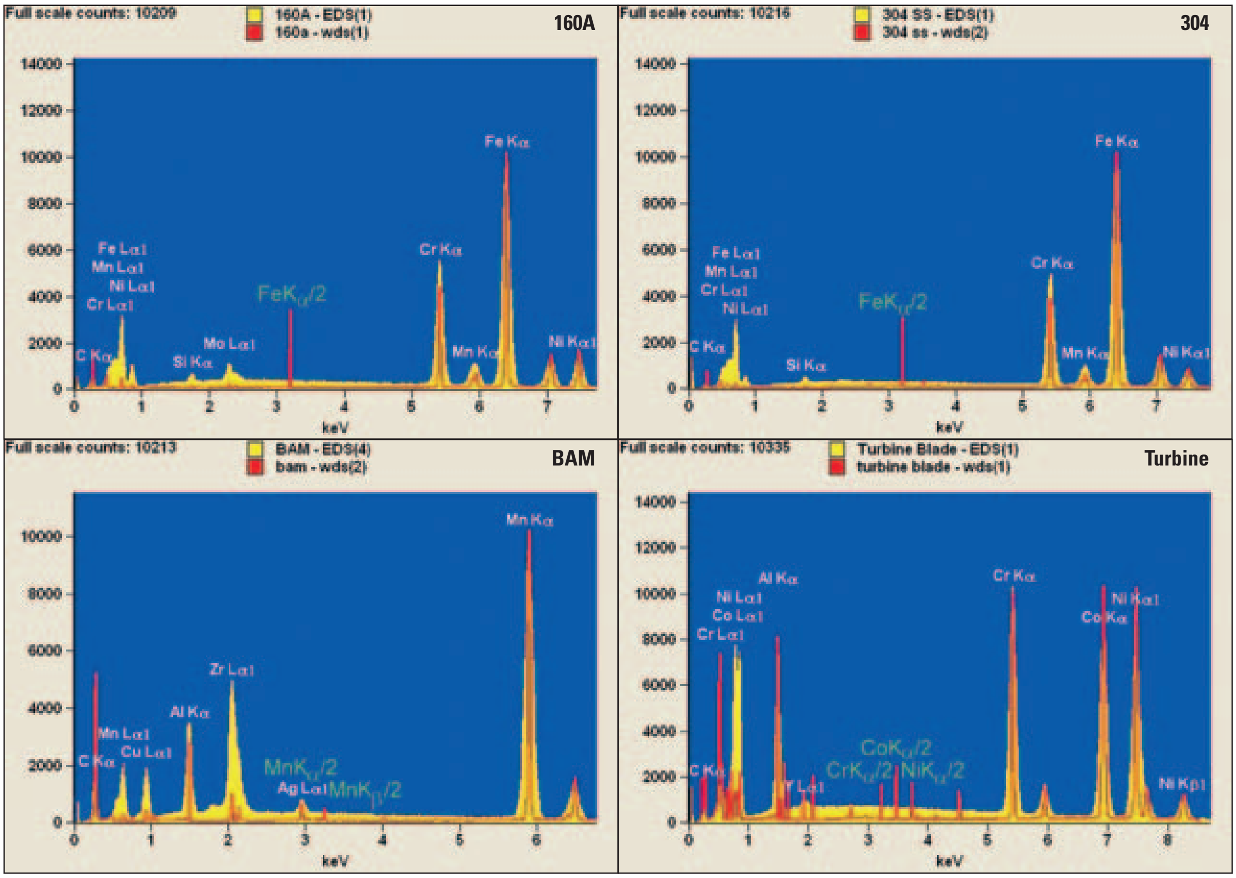
Peak Overlaps

These figures show EDS spectra in solid yellow overlaid with WDS spectra in red of the same material illustrating peak overlaps. The superior spectral resolution of WDS is easily observed by the narrow separate peaks in the WDS energy scans. Although WDS has enough spectral resolution to separate most overlapped EDS peaks (for instance Pb-S and Sb-Te), there exist a very small number of peak overlaps that are not fully resolved (for instance Mo-S).



High-Order Peaks

These figures show EDS spectra in solid yellow overlaid with WDS spectra in red. Note the relatively high peaks in some of the WDS spectra that do not correspond to any EDS peaks. These higher order peaks are labeled in green. Although only a few seem to be visible while the spectra are displayed with a linear scale, there are actually third-order peaks in all of these spectra, but at much reduced amplitudes. Most times they do not interfere with first order peaks, although some combinations may cause problems.



Summary

MagnaRay is a very powerful and flexible WD spectrometer that can be used to collect energy scans. Most of the settings are optimized by the system so the analysts can concentrate on the tasks at hand and not on the specifics of WDS operation. These energy scans are useful for manual peak identification confirmation over small energy ranges. However, MagnaRay also provides the capability to perform wide-energy scans using multiple crystals with no additional effort. Care must be taken in labeling WDS peaks to include higher order peaks not appearing in EDS spectra.

MagnaRay is the perfect complement to EDS for analysts to tackle difficult EDS spectra with overlaps. Restricting WDS peak identifications to EDS peaks avoids high order peak misinterpretations.

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