X-ray fluorescence microtomography with a Wolter mirror system


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Abstract

An X-ray fluorescence imaging microscope with a Wolter-type grazing-incidence mirror objective was constructed at beamline 39XU of SPring-8. Monochromatic X-rays in the energy range 6–10 keV were used for X-ray fluorescence excitation of the specimens. Recording X-ray fluorescence images of a test specimen (Cu, Ni and Fe wires) from 50 different angles of view, three-dimensional tomographic reconstructions were obtained. These wires could be reconstructed selectively by changing the energy of the excitation X-rays. A synthetic diamond could also be imaged three-dimensionally. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

A full-field X-ray fluorescence imaging microscope is a very convenient tool for real-time observation with elemental analysis. Only recently it has been successfully demonstrated by using a Wolter mirror [1]. Because a Wolter-type grazing-incidence mirror has small coma and no chromatic aberration, it can be used for simultaneous imaging of polychromatic X-ray fluorescence from multiple elements.

We have been developing an X-ray fluorescence microscope with a Wolter mirror objective at synchrotron radiation facilities, such as the Photon Factory [1,2] and SPring-8 [3,4]. Using this microscope, two-dimensional (2D) elemental maps of a test specimen (consisted of Cu, Co, Ni, Fe and Ti wires) and a rock specimen were obtained by the subtraction between images at different X-ray energies just above and below the absorption edges of the specific elements.

2D X-ray fluorescence computer tomography (CT) has been developed for medical use at the energy around 33 keV with no focussing element [5]. As a next step, we tried X-ray fluorescence tomography for three-dimensional (3D) elemental analysis by rotating a specimen. 3D X-ray fluorescence images of a test specimen which
consisted of metallic wires and a synthetic diamond could be obtained. These results are shown in this paper.

2. Optical system

An X-ray fluorescence microscope was constructed at beamline 39XU at SPring-8. This beamline had an in-vacuum type linear undulator \( \lambda_u = 32 \text{mm}, N = 140 \), a rotated double crystal monochromator \( \Delta E/E \sim 10^{-4} \) and Pt-coated plane mirror to eliminate higher order radiation [6]. The optical system of the microscope is shown in Fig. 1(a). Monochromatic X-rays in the energy range of 6–10 keV were used for excitation. The optical axis of the microscope was set normal to the excitation X-rays to reduce scattering X-rays from a specimen. The fluorescence image X-rays were enlarged by a Wolter mirror and focused onto a CCD camera (Hamamatsu, TI, TC-215, pixel size: 12 \( \mu \text{m} \times 12 \mu \text{m}, 1000 \times 1018 \) pixels). The specification of the Wolter mirror is the followings, magnification ratio: 10, object-image distance: 2200 mm, grazing angle: 7 mrad and Pt-coated surface. The spatial resolution of the mirror was found to be about 10 \( \mu \text{m} \) [1] and the depth of focus was about 1 mm. The excitation area of the specimen was restricted to be about 1 \( \times \) 1 mm\(^2\) by adjusting a slit width. The energy profile of the fluorescent X-rays could be measured by an SSD at the image plane. A transmission image of the specimen was also recorded by another CCD camera behind a specimen (Astro-med Ltd., CCD: EEV CCD 02-06, 22 \times 22 \mu\text{m}\(^2\)/pixel, 578 \times 385 pixels). The slide glasses for a visible microscope were used as an attenuator of the direct X-rays.

Fig. 1(b) shows the transmission X-ray image of an Fe wire of 100 \( \mu \text{m} \) diameter. The excitation X-rays were not so homogeneous as shown in Fig. 1(b). Fig. 1(c) shows the corresponding X-ray fluorescence image of the wire. The counting rate of X-ray fluorescence was 0.09 photons/s/pixel. The X-ray fluorescence image of the wire looks thinner than the actual thickness because of the absorption of the excitation X-rays. These results show that it is necessary to correct the inhomogeneous intensity distribution of the excitation X-rays and the absorption of the excitation and fluorescence X-rays for quantitative analysis. These two corrections seem to be possible at a time by X-ray fluorescence tomography in combination with X-ray transmission tomography. An absorption correction have been studied in 2D X-ray fluorescence tomography without a focusing element [5].

3. 3D reconstruction of X-ray fluorescence

In this experiment, the absorption corrections were not performed. The filtered back projection method for parallel projections with Shepp–Logan filter was used for reconstruction [7]. By rotating a specimen by 7.2° intervals, X-ray fluorescence images from 50 different angles of view were recorded. The CCD was operated in the binning mode of 4 \( \times \) 4 pixels. Then, the pixel size was
The median filter of $3 \times 3$ pixels and the averaging of $3 \times 3$ pixels were applied to the original images. The background data was subtracted.

A test specimen as shown in Fig. 2(a) was used for the evaluation of the system. The specimen consisted of an Fe wire of 100 $\mu$m diameter and Cu and Ni wires of 25 $\mu$m diameter.

Figs. 2(b)–(d) show the maximum intensity projections of the 3D reconstructed images of the test specimen. The energies of excitation X-rays were 9.000 keV (above Cu K absorption edge) in Fig. 2(b), 8.343 keV (between Cu and Ni K absorption edge) in Fig. 2(c) and 7.122 keV (between Ni and Fe K absorption edge) in Fig. 2(d). The Cu wire can be seen in Fig. 2(b), whereas it cannot be seen in Fig. 2(c) because of the lower energy of the excitation X-rays. Only the Fe wire could be seen in Fig. 2(d). These results show that this method is applicable to 3D elemental mapping of a specimen.

A synthetic diamond was also investigated by this method. Fig. 3(a) shows a transmission X-ray image using a conventional micro-focus X-ray tube with an Fe target. The diamond which was produced by the solvent method includes some metallic particles such as Fe, Co, Ni and so on [8]. Characterization of these particles is important for the synthesis of the diamonds. Figs. 3(b)–(d) show the maximum intensity projection of the reconstructed tomographic images of the synthetic diamond. The recording and reconstruction processes were the same as those of the test specimen. The exposure time of each 2D image was 8 min. The energy of excitation X-rays was 8.343 keV (10 eV above the Ni K edge) in Fig. 3(b), 7.719 keV (10 eV above the Co K edge) in Fig. 3(c) and 7.122 keV (10 eV above the Fe K edge) in Fig. 3(d). These images show that the proportion of the elementary composition of Ni, Co and Fe does not change very much between these impurities, which is the same result as our previous report [3].

4. Conclusion

Using the X-ray fluorescence microscope with the Wolter mirror, the 3D tomographic...
reconstructions of the X-ray fluorescence from the test specimen (Cu, Ni and Fe wires) and the impurities (Ni, Co, Fe) of the synthetic diamond at the excitation X-ray energy slightly above the K absorption edges of each elements could be obtained. The Cu, Ni, and Fe wires in the test specimen could be clearly distinguished. It is necessary to correct the inhomogenous intensity distribution of the excitation X-rays and the absorption of the excitation and fluorescence X-rays for quantitative analysis.

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References