DEVELOPMENT OF A WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE SPECTROMETER USING A MULTI-CAPILLARY X-RAY LENS FOR X-RAY DETECTION

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ABSTRACT

We developed a new type of wavelength dispersive X-ray spectrometer with a multi-capillary X-ray lens (MCX). We installed a MCX into the X-ray detection side (ie. the crystal spectrometer side) of a conventional WD-XRF machine (Shimadzu XRF-1800). The MCX collects fluorescent X-rays from a selected sample area of diameter 0.1 mm or less and collimates them into a nearly parallel beam form. This spectrometer is able to perform not only conventional macro-analysis but also microanalysis. It is easy to show the characteristic feature of the MCX by comparing the results obtained with those from a conventional aperture. The spatial resolution of analyzing the area was about 60 µm for FeKα. The FWHM of the FeKα line was 0.0089 Å. The energy separation of the MCX for the wavelength region of CrKβ and MnKα lines was better than that of the conventional aperture (3 mmφ). The FeKα intensity from a 60 µm area was about 200 times higher than the conventional aperture. Some mapping images were successfully obtained by using the conventional analyzing position control mechanism.

INTRODUCTION

A multi-capillary X-ray lens (MCX) is an attractive device because of the ability to focus or collimate a wide energy range of X-rays [1, 2]. Table 1 shows recent utilization of capillaries for X-ray fluorescence (XRF) spectroscopy. A single capillary or multi-capillary lens is used at the X-ray source side or the X-ray detection side. Many efforts have been made to use these types of X-ray lens for focusing the primary X-rays [3] and some energy dispersive spectrometers (ED-XRF) are commercially available already. X-ray spot sizes of less than 10 µm have been achieved.

In a previous study, Soejima and Narusawa proposed a new type of X-ray fluorescence wavelength dispersive spectrometer in which a MCX was set at the X-ray detection side for fluorescent X-ray detection [4]. The spectrometer consisted of a MCX and a flat crystal, and had a simple and compact construction with wavelength and spatial resolution good enough for small area analyses. They showed that this type of spectrometer was suitable for X-ray fluorescent analysis of small areas with flat crystal spectrometers due to the large collecting angle.
and the relatively low divergence. In this study, we have installed a MCX into a commercially
available WD-X-ray fluorescent spectrometer (Shimadzu XRF-1800). Since the MCX can be
removed easily from the X-ray optical path, the effect of using MCX can be measured with high
accuracy. Using the spectrometer, the spatial resolution, the wavelength resolution and the
overall detection efficiency for the analysis of small area (less than 100 µm φ) are investigated.

### EXPERIMENTAL

The MCX (XOS Inc.) is specially designed to fit the XRF-1800, and is 23 mm in length and 9
mm in diameter. The focal distance is 9 mm, and its focal point size was designed to be ~60 µm
for FeKα line. Figure 1 illustrates the arrangement of the XRF-1800 in which the MCX was
installed at the X-ray detection side. The original spectrometer has an aperture assembly of 5

<table>
<thead>
<tr>
<th>Lens position</th>
<th>Type</th>
<th>Spatial Resolution</th>
<th>Comment</th>
<th>Status</th>
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<tr>
<td>Source</td>
<td>Single Capillary</td>
<td>&lt;10 µm</td>
<td>low power</td>
<td>commercially available</td>
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<tr>
<td>Source</td>
<td>Multi Capillary</td>
<td>&lt;30 µm</td>
<td>high power</td>
<td>in development laboratory</td>
</tr>
<tr>
<td>Source</td>
<td>Multi Capillary</td>
<td>&lt;100 µm</td>
<td>high power</td>
<td>commercially available</td>
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<tr>
<td>Detection</td>
<td>Single Capillary</td>
<td>-</td>
<td>useless</td>
<td>-</td>
</tr>
<tr>
<td>Detection</td>
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<td>&lt;60 µm</td>
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Table 1  X-Ray Capillary Utilization in XRF

Figure 1  Schematic diagram of the newly developed spectrometer
diameters (0.5, 3, 10, 20, 30 mm). It is able to analyze samples and obtain mapping images with a spatial resolution of 0.5 mm because the aperture restricts the analyzing area. Furthermore, the sample-positioning mechanism of the XRF-1800 enables us to obtain elemental mapping images with a spatial resolution of 0.5 mm [5]. In this work, we have installed the MCX instead of the 0.5 mm aperture in the assembly. We have not modified anything else, including the sample-positioning mechanism. Thus, this spectrometer became able to perform not only conventional macro-analysis, but also microanalysis with spatial resolution of about 60 μm, by selecting the MCX or one of the apertures. Results of measurements from the use of the MCX or a conventional aperture were easily compared.

