

Advantages of X-ray Over Electron Excitation in Microanalysis

Introduction

X-ray analysis of small particles requires a small excitation beam. XRF systems typically have too large an excitation area for small particle analysis and therefore electron microscopes have traditionally been used for micro-Energy Dispersive Spectroscopy (EDS). By comparison, electron beams impose some difficulties in sample preparation, limitations in sample type, and analytical accuracy.

The following results give a comparison of both excitation with X-rays and excitation with electrons. Table 1 lists typical parameters for electron beam excited EDS and X-ray excited micro-XRF.

Parameter	Electron Beam	X-ray Beam
Analyzed Area (μm)	0.1 - 10	30 - 2000
Typical count rate (cps)	2K - 500K+	20K - 300K+
Analyzed element range	Be - U	Na - Bk

Table 1: Comparison of typical e-beam and m-XRF analytical parameters.

Sample Preparation

Sample preparation for X-ray excitation is much simpler in many ways:

- Unlike those used in electron excited EDS, XRF samples need not be electrically conductive. There is no electrical charge being transported in XRF samples.
- Measurement of liquid and organic samples is possible because the electron beam does not require vacuum. Samples may remain in an air environment during analysis.
- X-ray excitation does not heat a sample as there is no absorbed kinetic energy of electrons. This means that temperature sensitive samples, such as thin films, can be analyzed.

Analytical Accuracy

From an analytical point of view, there are important differences between X-ray and electron excitation:

- Detection limits (peak to background ratios) for X-ray excitation are much better for heavier elements because backgrounds are minimal in the absence of Bremsstrahlung radiation produced by electron excitation.
- The penetration depth differs for each method. The greater penetration depth for XRF analysis depends on the escape depth of signal X-rays. The much lower penetration depth of the EDS method depends on the penetration depth of the exciting electron beam.
- The excitation efficiency for single elements is different. This influences the detection of light elements.

Detection Limits

Deceleration of electrons in a sample generates not only heat, but also a Bremsstrahlung background spectrum. This can be seen clearly in Figures 1 and 2, where electron excited spectra are compared to X-ray excited spectra.

The detection limits using X-ray excitation are greatly improved by the lower background.

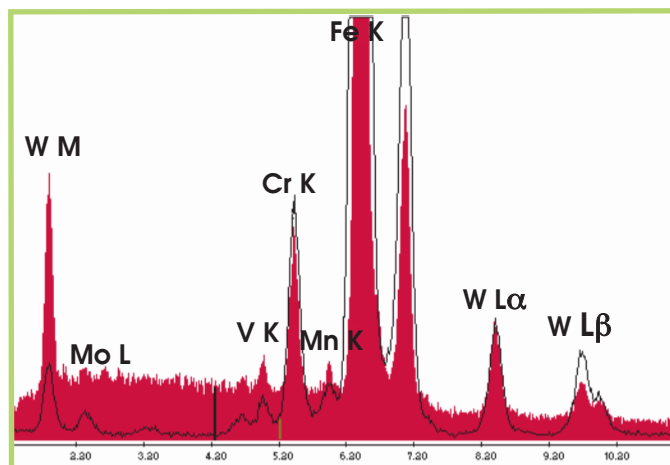


Figure 1: Steel spectra with electron (red) and X-ray (black) excitation.

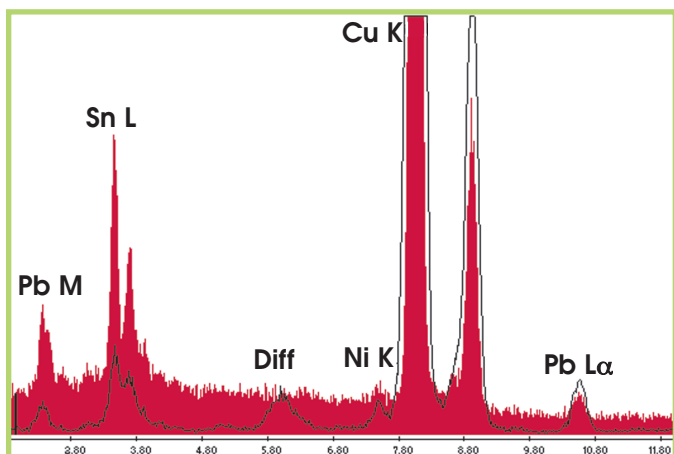


Figure 2: Bronze spectra with electron (red) and X-ray (black) excitation.

