

QUANTITATIVE COMBINED XRF AND EPMA ANALYSIS IN THE SEM

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Over the last decade *in-situ* XRF has become available [e.g., 1, 2] in many Scanning Electron Microscopes (SEMs), providing an additional quantitative analytical capability to conventional Electron Probe Micro-Analysis (EPMA), also known as SEM-EDS analysis. The advantages and disadvantages of XRF, compared with conventional SEM-EDS, are well known [e.g., 3], and the two techniques are naturally complementary. Electron beam excitation is superior for low-energy lines (with energies below about 2 kV), while x-ray photon beams are more suited for exciting higher-energy lines. In addition, XRF is much better for analyzing trace quantities of elements with lines above 2 kV, because of the much lower spectral backgrounds. Electron beam excitation is fundamentally limited by the deceleration of electrons in the sample (except for ultrathin films), producing a background continuum of emitted x rays from zero to the incident electron energy.

Of course, the relative beam sizes and penetration depths of electron and x-ray beams are quite different, so the sample should be homogeneous over the volume of the (larger) x-ray beam, in order to fully compare or integrate the two techniques. Electron beams typically produce x-ray analysis areas of about 1 micron, whereas the x-ray beam sizes are much larger. Some current x-ray sources use integrated optics to produce beam sizes below 10 microns, but often the beams are in the range between 50 and 500 microns. Analytical and penetration depths are also generally 1 to 2 orders of magnitude larger with XRF.

We will explore some of the different strategies that can be used to quantitatively analyze different samples with spectra from both x-ray and e-beam sources in the SEM. with examples to show how the strengths of each technique may be combined in a single analysis. The Figure below shows an example of the combined XRF and SEM-EDS analysis of an aluminum alloy, without the use of any calibration standards. In addition to quantitative analysis, these x-ray sources can be used to map samples by scanning the sample using a motorized XY-stage. This provides spatial information of trace elements over a large area.

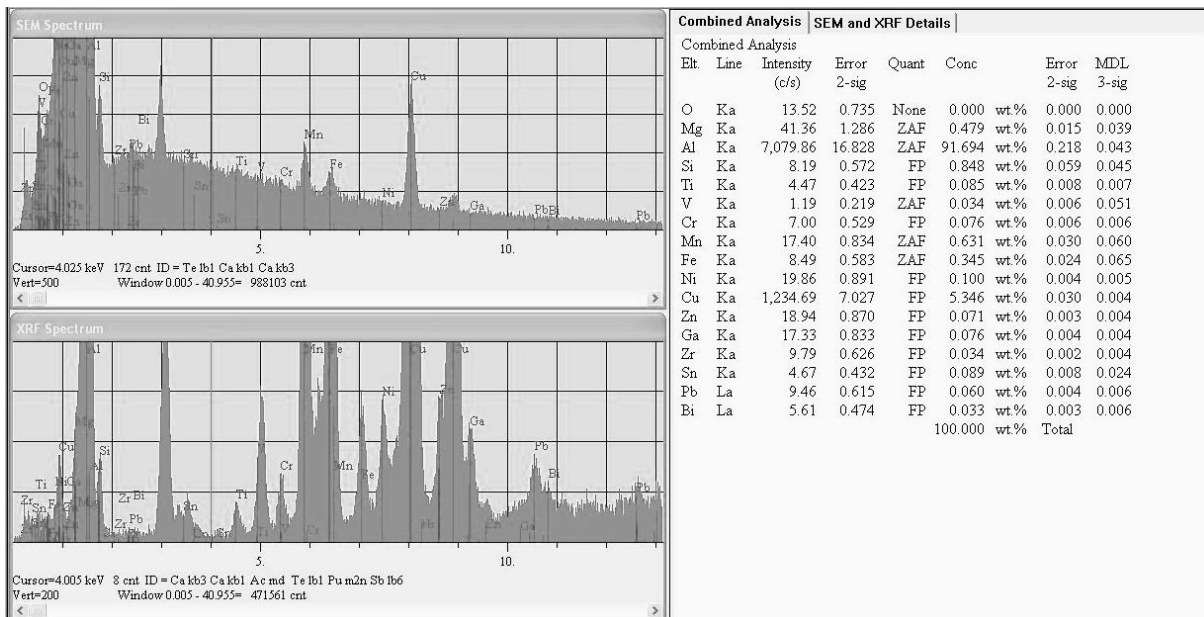


Figure: Combined XRF and SEM-EDS analysis of an aluminum alloy with no standards.

References:

- [1] B.J. Cross, & K.C. Witherspoon, *Microscopy & Microanalysis*, **10(2)** 104 (2004).
- [2] M. Procop and V.-D. Hodoroaba, *Microscopy & Microanalysis*, **13(2)** 1424 (2007).
- [3] B.J. Cross and J.E. Augenstine, *Advances in X-Ray Analysis*, **34** 57 (1991).