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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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[3] T. Ohigashi, N. Watanabe, H. Yokosuka, T. Aota, H. Takano, A. Takeuchi and S. Aoki. J. Synchrotron Rad. **9**, 128-131 (2002).

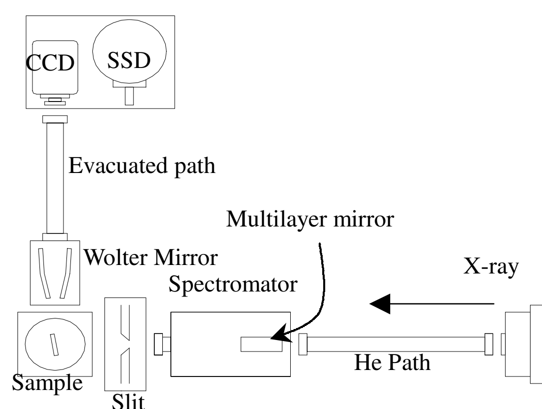

Fig.1 Experimental setup

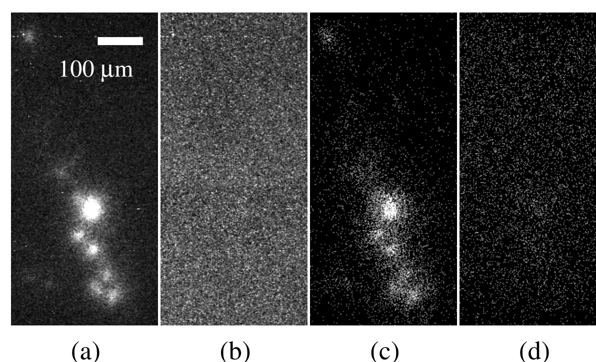

Fig.2 X-ray fluorescence images of marble. (a) and (b) are the selective excitation images at the excitation energy of 10 keV (a) and 6.8keV (b). (c) and (d) are the elemental maps of Fe and Ca calculated by the photon counting method. The excitation energy was 10 keV. The exposure times were 20min (a)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

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Observation of SOI wafers by X-ray topography

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Introduction

Silicon-on-insulator (SOI) wafers have been extensively studied as the key technology for the promising substrate material of low-power and high-speed devices. In these studies the quality of the SOI material such as the crystalline quality of the top Si layer, the morphology of its interfaces, and the buried oxide integrity has been characterized by several techniques. The crystalline quality of the top Si layer has generally been characterized by transmission electron microscopy (TEM) and chemical etching methods followed by optical observations, which are sensitive to dislocations and stacking faults. Atomic force microscopy (AFM) observations after chemical etching of overlayers show the morphology of the interfaces. The integrity of the buried oxide layer can be evaluated by electrical methods.

X-ray topography is one of the powerful tools to characterize crystalline quality, which provides us the distribution of dislocations and micro defects. But it has not been employed for the characterization of SOI wafers, because the SOI layer is much thinner than the extinction distance of x-rays. It causes drastic reduction of the contrast of the image of the defects. But it is possible to observe structural irregularity of the SOI layer in the framework of the kinematical diffraction theory. Therefore, we tried to observe the diffraction images of the SOI layers.

Experimental

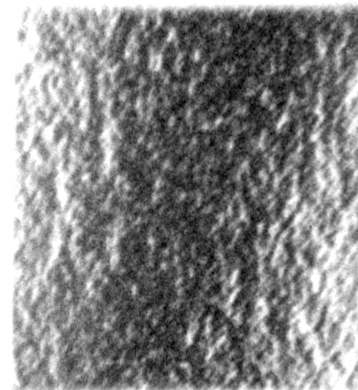
A bonded SOI wafer was prepared for this study. The thicknesses of the top Si and buried oxide (BOX) layers are about 2000 and 50 nm, respectively. The top Si layer was rotated 5 degree around [001] axis for the Si substrate in the bonding process. Two samples of 15x20-mm^2 rectangle were cut out from the wafer. The SOI layer of one of the samples was etched off to 240 nm by KOH solution.

X-ray topographs were taken by using 220 Bragg reflection in Laue geometry. The energy of the x-rays was selected to be 17.4 keV.

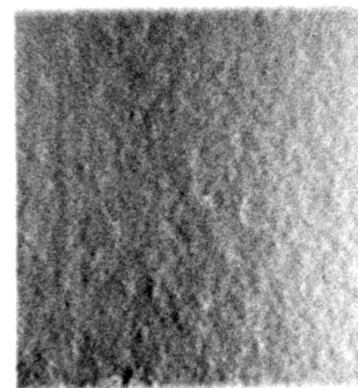
Results

X-ray topographs from the SOI layers are shown in Fig.1, in which a) and b) show the images from the SOI layers of thickness of 2000 and 240 nm, respectively. The images represent the 2x2-mm^2 area. We see the rugged images in both topographs, although the thickness of the SOI layers is much less than the extinction distance of 35000 nm. Therefore, this contrast can be understood in the kinematical diffraction theory.

As the results of further investigation, the lattice of the SOI layers undulated in more than ten arc seconds. Typical special interval is about dozens of micrometers.



(a)



(b)

Fig.1 X-ray topographs of SOI layers. Thicknesses of the SOI layers are 2000 nm and 240 nm for a) and b), respectively. The size of the images correspond to 2x2 mm^2.

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