The primary X-rays from Rh-target X-ray tube (40 kV, 95 mA) hits the surface of samples with the incident angle of 25 degree to the sample normal. The fluorescent X-rays are collected by the MCX or collimated by an aperture with the detection angle of 55 degree to the sample normal. Then the fluorescent X-rays are collimated by the slit 1 (Soller slit), analyzed by a flat crystal, and collimated again by the slit 2 (Soller slit) before being counted by an X-ray detector such as a scintillation counter. For slit 1, three choices of slit spacing are available (750, 450, 150 μm). The slit 1 with a spacing of 750 μm was used in combination with the MCX.

RESULTS AND DISCUSSION

A typical spectrum of a stainless steel sample using the MCX is shown in figure 2, where the detector count rates (kcps) are plotted against the detector angle (2θ). As is shown, peaks over a wide wavelength range are detected by the MCX. The prominent peak at ~57.5° corresponds to the FeKα line. Some lower intensity peaks, such as CoKα, CuKα lines, are also clearly observed. We have confirmed that the MCX is effective for the full range (uranium to carbon) of the spectrometer. However, some properties, such as spatial resolution of the lens, depend on the wavelength. Therefore, the following experiments focus on the X-ray wavelength of around

![Figure 2. Typical spectrum from a stainless steel sample](image-url)
2 Å (FeKα ~ MnKα).

The spatial resolution of the analyzing area was measured by the knife-edge method. The MCX position was scanned across an edge of stainless steel foil in 3 µm steps while the FeKα intensity was monitored. Figure 3 shows the FeKα intensity profile and its derivative, which was smoothed and then fitted with a Gaussian curve. The spatial resolution was evaluated to be 57 µm from the FWHM of the curve, which is close to design specification. The results suggest that the spectrometer is promising for the XRF of small areas.

Figure 3. FeKα intensity profile (dot) and its derivative (solid line) obtained by scanning the MCX across the edge of a stainless steel foil. The derivative was smoothed and fitted to a Gaussian curve.

Figure 4. Spectra of the FeKα line obtained by using the MCX and a conventional 3 mmφ aperture. The spacings of the slit 1 were 750 µm for the MCX and 450 µm for the aperture, respectively.
In figure 4, the FeKα line spectra obtained using the MCX and the 3 mmφ aperture are shown. The wavelength resolution of the MCX was better than that of the aperture in which the spacing of the slit 1 was 450 µm. The reason for this result is that the MCX functions as a good collimator [6]. This suggests that the divergence of the X-ray from the MCX is less than that of the 450µm Soller slit. The line width (FWHM) is 0.0089 Å at 1.937 Å, or energy-wise ~40 eV at 6.4 keV. In fact, the separation of CrKβ and MnKα lines, which is sometimes referred to as the wavelength resolution check, is better for the MCX as shown in figure 5. In Table 2, the wavelength resolution and sensitivity ratio data are summarized. The FeKα intensity from a 60 µm area was about 200 times higher than the estimated intensity for conventional apertures.

![Graph showing spectra](image)

Figure 5. Spectra in wavelength region of CrKβ and MnKα obtained by using the MCX and a conventional 3 mmφ aperture. The spacings of the slit 1 were 750 µm for the MCX and 450 µm for the aperture, respectively.

<table>
<thead>
<tr>
<th></th>
<th>Aperture(3 mmφ)</th>
<th>MCX(60 µmφ)</th>
</tr>
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<tbody>
<tr>
<td>Slit 1</td>
<td>450 µm</td>
<td>750 µm</td>
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<tr>
<td>Wavelength resolution (FWHM ∆λ)</td>
<td>0.0110 Å</td>
<td>0.0089 Å</td>
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<tr>
<td>Peak intensity (kcps)</td>
<td>30.4</td>
<td>2.52</td>
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<tr>
<td>Sensitivity ratio (a.u. / 60 µmφ)</td>
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<td>207</td>
</tr>
</tbody>
</table>

Table 2. Summary of the wavelength resolution and sensitivity

Figure 6 shows an elemental mapping of a Ni mesh obtained using the mapping function of XRF-1800 with the MCX. The pixel size is 100 µm × 100 µm and image size is 2 mm × 2 mm. This is believed to be the first map produced using MCX-WD-XRF. We envisage new
application fields for this type of spectrometer because of its good spatial resolution, good wavelength resolution and good sensitivity.

![Mapping image of NiKα line from a nickel mesh.](image)

**Figure 6.** Mapping image of NiKα line from a nickel mesh.

**CONCLUSION**

A multi-capillary X-ray lens (MCX) was installed into a conventional WD-XRF machine (Shimadzu XRF-1800). It enabled both macro and micro analysis with the same spectrometer. We have shown that the MCX is effective for X-ray detection for wide range wavelength. It has a spatial resolution of 57 µm, and good wavelength resolution of 0.0089 Å for FeKα line. The intensity of the analyzed FeKα line per unit area for the MCX was about 200 times higher than that of the conventional aperture. As the results displayed good spatial and wavelength resolution and high efficiency, some elemental maps with a 100 µm step were successfully obtained. This XRF system shows great merit for small area XRF analyses, and its sensitivity for small area analysis will make it possible to analyze very subtle region of solids such as, for instance, semiconductor-metal interfaces.

**REFERENCES**