MICRO-XRF

Analytical performance of the M4 TORNADO

Application Note # XRF 442

Introduction

Micro X-ray fluorescence (micro-XRF) is an analytical method for the non-destructive examination of small sample areas and the determination of elemental distribution. The analytical performance of micro-XRF instruments is defined by several parameters. This report studies that of Bruker's M4 TORNADO regarding some of the most important measurement parameters.

Instrumentation

The micro-XRF spectrometer M4 TORNADO is characterized by the following features:

- large vacuum chamber for samples up to 200 x 300 x 150 mm³
- fast X-Y-Z stage with TurboSpeed for fast mapping and distribution analysis on-the-fly
- effective excitation of fluorescence through high brightness X-ray tube together with X-ray optics for concentration of tube radiation to spot sizes down to 25 µm

- detection of fluorescence radiation using silicon drift detectors (SDD) with high count rate capability
- quantification with standardless models and use of full pattern fitting.

Spatial resolution

The spatial resolution of a position-sensitive measurement is influenced by different factors like spot size, step size, and measured intensity. The influence of these factors on the spatial resolution will be examined in the next sections.

Spot size

The spot size is one of the main factors that influence the spatial resolution of images. The determination of the spot size is important for the optimization of the analysis results. It is usually measured with the knife edge method. This technique measures the beam intensity in function of the position of the sharp edge of a material.

Figure 1 shows the measured intensity against the position of a Mo-edge and its calculated derivative. This allows the determination of the spot size as the full width at half maximum (FWHM).

Step size

Other factors influencing the spatial resolution of elemental distribution images are the pixel size and the contrast between pixels. In micro-XRF the pixel size is determined not only by the spot size but also by the step size. The spatial resolution can be improved if the step size is smaller than the spot size. On the other hand, a minimum level of intensity is required for good contrast between neighbouring pixels. The pixel intensity can be changed by varying the acquisition time per pixel. This can be done in one step or, if a stage with high reproducibility is available, in repeated frames.

According to these facts, the step size and the pixel intensity have to be optimized for a given spot size. Figures 3 and 4 display element distribution images of a Siemens star, which is a star of thin Cr-coating with reduced distance between the lines used to test the resolution of optical instruments. The thickness of the Cr-coating is approximately 1 µm.

As it can be seen in Figure 3, the reduction of the step size improves the image quality for the same intensity per pixel. This is due to the availability of more information (pixels) for the same distance.

Figure 4 shows the distribution images for the same step size of 5 µm but different acquisition time per pixel. The resolution improves if the measurement time is increased.

The spatial resolution for this sample can be estimated for 5 µm step size and 50 ms acquisition time per pixel to be approximately 25 µm.

These images offer a visual impression of the spatial resolution. Quantitative data can be obtained through measurements on a knife edge. Figure 5 displays the intensity distribution of measurements on a knife edge at different step sizes. The diagram shows that a smaller step size results in more steps between the two intensity levels and therefore allows the measurement of smaller

intensity changes. If spatial resolution is defined as the distance corresponding to the intensity drop from 75 to 25% of full intensity, it improves according to these measurements from 40 µm for a step size of 50 µm to 25 µm for a step size of 5 µm.

Figure 1

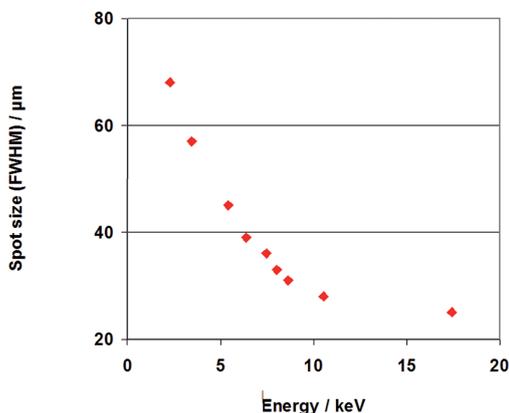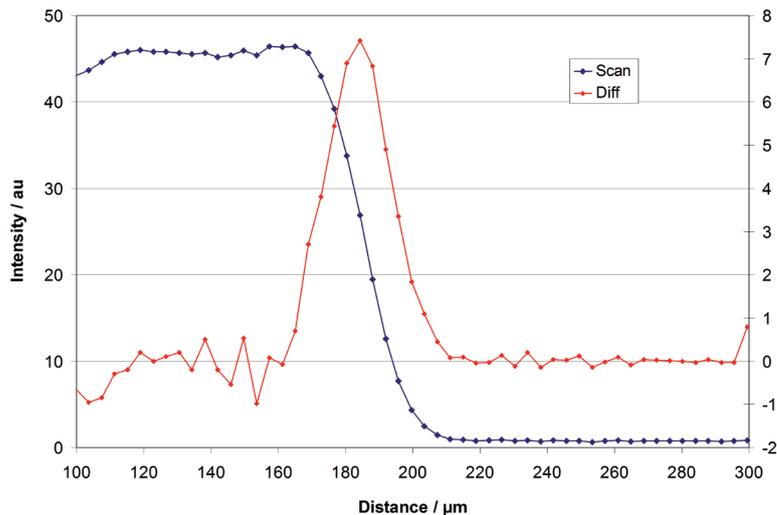Intensity distribution of a knife edge measurement for Mo-K and its derivative.

