High-Spatial-Resolution Phase Measurement by Micro-Interferometry
Using a Hard X-Ray Imaging Microscope

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With the aim towards high-spatial-resolution phase measurement, a novel hard X-ray micro-interferometer using an imaging microscope has been proposed and constructed at Hyogo-BL of SPring-8. It is a wavefront-division-type interferometer consisting of a twin zone plate arranged in the same plane. We have succeeded in producing good interference fringes with a visibility of as high as 60% at the photon energy of 9 keV. The fringe scanning method was applied to retrieve phase-shift distribution of a sample. The phase-shift distribution of a 75-µm-thick kapton film and polystyrene microparticles could be imaged clearly with a spatial resolution of 160 nm and the obtained phase shift agreed well with the expected value.

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Phase-contrast X-ray imaging techniques have been studied actively in the hard X-ray region since the advent of third-generation synchrotron radiation facilities. This is because the phase-shift cross section is almost a thousand times larger than the absorption cross section for light elements in the hard X-ray region.1,2 If we focus on X-ray microscopy, several phase-contrast microscopes such as Zernike’s phase-contrast microscope have been developed.2–4 Although their images show a high image contrast, it would be difficult to retrieve the phase information precisely from the images because the relationship between the image contrast and the phase shift cannot be written as a simple numerical function except in ideal cases. On the other hand, various methods of phase retrieval have been reported. Among them, interferometry offers the most direct method for phase retrieval. In the hard X-ray region, Bonse-Hart interferometers5–8 and shearing interferometers9,10 are used for phase retrieval. Furthermore, holography11 and Talbot interferometry12 are other approaches for phase retrieval. However, no interferometer using a magnifying optical system has ever been attempted yet. The spatial resolution is expected to be much improved as compared with the above interferometers by employing a magnifying optical system. In this paper, we propose a novel optical system for hard X-ray micro-interferometry using an imaging microscope and present some experimental results demonstrating the feasibility of this system for high-spatial-resolution phase measurement.

In order to form of a wavefront-division-type interferometer, both object and reference waves are essential. The proposed optical system is shown in Fig. 1. In order to preserve the spatial coherence, quasi-parallel illumination13 was used. By arranging two zone plates closely in the same plane perpendicular to the beam axis, which we call “twin zone plate”, the +1st order diffractive waves from the two zone plates can overlap each other and an interference region can be formed at an image plane. In order to prevent the −1st order diffractive waves from being mixed in the interference region, one zone plate (ZP-A) forming reference waves was designed to have a half-moon shape and only one-half of the other zone plate (ZP-B) functioning as a magnifying lens is illuminated.13 The detailed parameters of the twin zone plate are shown in Fig. 2. Since its outermost zone width is 100 nm, approximately 160 nm spatial resolution is ideally expected.15 By using a part of a zone plate as a reference wave producer, and not simply being a normal transmission diffraction grating,16 the interferometer becomes analogous to a Young’s interferometer, and therefore, much higher visibility can be expected. From Fig. 1, the
interference condition can be written as

\[ \Delta \ell = |\ell_1 - \ell_2| \approx \frac{h x_m}{L} = m \lambda (m = 0, \pm 1, \pm 2, \ldots), \]

(1)

where \( \ell_1 \) and \( \ell_2 \) are distances from two secondary point sources \( S_A \) and \( S_B \), respectively, which are foci formed by the two zone plates to an arbitrary point \( P \) on the image plane, \( L \) is the distance from the back focal plane of the objective to the image plane, \( h \) is the distance between the two secondary point sources, \( x_m \) is the distance of a point satisfying the interference condition with the order \( m \) measured from the origin of coordinate \( O \) on the image plane and \( \lambda \) is the X-ray wavelength. Since the two zone plates are arranged in the vertical direction, a horizontal fringe pattern will appear with regular intervals such as that obtained using a Young’s interferometer. The interval of the fringe pattern \( \Delta x \) can be written as \( \Delta x = L \lambda / h \) from eq. (1).

