EAGLE II - Capillary Optics in Micro XRF Spectrometry (μ - XRF)

Problem

Elemental microanalysis measurement techniques are required today that can keep up with the growing trend of miniaturization in science and technology. Required tasks include determination of composition of small particles, forensic investigation of elemental distributions in linear and rectangular sample areas, microstructural analysis, and analysis of small impurities.

Energy dispersive X-ray spectrometry has always offered a key advantage for elemental microanalysis in the ability to analyze a wide range of elements simultaneously. This method has long been used extensively in electron microprobe analysis, but requires an electrically conductive sample under high vacuum. Another significant limitation with electron excited microanalysis is the relatively poor detection limits due to electron excited bremsstrahlung radiation.

Excitation with X-rays enables significant improvements in sensitivity. In the past, creating a high intensity X-ray beam with diameters <100 μm has been difficult. Traditionally, apertures were used to reduce the beam's diameter. Using apertures however, results in several problems:

- Only a small portion of the X-rays emitted by the tube is used for sample excitation.
- Most of the significant beam intensity is lost over the path between the tube, aperture and sample.
- Short distances between the tube and aperture cause high divergence of the X-ray beam.
- Irregular shaped samples are difficult to analyze with these short distances.

Solution

The recent breakthrough for focusing the X-ray beam has been capillary optics. By this technique, X-rays are totally reflected through a special glass tube without losses. The principle relies on maintaining the critical angle (θ_C) for total reflection by:

$$\theta_c (E) \cong 2.04 * 10^{-2} \sqrt{\frac{\rho}{E}}$$

With : ρ = material density (g/cm^3)

E = radiation energy (KeV)

Radiation incident at angles $<\theta_c$ are totally reflected by the capillary wall and focused on the sample (Figure 1).

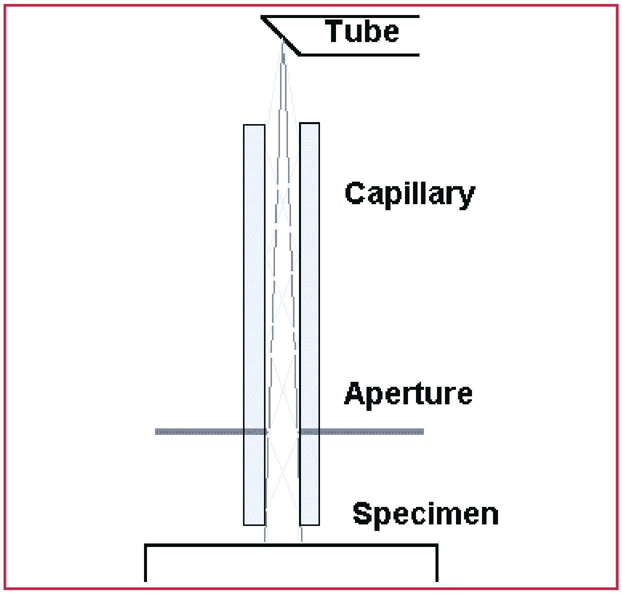

Figure1: Illustration of improved solid angle as a result of capillary optics compared with use of an aperture.

Small capillary diameters allow multiple total reflections. Therefore, radiation leaves the capillary with a divergence angle which is equal to the critical angle (i.e. very little divergence).

Radiation can be collected within a large solid angle because of the short distance between the tube window and the capillary. The increased incident X-ray intensity on the sample results in greatly improved elemental sensitivity. The intensity improvement from capillary optics is especially enhanced in the lower energy range. Figure 2 illustrates the factor of 5-20x improvement in incident beam intensity below 15 keV.

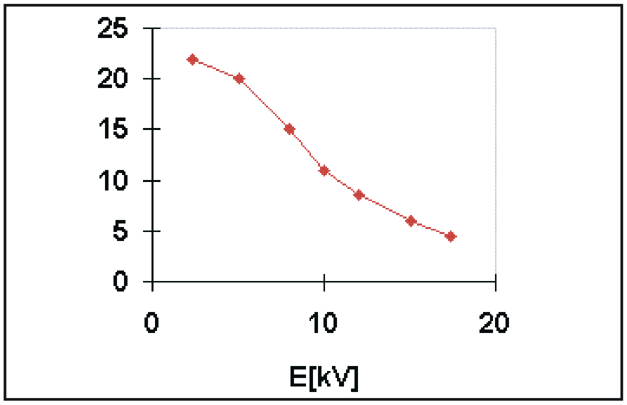

Figure 2: Ratio of intensity improvement by use of capillary optics over use of aperture.

Modification of Excitation Spectrum

The multiple total reflection of the excitation radiation in the capillary modifies the excitation spectrum: higher energies are suppressed, while lower energies are enhanced. This results from the energy dependence of the critical angle (equation above) as illustrated in Figure 2.

A comparison of the primary and capillary spectrum is shown in Figure 3. The two spectra are recorded from the tube spectrum with and without the capillary.

This modified incident energy distribution results in more efficient excitation of elements sodium (Z =11) through Niobium (Z = 41). The enhanced sensitivity is taken into account when calculating quantitative analysis results.

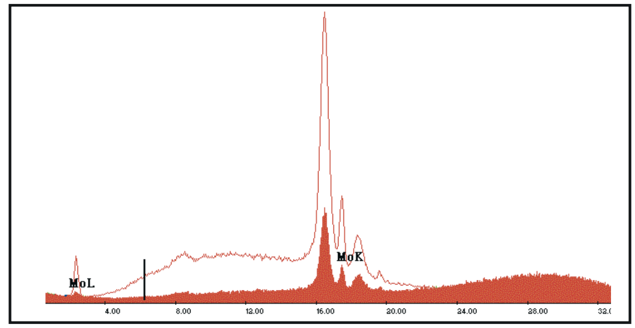

Figure 3: Comparison of excitation spectra with (dots) and without capillary (solid).

Intensity Distribution of Radiation

The beam intensity at the capillary outlet exhibits a spatial distribution as shown in Figure 4. The distribution can be characterized by a Gaussian function. Therefore, X-ray spot size is typically specified at the full width at half-maximum (FWHM) of the distribution.

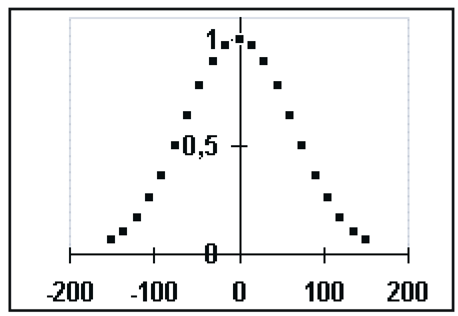

Figure 4: Intensity distribution at capillary outlet.

Advancements in X-ray Focussing Optics

The field of X-ray focusing optics for use in XRF is demonstrating improvements for intensity and spot size all the time. Currently, EDAX uses mono-capillaries to generate spot sizes of 300-100 μm in the Eagle II.

In a new development, EDAX uses a poly-capillary lens to generate high intensity spot sizes < 50 μm in the Eagle II XPL. The poly-capillary lens is a mono-lithic glass structure comprised of thousands of tiny capillaries.

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