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X-ray phase-contrast imaging with submicron resolution by using extremely asymmetric Bragg diffractions

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We have obtained x-ray phase-contrast images with high spatial resolution by using extremely asymmetric Si 111 Bragg diffractions near the critical angle of the total reflection. The x-ray image could be magnified to 294 times in both vertical and horizontal directions. By using this x-ray microscopy system, we have observed clear phase-contrast images of a 0.7-μm-wide gold-line pattern. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1337621]

X-ray phase-contrast imaging is a powerful tool for investigating weakly absorbing materials because this method is based on the refraction effect due to differences in material density.[1–4] The availability of intense and coherent synchrotron radiation (SR) x rays should make this method a useful tool for material science.[5–7] Real-time measurements especially will have a great impact on industrial applications. However, such real-time measurements have been reported for only biological samples[7] because of the insufficient spatial resolution of detectors. One solution for this problem is the use of microscopy. Although Lagomarsino *et al.* have developed a microscopy system with submicron resolution by using an x-ray waveguide,[8] the magnified image obtained was in only one dimension.

Recently, we have modified the x-ray microscopy system by using magnifier crystals with asymmetric Bragg diffractions,[9] taking advantage of the high-quality SR x rays in SPring-8, and have measured real-time formation and movement of a bubble in the battery cell with a spatial resolution of 5 μm and a time resolution of 1/30 s.[10] However, further improvement of the spatial resolution was needed for examining many devices. The image blur in our microscopy system was mainly caused by the penetration depth of x rays inside the magnifier crystal. To improve the spatial resolution, therefore, it is required to make the penetration depth shallow. The penetration depth can be decreased to several tens of nm by using extremely asymmetric diffraction with the glancing angle near the critical angle of the total reflection. This lower glancing angle also gives a larger magnification factor.

In this letter, we report x-ray phase-contrast images with submicron resolution. By using magnifier crystals with extremely asymmetric Si 111 Bragg diffractions, we obtained a magnification factor of 294 in both vertical and horizontal directions. We could also clearly observe a phase-contrast image of a gold pattern with a width of 0.7 μm on a Si_3N_4 substrate.

The measurements were performed at Hyogo beamline BL24XU at SPring-8.[7] The x rays emitted from the undulator were monochromatized by using a Si(111) double-crystal monochromator. The wavelength used was $\lambda = 0.0826$ nm. The beam size at the sample was $\sim 1 \times 1$ mm^2. To improve the spatial resolution, we used x-ray optics composed of four Si perfect crystals with asymmetric diffractions (Fig. 1). By using the crystals with asymmetric Bragg diffractions, the beam size of the transmitted x rays was magnified to the inverse of the following asymmetric factor $|b|$:[10]

$$b = \sin(\theta_B - \theta_a)/\sin(\theta_B + \theta_a), \qquad (1)$$

where θ_B is the Bragg angle and θ_a is the angle between the crystal surface and the diffraction plane. Equation (1) shows a large magnification factor can be obtained by decreasing the glancing angle of the incident beam. A small glancing angle also leads to a shallower penetration depth of the x rays, preventing the image blur. We used the extremely asymmetric Si 111 Bragg diffractions with a glancing angle of slightly over the critical angle of the total reflection 0.12° for the first and third magnifier crystals ($b_{1,3} = 0.0084$) in order to obtain a large magnification factor and a shallow penetration depth. We also used the asymmetric Si 111 diffractions with a glancing angle of 4.35° for the second and fourth crystals ($b_{2,4} = 0.4048$) to maintain sufficient x-ray intensity. As a result, the beam size of the transmitted x ray was magnified to 294 times in both the vertical and horizontal directions. Since the glancing angle for the first and third

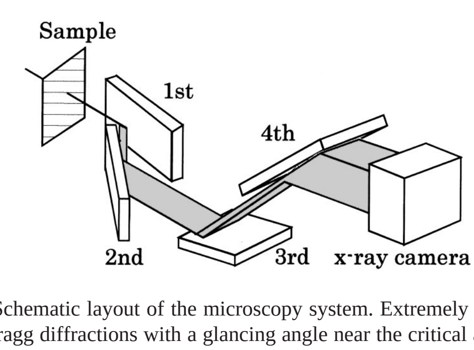

FIG. 1. Schematic layout of the microscopy system. Extremely asymmetric Si 111 Bragg diffractions with a glancing angle near the critical angle of the total reflection were used for the first and third crystals.

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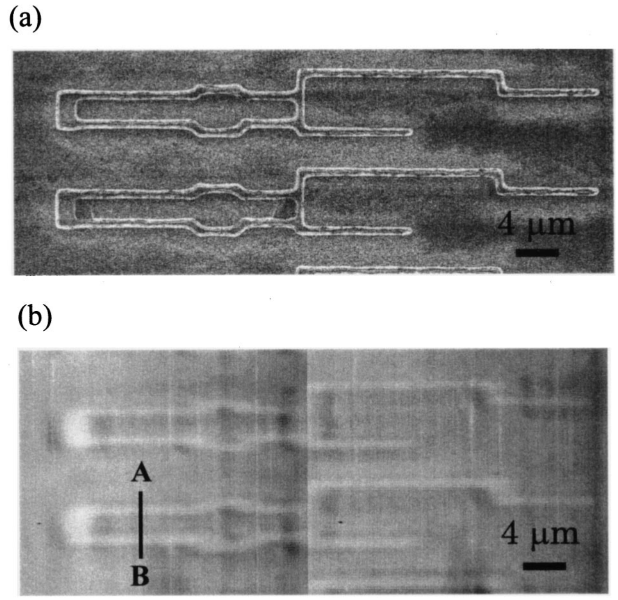

FIG. 2. (a) SEM and (b) x-ray images of a 0.8-μm-thick gold-line pattern on Si_3N_4; pattern width, 0.68 μm.

