Development of a full-field x-ray fluorescence microscope for elemental analysis

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Introduction

A full-field x-ray fluorescence microscope is a proper method for 3-dimensional observation by tomography and dynamic observation because it can obtain images relatively faster than a scanning microscope. We have been developing it with a Wolter mirror [1] for elemental analysis. 2-dimensional elemental analysis was performed not only by selective excitation [2] but also a CCD photon counting [3]. This year, the CCD camera was renewed with a deep depletion type one to increase the detectable efficiency. A double multilayer monochromator was replaced with a single multilayer one to improve the homogeneity of excitation x-rays. These improvements made it possible to perform an elemental analysis of a specimen, such as marble.

Optical system

The schematic of the experimental setup is shown in Fig.1. A monochromator with a W/B_4C (2d = 5.0 nm) multilayer mirror (manufactured by Osmic Inc.) was developed. The energy from 10.24 to 6.24 keV with the energy resolution of $\Delta E \approx 0.38$ keV (at 9.10 keV) could be available. X-ray fluorescence emitted from a sample was imaged by the Wolter mirror (magnification: 10, average grazing angle: 7 mrad) onto a CCD camera system (Roper Scientific, PI, SCX-TE/CCD-1300 EM/1). The optical axis of the Wolter mirror was set normal to the incident beam. The Wolter mirror chamber was transferred by He and the path of x-ray fluorescence and the monochromator was evacuated to ~10^{-3} Torr.

Experiment

As test samples, Fe, Ni, Cu and Ti wires were used. Each wire could be observed selectively by both the selective excitation method and the CCD photon counting method. The energy resolution of 210 eV (FWHM) could be obtained by the latter method. It was much better than the previous result of 350 eV [3].

A flat piece of marble was observed for an application. The selective excitation images are shown in Figs.2 (a) and (b). The excitation energy of Fig.2 (a) was 10.04 keV and that of Fig. 2 (b) was 6.84 keV which was under the K-edge of Fe. The difference between Figs. 2 (a) and (b) was the distribution of Fe. The peaks of Ca and Fe were observed clearly in energy profile of the photon counting and were extracted from 1000 x-ray fluorescence images in exposure time of 1 sec. Figures 2 (c) and (d) show elemental maps of Fe and Ca. These images show that both methods can be applicable to elemental analysis.

References

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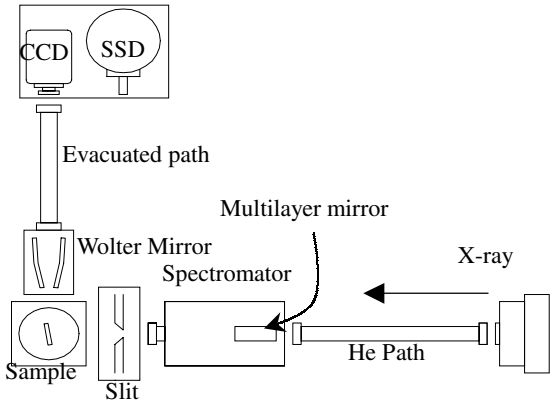
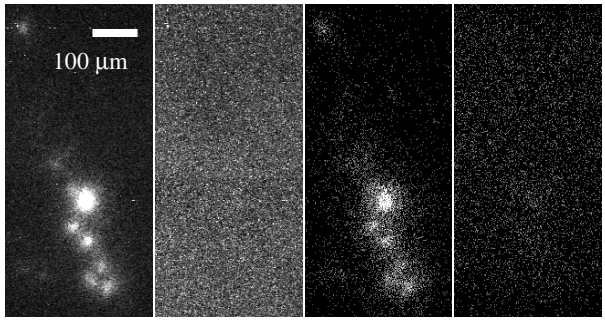


Fig.1 Experimental setup



(a) (b) (c) (d)

Fig.2 X-ray fluorescence images of marble. (a) and (b) are the selective excitation images at the excitation energy of 10 keV (a) and 6.8keV (b). (c) and (d) are the elemental maps of Fe and Ca calculated by the photon counting method. The excitation energy was 10 keV. The exposure times were 20min (a) and 10min (b) and 1 sec × 1000 images (c, d).

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