We constructed the hard X-ray micro-interferometer at Hyogo-BL (BL24XU)\(^{(17)} \) of SPring-8 as shown in Fig. 3. The photon energy of the fundamental harmonic peak of an undulator was tuned to 9 keV (\( \lambda = 0.138 \text{ nm} \)). The relevant photon beam parameters at 9 keV are as follows: \( \Sigma_x = 289 \mu \text{m}, \Sigma_y = 7.49 \mu \text{m}, \Sigma_{x'} = 13.0 \mu \text{rad}, \) and \( \Sigma_{y'} = 5.70 \mu \text{rad} \). where \( \Sigma_x (\Sigma_y) \) is the horizontal (vertical) effective photon beam size and \( \Sigma_{x'} (\Sigma_{y'}) \) is the horizontal (vertical) effective photon beam divergence. The X-ray beam was first horizontally collimated by a slit (TC3SLIT1) with 0.1 mm width placed at a distance of 56 m from the source point. The beam was next monochromatized using a horizontal-dispersion silicon double-crystal monochromator with 111 Bragg reflections. Since the horizontal angular acceptance of the slit \( \Delta \theta \) is 12.1 \mu rad, the angular spread \( \omega \) of the silicon 111 rocking curve at 9 keV is 29.0 \mu rad, and the Bragg angle \( \theta_B \) is 12.7 deg, the monochromaticity \( \Delta \lambda / \lambda \) of the output beam is estimated to be \( 1.40 \times 10^{-4} \), where \( \Delta \lambda / \lambda = \sqrt{\Delta \theta^2 + \omega^2 \cot \theta_B} \).

X-rays transmitting a sample (object waves) are magnified by ZP-B, while ZP-A produces reference waves (not transmitting a sample). The spatial coherence region in the vertical direction \( D_{coh} \), which is determined by a vertical source size \( 2 \Sigma_x \) and a source-to-zone plate distance \( R \) (\( = 67 \text{ m} \)), is about 200 \mu m. These values are related by the following equations, \( \theta_h \cdot \Sigma_x \leq \frac{1}{4 \pi} \) and \( D_{coh} = R \cdot 2 \theta_h \), where \( \theta_h \) is the acceptance half angle satisfying spatial coherence. From Figs. 1 and 2, the illuminated region \( D \) is 175 \mu m, therefore, this region is within the spatial coherence region \( D_{coh} \). A temporal coherence length determined by \( \lambda^2 / 2 \Delta \lambda \) is about 0.5 \mu m. Since the maximum optical path difference between the two waves \( \Delta \ell_{max} \) is 0.034 \mu m, it is much shorter than the temporal coherence length. Therefore, the object and the reference waves completely interfere with each other in the image plane. In order to observe magnified images of a sample and interference patterns, an X-ray zooming tube\(^{(19)} \) with a spatial resolution of about 500 nm was employed.

It is difficult to determine the structure inside an object from an interference pattern directly because interference fringes are contour lines appearing at \( 2\pi \) phase-shift intervals. It is also necessary to extract a phase shift of less than \( 2\pi \). To convert the interference pattern into the phase-shift distribution, subfringe analysis must be applied to calculate the phase shift precisely.

Generally, the intensity distribution of an interference pattern \( I(x, y) \) can be written as

\[ I(x, y) = a(x, y) + b(x, y) \cos[\Phi(x, y) + \Delta(x, y)], \]

(2)

where \( a \) and \( b \) are, respectively, the average fringe intensity and fringe contrast, \( \Phi \) is the phase shift caused by a sample, and \( \Delta \) is the phase shift originating from the optical system. In practical cases, \( a \) and \( b \) are not uniform because of the uneven sensitivity of the detector and the nonuniform amplitude of the wavefront. Therefore, it is impossible to obtain \( \Phi \) simply by operating an arccosine function to eq. (2). In this work, the fringe scanning method\(^{(20)} \) was applied to determine \( \Phi \) quantitatively. A 125-\mu m-thick kapton film was used as the variable phase plate and arranged in the path of reference waves as shown in Fig. 3. The tuning of a phase shift of the reference waves by \( 2\pi / M \) step (\( M \): integer) carried out by rotating the kapton film at appropriate angles around an axis normal to the X-ray beam. Rotation angles giving a \( 2\pi / M \) step phase shift were determined in advance by analyzing the fringe patterns obtained at many different rotation angles of the phase plate. The resultant interference patterns are written as

\[ I(x, y; k) = a(x, y) + b(x, y) \cos[\Phi(x, y) + \Delta(x, y) + 2\pi k / M], \]

