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Enhancement of Low Energy / Light Element Energy Dispersive Analysis in the Scanning Electron Microscope Using a Non-Focusing X-Ray Optic

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Microanalysis with Energy Dispersive Spectroscopy, EDS, is commonly performed in the SEM chamber. Interaction of the electron beam with the surface of an unknown sample leads to the emission of secondary characteristic x-rays which can be collected by an energy-dispersive detector. The resulting x-ray spectrum can be used to perform qualitative and quantitative elemental analysis of the sample.

The weakest portion of the spectrum collection and subsequent analysis is the range from 100 to 1000eV, which is caused partly by the absorbance of the low energy x-rays in the sample matrix itself, as well as in the detector.

The use of a non-focusing optic to increase the solid angle collection efficiency of the sample x-rays between 100 and 1000eV and the resulting increase in x-ray counts will be examined with several samples to compare with data from O'Hara et al. [1]. X-ray spectra, x-ray maps, and line-scan data will be presented to show the resulting increase in sensitivity using the optic with both $10mm^2$ and $30mm^2$ active area x-ray detectors and contrasted with data collected without use of the optic. Reduction in scan times, improved sensitivities, qualitative analysis, quantitative analysis, and precision are discussed. The Be sensitivity for analyses such as published by Butnor et al. [2] may have been improved with the use of such a device.

Details and characteristics of the optic will be presented. The optic, a polished grazing incident mirror, is a metallic cone with a compound elliptical shape. When mounted and aligned in front of the detector, the optic acts as an x-ray concentrator by collecting x-rays diverging from the sample at a wider angle and redirecting them into a smaller diverging angle towards the detector, resulting in an effective solid collection angle gain. Techniques and hardware associated with the optic will be explained.

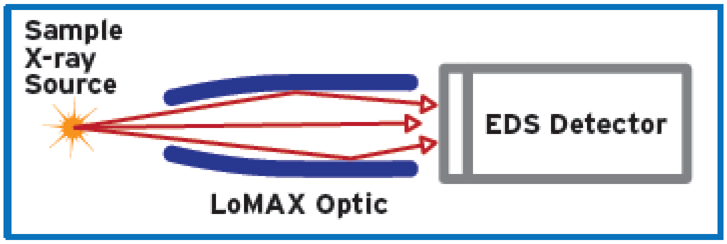
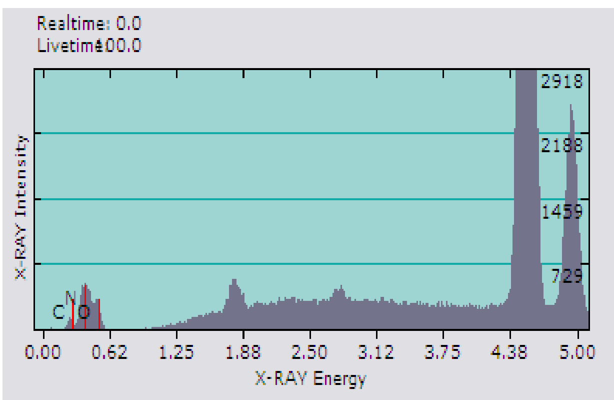
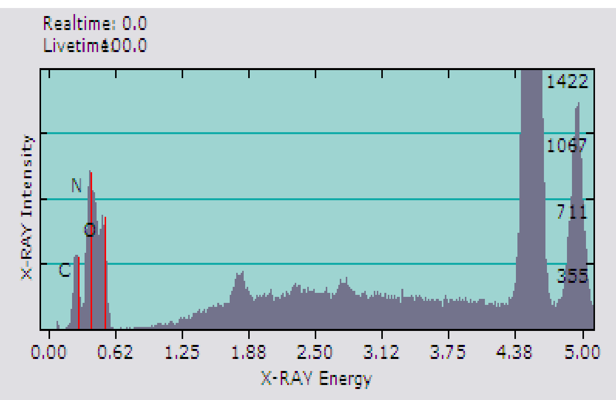


Figure 1

Figure 1 shows schematically how diverging soft x-rays, that would otherwise miss the detector, are reflected towards the detector. The optic is made with a compound elliptical shape and interior metallic reflecting surface polished to less than 1 nm (rms). Using a nickel surface, increases in solid angle gain range from 2X to 10X, depending on x-ray energy and analytical conditions, can be obtained for x-rays from 100 to 1000eV.



Spectrum 1



Spectrum 2

Spectrum 1 is from a TiN – TiC powder using a light element detector with the standard collimator attached. With the LoMAX® reflective optic attached, and the entire spectra normalized at the Ti peaks in Spectrum 2, the gains shown in this initial experiment are approximately 6X for Carbon, 4X for Nitrogen, and 3X for Oxygen.

References

[1] D. O'Hara et al., Improving EDS For Low Energy X-Rays Under 1000eV Using an Attachable Detector Optic Microscopy Today (Mar 2008) (Vol 16 - #2) 6 [2]

[2] Butnor K. J., et al. Beryllium detection in human lung tissue using electron probe X-ray microanalysis Modern Pathology 2003, vol. 16, n°11, pp. 1171-1177

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