Table 2 compares limits of detection for various elements found in typical alloys.

Element	Orbis	EDX (at 40 kV)
Ti	40	1000
Cr	35	800
Mn	30	800
Fe	25	800
Ni	25	900
Cu	25	1000
Mo	150	2000
Sn	250	4000
Pb	150	5000

Table 2: Detection Limits (ppm) for metal alloys.

Analyzed Volume

The sample penetration depth is greater for X-ray excitation. This results in a greater volume of analyzed sample. Figure 3 shows this effect by comparing spectra from an X-ray excited sample with an electron excited sample.

In addition to the background difference, the Cu from the substrate is easily detected in the X-ray excited (red) spectrum. From the electron excited spectrum, it appears that the electrons are mostly absorbed in the upper Au layer of the sample and do not excite the deeper layers. Therefore, the electron excited spectrum exhibits very high intensity Au-L, while the Cu peak is missing. Electron excitation can be insensitive to layered systems.

Excitation Efficiency

While X-ray excitation delivers the greatly improved detection limits listed in Table 2, electron beam excitation does provide better low energy excitation. Figure 3 shows the greater low energy (Au-M) sensitivity with electron excitation.

For the Au/Ni/Cu layered system (Figure 3), the greater Au-M intensity is due both to the high absorption of electrons in the Au upper layer and to a higher cross section for electrons at lower energies. The latter effect is also exhibited by the relatively higher W-M peak in the steel sample of Figure 1.

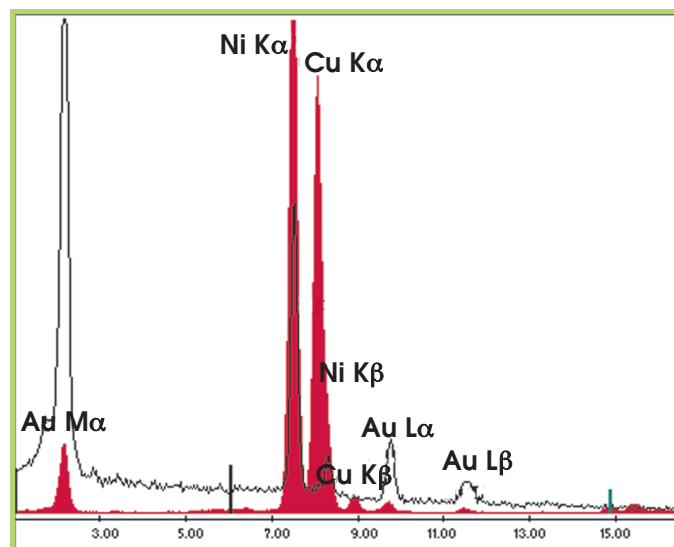


Figure 3: Au/Ni/Cu layer system with electron (black) and X-ray (red) excitation.

Conclusion

The Orbis Micro-XRF Elemental Analyzer utilizes intensifying capillary optics to focus an X-ray beam to less than 100 μm in diameter for micro-XRF analysis. Micro-XRF is a non-destructive analysis method requiring little if any sample preparation, with lower detection limits for heavier elements. EDS has better detection limits for light elements, approximately Be to Cl, but this can be a destructive method requiring sample preparation.

Notes

1. For the spectra shown, it is important to note that the detector used for electron excited EDS is 10 mm^2 , and XRF is 80 mm^2 SiLi.
2. Figures 1 and 2 exhibit diffraction peaks from crystalline samples (labeled as "diff"). These are taken into consideration when identifying the spectra.