(3)

When a function \( S \) is determined as

\[ S(x, y) = \sum_{k=1}^{M} I(x, y; k) \exp[-2\pi ik / M], \]

(4)

\[ \Phi(x, y) + \Delta(x, y) = \tan^{-1}[\text{Im}[S(x, y)] / \text{Re}[S(x, y)]], \]

(5)

where \text{Im}[S(x, y)] \text{ and } \text{Re}[S(x, y)] \text{ represent the imaginary and real parts of } S, \text{ respectively. } \Delta \text{ can be obtained by the same procedure without the sample (namely, } \Phi(x, y) = 0). \text{ Furthermore, the arctangent operation takes values ranging from } -\pi \text{ to } \pi. \text{ If } \Phi(x, y) + \Delta(x, y) \text{ exceeds } 2\pi, \text{ jumps of } 2\pi \text{ inevitably appear in a resultant image. Therefore, to obtain } \Phi \text{ precisely, an appropriate process correcting these jumps is necessary. If the spatial change of the phase shift is smooth compared to the pixel size of the detector, it is possible to

Fig. 3. Experimental setup of the hard X-ray micro-interferometer constructed at Hyogo-BL (BL24XU)\(^{(17)} \) of SPring-8. Undulator radiation is monochromatized by a silicon double-crystal monochromator. The photon energy used was 9 keV. The optical magnification was \( \times 20 \). The rotatable phase plate arranged in the path of reference waves was used for the fringe scanning method.
remove jumps by simply adding or subtracting $2\pi$ to one pixel when the phase differences between neighboring pixels are nearly $2\pi$. Even if the jumping positions are partly unclear because of detector noises, it is still possible to obtain $\Phi$ precisely by using the phase-unwrapping algorithm called the cut-line method.\(^{21}\)

We first used a 75-\(\mu\)m-thick kapton film as a pure phase sample of which the transmission is 96.3\%. By analyzing the obtained fringe pattern, the fringe interval $\Delta x$ was evaluated to be 10\(\mu\)m, which was in good agreement with the value expected by the previous equation. Furthermore, a visibility of as high as 60% was also achieved, where the visibility $V$ is determined to be $V = (I_{\text{max}} - I_{\text{min}}) / (I_{\text{max}} + I_{\text{min}})$. The sample images obtained are shown in Fig. 4. The absorption-contrast image, the interference pattern, and the phase-shift-distribution image obtained using the previously mentioned procedure are shown in Figs. 4(a), 4(b), and 4(c), respectively. In Fig. 4(b), the fringe dislocations are clearly observed. The 75-\(\mu\)m-thick kapton gives a phase shift of 12.9 rad by calculation assuming that $\delta = 3.77 \times 10^{-6}$,\(^{22}\) where $\delta$ is the real part of the refractive index decrement from unity. This value agreed well with the resultant distribution in Fig. 4(c).

Next, polystyrene microparticles with a diameter of 7\(\mu\)m were tried as another pure phase sample of which the transmission is 99.8\%. The sample images obtained are shown in Fig. 5. Even though the fringe dislocations could be hardly recognized in Fig. 5(b), the sample images clearly appeared by using the fringe scanning method, as shown in Fig. 5(c). The spatial resolution of this system was about 160 nm, which was estimated by an edge response of the phase-retrieved image of a copper $\#2000$ mesh. Therefore, the theoretical resolution limit was almost achieved. From these results, it can be concluded that we have succeeded in realizing the micro-interferometry with our new optical system in the hard X-ray region.

We are now improving the sensitivity to the phase shift. It will be realized by increasing the signal-to-noise ratio and expanding the interval of the fringe pattern. Furthermore, by combining our micro-interferometer with the tomographic technique, a three-dimensional refractive index distribution can be measured for biological specimens, micropolymers, and optoelectronic devices without any destruction.

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