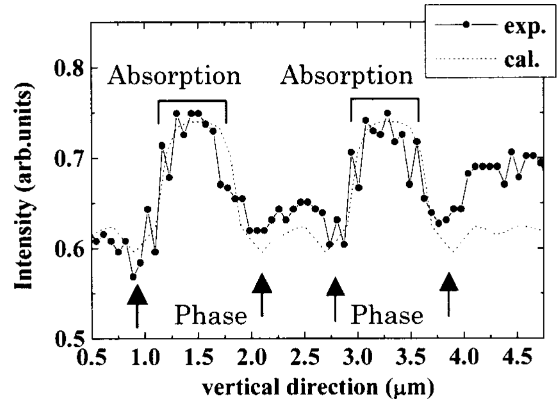

FIG. 3. Intensity profile along line $A-B$ in Fig. 2(b).

crystals was set near the critical angle, the penetration depth of x rays l_0 was about 30 nm. The blur of the image due to the penetration depth is expressed as $l_0\{\sin^2\theta_B/\sin(\theta_B-\theta_a)\}\times b$, taking into account the widening of a single incoming ray. By using the present parameters for the first and third crystals, the blur of the image was almost equal to the penetration depth. Although the penetration depth for the second and fourth crystals is not short ($\sim$4 μm), the blur of the image due to the penetration depth was about 50 nm because its influence acts on the magnified image. The magnified images were recorded with an x-ray saticon camera. The exposure time was below 1 s, which is short enough for real-time measurements.

In addition to improving the spatial resolution, the extremely asymmetric Bragg diffractions provide another advantage. When the x rays pass through an object, the phase contrast is observed as the modulation of the x-ray intensity due to the refraction effect. The acceptance angle of the extremely asymmetric Bragg diffraction is much larger than the refraction angle, allowing the magnifier crystals to receive a much larger portion of the modulation of the x-ray intensity without changing the crystal angle. Actually, the acceptance angle was estimated to be about 1×10^{-4} rad from the rocking-curve profile. Therefore, the images obtained with our optical system should be similar to those obtained in the in-line method, where the intensity modulation is obtained by setting a large sample to the detector distance.

To evaluate the effectiveness of this x-ray microscopy system, we prepared 0.8-μm-thick gold patterns deposited on a Si_3N_4 substrate.[10] Figure 2(a) shows the scanning electron microscopy (SEM) image of this sample. We found that a 0.68-μm-wide gold line was patterned, except for a 1.6-μm-wide vertical line on the left side. The x-ray image of this gold-line pattern is shown in Fig. 2(b). This x-ray image corresponds well to the (SEM) image. Therefore, we conclude that an image with submicron resolution was obtained by using the magnifier crystals with extremely asymmetric Bragg diffractions. We note that this x-ray imaging method can reveal the inside structure of the sample nondestructively. Figure 3 shows the intensity profile along the line $A-B$ in Fig. 2(b). The phase contrast appeared on both sides of the edge for the gold line (indicated with arrows in Fig. 3). This result is consistent with the feature of the image expected from the optical property of the magnifier crystals. Thus, the image obtained in our x-ray microscopy system corresponded to an image obtained using the in-line method, where it is difficult to obtain a magnified image by using parallel SR x rays. We simulated the intensity profile[3–5] shown in Fig. 3. The profile could be reproduced by taking the bent surface around the edge into account. This simulation showed that the phase-contrast profile of a small, thin sample is strongly affected by the sample shape.[4,5] This means that it is difficult to evaluate the phase-contrast profile quantitatively without having information about the sample shape.

Next, we discuss the spatial resolution of the image obtained in this x-ray microscopy system. In principle, the spatial resolution for the phase contrast is given by

$$\Delta=(\sigma/L_1)\times L_2, \quad (2)$$

where σ is the source size, L_1 is the source-to-object distance, and L_2 is the object-to-detector distance. In our optics, $\sigma_V=6.8\,\mu$m, $\sigma_H=384\,\mu$m, $L_1=66$ m, and $L_2=0.6$ m. Using Eq. (2), we calculated the spatial resolution for the vertical (horizontal) direction to be about 0.06 μm (3.4 μm). The resolution for the vertical direction was almost equal to the blur of the image due to the penetration depth ($\Delta_0\sim0.05\,\mu$m). This result is consistent with the vertical (horizontal) spatial resolution of 0.2 μm (1.5 μm) estimated from the intensity profile. Thus, the image blur was more apparent in the horizontal direction because the horizontal divergence from the x-ray source was much larger than the vertical one.[7] In order to improve the horizontal resolution, it will be useful to suppress the divergence by putting the crystal with asymmetric Bragg diffractions before the sample.

In summary, we have obtained x-ray phase-contrast images with submicron spatial resolution by using magnifier crystals with extremely asymmetric Bragg diffractions. Since the glancing angle was near the critical angle for the total reflection condition, a magnification factor of 294 was achieved. By using this x-ray microscopy system, we have obtained phase-contrast images of a 0.7-μm-wide gold-line pattern.

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