Development of 2D dispersive device for XRF imaging spectrometer

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ABSTRACT
Micro-XRF analysis provides us with elemental maps, which are very useful for understanding the samples under test. Usually, scanning-type elemental mapping is performed. That means, a sample stage is scanned to a fixed X-ray micro beam. XRF analysis is performed at the scanned points, leading to 2D elemental mapping. One of the drawbacks of this technique is the long acquisition time depending on the area being mapped and the lateral resolution required. Thus, projection-type elemental mapping has been studied. We have studied the projection type XRF imaging by using a straight polycapillary optic combined with an X-ray CCD camera. To obtain the elemental map, we applied a wavelength dispersive spectrometer (WDS). In this paper, we report a newly developed 2D dispersive device. The construction and analytical performance of this X-ray optic will be explained.

INTRODUCTION
Micro X-ray beams are obtained by using several types of X-ray focusing optics in the laboratory. For example, a recent advanced polycapillary focusing optic gives an X-ray micro beam that is 10 μm in diameter by combining it with a micro focused X-ray tube. In micro-XRF, the micro X-ray beam irradiates the localized point on the sample, and then XRF analysis is performed at the localized region. In advanced materials and micro-structured devices, micro-XRF analysis is very useful for the analysis of contamination particles on the surfaces of devices. Another important application of micro-XRF is elemental mapping (Tsuji and Nakano, 2011). The sample stage is scanned with a fixed X-ray micro beam. XRF analysis is performed at different positions, leading to 2D XRF images. This technique gives XRF images for which the resolution depends in part on the size of the X-ray beam. However, one drawback of this technique is long acquisition times if the imaging area is large. Thus, projection-type XRF imaging has been studied. This method does not require a
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scanning process because the XRF images are obtained by use of an X-ray CCD camera. Sakurai, et al. studied a quick XRF imaging method using synchrotron radiation (SR) and an X-ray CCD camera equipped with a 2D collimator (Sakurai and Eba, 2003; Sakurai and Mizusawa, 2010). Adjusting the SR energy can produce a specific XRF image. A color X-ray CCD camera is also feasible for this process (Strüder et al., 2010; Scharf et al., 2011). Photon counting analysis with a color X-ray CCD camera provided elemental XRF images in a short time.

We have studied XRF imaging by using a straight polycapillary optic and a conventional X-ray CCD camera (Tsuji et al., 2008; Yonehara et al., 2010). Since a conventional X-ray CCD camera does not have an energy analysis function, we considered combining it with a wavelength dispersive spectrometer (WDS). Fig. 1 shows a WDXRF spectrometer equipped with a 2D dispersive device. In this paper, a newly developed 2D dispersive device is reported.

![2D-dispersive device](image)

**Fig.1** WDXRF spectrometer equipped with 2D dispersive device, polycapillary optics and X-ray CCD camera.

### EXPERIMENT

**2D dispersive device**

The 2D dispersive device was consisted of the multiple cells having x-ray reflective multi-layer coatings on the inner walls. Fig. 2 shows the fabrication processes of this device. First, W/C multilayers (d=7.56 nm, N=15) were deposited on a 200 μm thick Si wafer using magnetron sputtering (Fig. 2(a)). Secondly, line and space Au structures were formed on the multilayers using photolithography process (Fig. 2(b)) and selective metal plating process (Figs. 2(c)-(d)). The pattern width and thickness of Au structure were 11 μm and 14 μm,
respectively, and the pattern pitch was 20 μm. In the third step, the Si wafer was cut into 10 mm × 1 mm, and then, these chips were stacked vertically. Finally, multilayer coated multiple cells were formed.

Figure 3 shows the photograph (Fig. 3(a)) and SEM images (Figs. 3(b)-(d)) of the fabricated 2D dispersive device with different magnifications. Dimensions of this device is 10 mm × 10 mm × 1 mm, and each pixel size and pixel pitch are 11 μm × 14 μm and 20 μm × 214 μm, respectively. Thus, the total pixel number of this 2D dispersive device is 500 × 46, and effective efficiency is estimated to be 3.5%.

**WDXRF spectrometer**

The concept of XRF imaging using a 2D dispersive device is shown in Fig. 1. X-ray fluorescence is guided through the straight polycapillary by total reflection. The collimated X-ray beams are respectively dispersed by multilayers of the corresponding cells. Therefore, the X-ray CCD camera can obtain images of X-ray fluorescence without losing the information on element distribution on the surface of the sample.

Fig. 4 is a schematic drawing of the experimental setup used in this experiment. The X-ray tube (Mo target) was operated at 40 kV and 30 mA. The sample was irradiated with primary X-rays through an Al collimator with an inner diameter of 8 mm. Straight polycapillary optics,
especially developed by XOS, Ltd., were applied. These optics consist of several million capillaries. The 1st straight polycapillary has a channel diameter of 10 μm, an enclosure length of 10.5 mm and an outer diameter of 8.3 mm with an open area of 60%. The 2nd straight polycapillary has a channel diameter of 5 μm, an enclosure length of 10.5 mm and an outer diameter of 10.7 mm with an open area of 75%. The 2nd straight polycapillary was fixed to the detector. The 2D dispersive device was attached to the center of an inner rotation stage, as shown in Fig. 4. To investigate the fundamental dispersive characteristics of the device, an EDS detector (Silicon drift detector, Vortex-EX-60, SII Nano Technology USA Inc., sensitive area: 50 mm², energy resolution: 136 eV at 5.9 keV) was attached to an outer rotation stage. The two rotation stages equipped with stepping motors were controlled by a personal computer using motor drive and a motor controller (NT-2400, Laboratory Equipment Co., Japan).

RESULTS AND DISCUSSION
A stainless steel plate and a Ti plate were measured. Fig. 5 shows the XRF intensities measured for these plates as a function of rotation angle (diffracted angle). Since the W/C
multilayer was deposited on one side of the Si substrate, X-ray diffraction should occur only from this side, as shown in the inset of Fig. 4. As expected, additional peaks were only observed at the side of the multilayer in an angle range from 0.3 to 0.8 degrees (Fig. 5).

In Fig. 5, the strong peak at zero angle represents transmitted XRF X-rays. Unfortunately, these strong peaks overlap the diffracted peaks. Thus, we assume that the transmitted peaks had a symmetrical shape. Therefore, the transmitted peaks were subtracted from the intensity profiles shown in Fig. 5 producing the XRF intensity curves shown in Fig. 6. Each curve showed a maximum at different angles, i.e., Fe Kα at 0.47 degrees, Cr Kα at 0.52 degrees, and Ti Kα at 0.61 degrees. The peak angle depended on the energy of X-ray fluorescence according to the Bragg diffraction equation. These results indicate that the WDS spectrometer shown in Fig. 4 performed well, although they were the first results obtained using a newly developed 2D dispersive device.

CONCLUSIONS
We investigated the fundamental characteristics of a 2D dispersive device by using a stainless steel plate and a Ti plate. As a result, we confirmed that the 2D dispersive device performed the role of a WDX spectrometer. The angle for the peak of Fe Kα was close to that of Cr Kα; the two were difficult to distinguish. This is because the energy resolution of the present system was poor. However, we expect to be able to solve this problem if the periodic-length of the multilayer could be shortened. In the future, we plan to replace the EDS detector with an X-ray CCD camera, and obtain X-ray elemental images.
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