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Development of incident x-ray flux monitor for coherent x-ray diffraction microscopy

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Abstract. An incident x-ray flux monitor for coherent x-ray diffraction microscopy was developed. The intensities of x-rays passing through the sample were measured using an x-ray photodiode, with the simultaneous measurement of the x-ray diffraction intensities of the sample. As a result of the normalization of the x-ray diffraction intensities by the incident x-ray flux determined from the monitor, the fluctuation of the speckle intensities was successfully suppressed.

1. Introduction

Coherent x-ray diffraction microscopy[1] (CXDM) is a promising technique for observing a micrometer-sized sample with nanometer-scale resolution. In CXDM, it is important, for quantitative image reconstruction, to measure the incident x-ray flux. For example, to evaluate the electron-density distribution of a sample by CXDM, the absolute value of the structure factor must be derived using the incident x-ray flux[2]. Two or more items of two-dimensional (2D) diffraction data, e.g., diffraction patterns measured at different incident x-ray angles for a three-dimensional reconstruction[3] or different x-ray energies for elemental identification[4, 5], should be normalized by the incident x-ray flux. In typical CXDM measurements in the transmission scheme, an isolated sample is mounted on a Si_3N_4 membrane. It is necessary not only to measure the x-rays scattered from the sample including the Si_3N_4 membrane (i.e., sample data) but also to measure the x-rays scattered only from the Si_3N_4 membrane (i.e., background data). It takes a few hours to measure a 2D diffraction pattern with a good signal-to-noise ratio. Since the intensities of incident x-rays fluctuate during the period of a few hours, it is necessary to independently determine the incident x-ray flux for both the sample data and the background data to obtain an exact background subtraction. In order to determine the exact incident x-ray flux, the incident intensities must be simultaneously measured with the x-ray diffraction of the

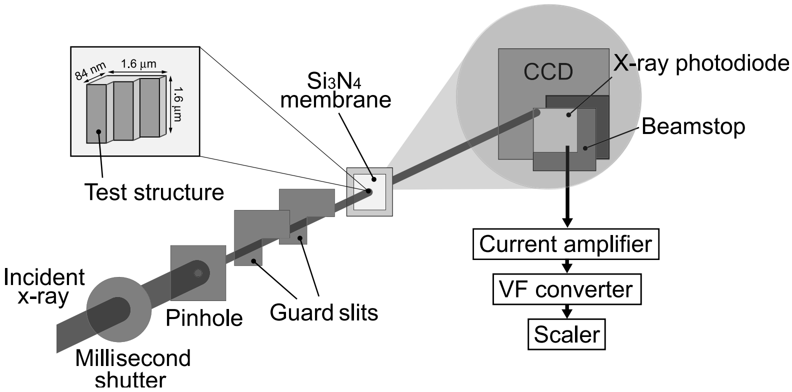

Figure 1. Schematic drawing of the coherent x-ray diffraction microscopy apparatus installed with the incident x-ray flux monitor.

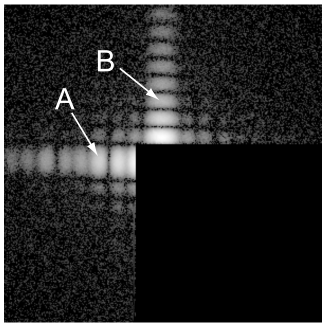

Figure 2. Coherent x-ray diffraction pattern of the test structure in 301×301 pixels.

sample. In this study, an incident x-ray flux monitor for CXDM measurements was developed and its performance was evaluated.

2. Experimental

A test structure was fabricated as a sample on a Si_3N_4 membrane by the focused ion beam deposition of tungsten. Figure 1 shows the schematic drawing of both the sample and the CXDM apparatus installed with the incident x-ray flux monitor. Incident x-rays of 5 keV were irradiated onto the sample through a 20 μm pinhole slit. Two L-shaped guard slits, which were set between the pinhole and the sample, suppressed parasitic scattering x-rays from the pinhole. Forward x-ray diffraction intensities were collected by a charge-couple device (CCD) detector with a pixel size of 20×20 μm^2 placed 2.181 m downstream of the sample. In order to control the x-ray exposure time, a millisecond shutter was set upstream of the pinhole. A direct beamstop was positioned before the CCD detector. An x-ray photodiode with 30-μm-thick Si (International Radiation Detector, INC., AXUVPSV) was mounted on the beamstop. The dead region of the phototiode is less than 50 μm from the beamstop edge. The photodiode detected the x-ray photons passing through the sample, while the CCD measured the x-ray diffraction intensities of the sample. Data signals from the photodiode were processed by a current amplifier, VF converter, and scaler. The performance of the x-ray flux monitor was evaluated. Fifteen independent items of sample data and background data were collected. The background was measured just after each sample measurement. The sample and background data, respectively, were obtained by accumulating 50 and 25 scattering data with 4 s exposure time. The measurement times including the read-out time of the CCD detector were 300 s and 150 s for the sample and the background, respectively. It took ∼7000 s to collect all the data.

3. Results and discussion

Figure 2 shows one of the 15 diffraction patterns of the sample. The lower right of the diffraction pattern is the ummeasurable region owing to the presence of the beamstop. The incident x-ray flux determined for each diffraction pattern is plotted in Fig. 3. The flux was normalized by the average value of the 15 flux data. The flux decreases as time passes. It is thought that the decrease is due to not only the fluctuation of the intensities of the optical source but also the drift of the position of the pinhole, the guard slits, and the sample. The repetition number dependence of the integral intensities of speckles indicated by arrows in Fig. 2 is shown in Fig. 3. The intensities were normalized by the average value of 15 intensity data. The magnitude of

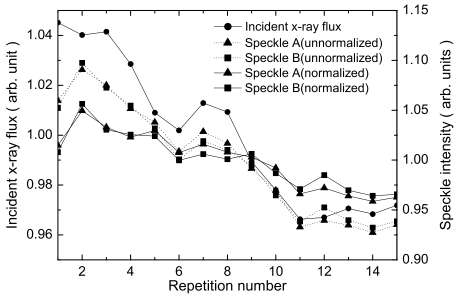

Figure 3. Repetition number dependence of the incident x-ray flux and intensities of speckles shown by arrows in Fig. 2. Each plot was normalized by the average value of 15 data. Speckle intensities shown by solid lines were normalized by the incident x-ray flux.

the noise of speckle intensities was estimated to be ∼0.3% from $(N_r^2 + N_D^2 + N_{ph}^2)^{1/2}$, where N_r is the read-out noise, N_D the dark charge noise, and N_{ph} the photon shot noise. The diffraction intensities decrease as the incident x-ray flux decreases. Next, all the sample and background data were normalized based on each incident x-ray flux. The diffraction data from the sample were derived by subtracting the normalized background data from the normalized sample data. The normalized integral intensity of each speckle is plotted in Fig. 3. Comparison with the plot of unnormalized intensities indicates that the fluctuation of speckle intensities is suppressed.

4. Conclusion

In this study, an incident x-ray flux monitor for CXDM was developed. The Si photodiode was mounted on the beamstop. The intensities of x-rays passing through the sample were measured by the photodiode simultaneously with the measurement of the x-ray diffraction intensities. The diffraction data were normalized by the incident x-ray flux. The fluctuation of the normalized diffraction intensities was successfully suppressed in comparison with that of the unnormalized intensities. By using this CXDM system, the quantitative analysis of the electron density distribution of alloys[6] or the evaluation of the elemental distribution of unknown samples will be realized.

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