Figure 2

Spot size versus energy Spot size of a poly-capillary lens against energy

Measured intensity

A prerequisite for good spatial resolution is an optimized measured intensity to step size ratio. This ratio can vary due to the dependence of measured intensity on sample composition, measurement time and excitation conditions. The intensity of the excitation depends not only on the brightness of the X-ray tube and the transmission efficiency of the optics but also on the sample-detector distance. Another important factor is the count rate capability of the detection channel.

The M4 TORNADO typically delivers count rates in the range of 250 to 350 kcps. This excellent count rate capability results from

the use of Bruker´s advanced silicon drift detector technology, which permits processing high count rates without compromising the energy resolution.

Limits of detection

The sensitivity of micro-XRF is comparable to that of conventional energy dispersive XRF (EDXRF). It is determined by the excitation probability of the analyte and the peak-to-background-ratio.

When performing micro-XRF, the transmission function of the X-ray optics must also be taken into account, apart from the tube spectrum. This influences the limits of detection (LOD) for elements with low and high excitation energies. The LOD are low when compared to conventional EDXRF. They were determined using the following formula:

$$ LOD = \frac{3 \cdot \sqrt{BG}}{I^{sample}} \, C^{sample} $$

The limits of detection for different sample qualities are given in Figure 6.

Light element detection

Light elements have low fluorescence energy, for which absorption is high, even in air. Therefore the measurement of these elements should be performed in vacuum. Figure 7 shows the difference between measuring Si in vacuum and in air, which is approximately a factor of 8. The intensity enhancement for the measurement in vacuum depends on the energy of the fluorescence line. The relation between these two factors is shown in Figure 8.

Conclusions

Micro-XRF is a powerful tool for the analysis and the determination of elemental distribution of small particles and inclusions. The analytical performance depends on the measurement conditions. Spot size, measured intensity and step size are crucial parameters that determine the sensitivity and the spatial resolution of the measurement. They have to be selected carefully according to the required analytical task.
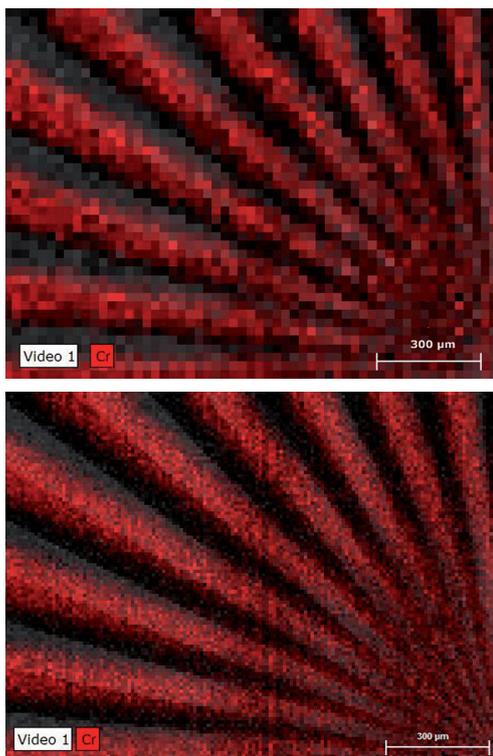

