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Multi-elemental observation by full-field imaging x-ray fluorescence microtomography

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Introduction

X-ray Fluorescence Computed Tomography (XFCT) is an application of an x-ray fluorescence microscope and a useful method to observe elemental distribution in a cross section of a sample. The advantage of the method is good for quantitative analysis of trace elements without any destructive process. We have been developing XFCT with a full-field imaging system. In the previous report, we observed Fe included in a synthesized diamond as impurities [1] but the quantity of Fe did not have so much reliability. To obtain quantitative elemental distributions of Fe and Ni in a diamond, it is necessary to calibrate the XFCT system. Then, a standard sample of Fe and Ni solution was evaluated by the XFCT.

Optical System

A monochromator with double W/B_4C multilayer mirrors ($\Delta E/E \approx 1.9\times10^{-2}$) was used for selective excitation [2]. X-ray fluorescence from a sample was imaged by a Wolter mirror (magnification: 10) onto a CCD camera (Roper Scientific, PI, LCX-TE/CCD-1300 EM/1). The optical axis of the Wolter mirror was set normal to the incident beam. Then transmission x-ray image of the sample was recorded by another CCD camera with a phosphor screen. The chamber of the Wolter mirror and the path of the incident beam were filled with He gas. The monochromator and the path of the imaging system were evacuated to ~ 10^{-3} Torr.

Experiment

Mixture solution of $FeCl_2$ (II) (1.14 mol/l) and $NiCl_2$ (0.286 mol/l) was used as a standard sample. The solution was flowed inside of a capillary tube (ϕ: 500 μm) by a solution pump to keep the uniform density.

To obtain elemental distributions of Fe and Ni, x-ray fluorescence images were obtained at the excitation energies of 7.47 (above Fe K-edge) and 8.40 keV (above Ni K-edge). The sample was rotated to obtain 100 projections over 360 degrees. The exposure times were 4 min. In order to extract the Ni signals from the images at 8.40 keV, the x-ray subtraction method [3] was used with taking into account the intensity of the incident beam. At the same time, the transmission x-ray images of 6.40 (Fe Kα line), 7.47 (Ni Kα line and above Fe K-edge) and 8.40 keV (above Ni K-edge) were obtained for the correction of the absorption effects of the incident beam and the x-ray fluorescence by the sample itself. The sample was rotated to obtain 100 projections over 180 degree. The exposure times were 0.7 ~ 1.2 sec.

The reconstruction was performed with the algebraic reconstruction technique taking into account the absorption effects. The reconstructed images of elemental distributions of Fe and Ni are shown in Fig. 1 and 2 respectively. Average reconstructed quantity of Fe was 2.76×10^{-13} [g/voxel (=2×2×2 μm^3)] and was 54.1 % of the calculated quantity (5.10×10^{-13} [g/voxel]). Average reconstructed quantity of Ni was 1.01×10^{-13} [g/voxel] and was 75.4 % of the calculated quantity (1.34×10^{-13} [g/voxel]). The differences of the reconstructed and the calculated quantities were considered to come from the matrix effect and the error of the absorption coefficients of the transmission images. For better quantitative observation, the matrix effect between Fe and Ni has to be considered.

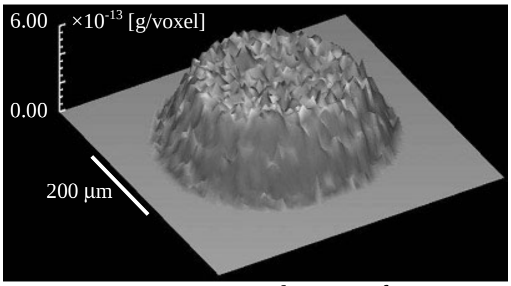

Fig.1 Reconstructed image of Fe

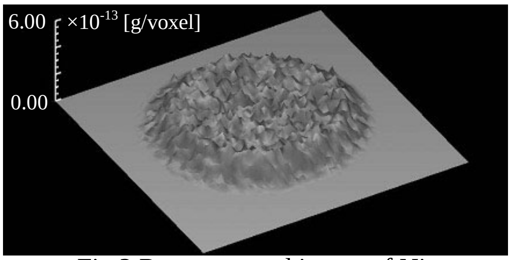

Fig.2 Reconstructed image of Ni

References

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