Figure 3

Images of a Siemens star for different step size but same acquisition time per pixel: 25 µm/10 ms (top) and 10 µm/10 ms (bottom)
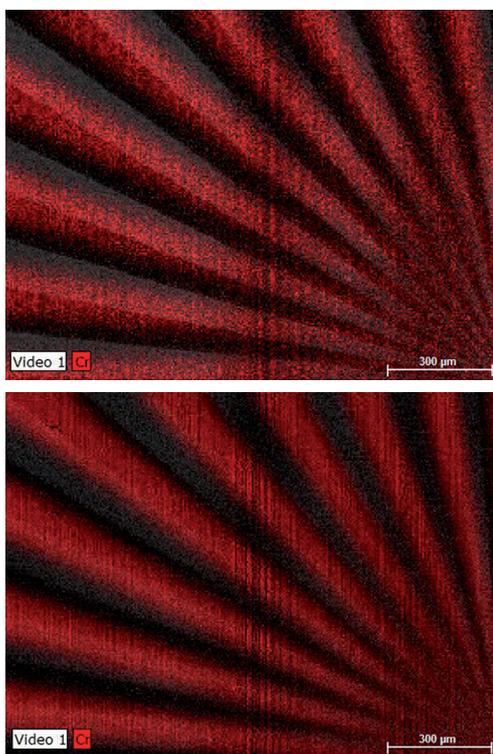

Figure 4

Images of a Siemens star for same spot size but different acquisition time per pixel: 5 µm/5 ms (top) and 5 µm/50 ms (bottom)
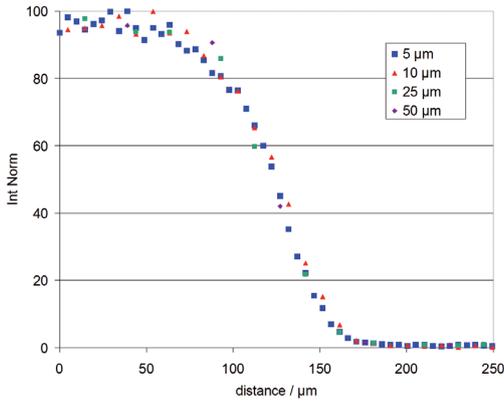
Figure 5

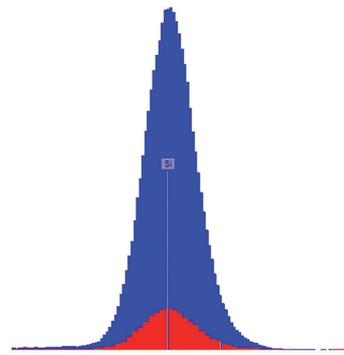Intensity distribution on a
Mo knife edge for different
step sizes

Figure 7

Comparison of Si spectra
measured in vacuum (blue)
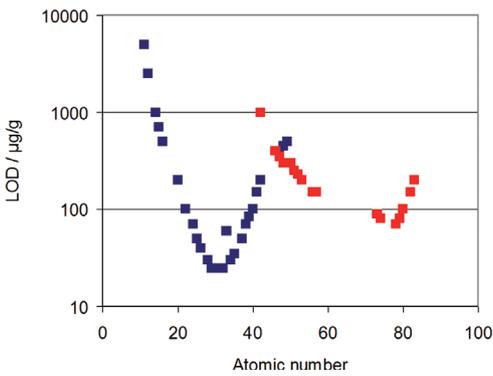and air due to measure-
ment in vacuum (red)

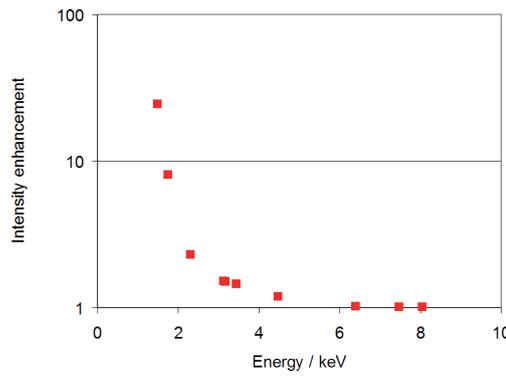**Figure 6**

Typical limits of detection
for micro-XRF

Figure 8

Intensity enhancement due
to measurement in vacuum

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