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Electron-beam writing of large-area Fresnel zone plates

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High-resolution X-ray microscopy requires advanced fabrication technology for Fresnel zone plates (FZPs). As the resolution of an FZP depends on the width of the outermost zone, fine zone patterns for objective lenses have to be replicated. On the other hand, to achieve highly condensed X-ray beams by using FZPs for condenser lenses, large-field replication is required. A method of pattern replication of FZPs for X-ray microscopy is reported. Utilizing a 30 keV electron-beam writing tool and an FZP-generation computer program, FZP patterns for a condenser lens 1 mm in diameter with an outermost-zone width of 0.2 µm and for an objective lens 0.5 mm in diameter with an outermost-zone width of 0.1 µm were replicated.

Keywords: electron-beam direct writing; Fresnel zone plates; X-ray lenses.

1. Introduction

Fresnel zone plates (FZPs) are well suited for high-resolution X-ray microscopy in biology, as they have the advantage of an inherent contrast mechanism (Sayre, 1987; Kinjo *et al.*, 1990; Thieme *et al.*, 1993). Sensitive and highly resolved X-ray microscopy requires advanced fabrication technology for FZPs (Schmahl & Rudolph, 1987; Hilkenbach *et al.*, 1988; Kagoshima *et al.*, 1988; Kihara *et al.*, 1990; Aritome *et al.*, 1990; Tamamura *et al.*, 1995). X-ray microscopy requires condenser-lens FZPs to have a larger field than those for objective lenses, because the condenser lens is required to illuminate the sample with high photon flux. In addition, as the resolution of an FZP depends on the width of the outermost zone, fine zone patterns have to be produced. High-resolution X-ray microscopy requires that the condenser resolution be as high as the objective resolution. Therefore, to achieve high resolution, both the objective lens and the condenser lens require FZPs with fine patterns. In general, large-area FZPs with fine patterns have been produced by high-voltage electron-beam (EB) writing systems (Tamamura *et al.*, 1995). It has been reported that an FZP which has a diameter of 1.0 mm, a zone number of 1250 and an outermost-zone width of 0.1 µm has been patterned by a 50 kV EB direct-writing system. This writing system is very expensive.

This paper describes an FZP-pattern-writing method utilizing a very compact EB direct-writing tool which can operate at 30 kV acceleration voltage. Our goal is to have a large-field FZP as a condenser lens and a high-resolution FZP as an objective lens. The specifications of the FZPs are shown in Table 1.

Table 1
Specifications of an FZP for a condenser lens and an objective lens.

Use	Condenser lens	Objective lens
Diameter	1000 µm	500 µm
Outermost-zone width	0.2 µm	0.1 µm
Total zones	1250	1250
Focal length	50.0 mm	12.5 mm
Wavelength	4.0 nm	4.0 nm

Table 2
FZP representative-zone width sets.

(*a*) FZP as a condenser lens

Width set	Element	Line width (µm)	Increment (µm)
A	a_i	0.2–0.6	0.05
B	b_i	0.6–2.0	0.1
C	c_i	2.0–10.0	0.2
D	d_i	>10.0	0.4

(*b*) FZP as an objective lens

Width set	Element	Line width (µm)	Increment (µm)
A	a_i	0.1–0.3	0.025
B	b_i	0.3–1.0	0.05
C	c_i	1.0–5.0	0.1
D	d_i	>5.0	0.2

2. Fresnel zone pattern replication

Elionix model ELS-3700, an EB direct-writing tool of a vector-scan type, was employed as an exposure tool for FZP pattern replication. When replicating high-resolution and large-field FZPs, the affect of the proximity effect increases. The proximity effect in the outer zone with fine patterns has to be corrected.

The proximity-effect correction was carried out by our original computer program. The program for FZP pattern generation was coded on the CAD system of an ELS-3700 exposure tool. In this program, circular patterns are mapped onto orthogonal Cartesian coordinates, and the centre of the FZP and the point which defines the radius of the circle are located on lattice points of 20000 × 20000 dots in the exposure area. Each circle of each zone can be exposed on less than 60000 dots around the circle. Exposure sizes of 1 × 1 mm and 0.5 × 0.5 mm were set for writing an FZP as a condenser lens which has a diameter of 1 mm and outermost-zone width of 0.2 µm, and an FZP as an objective lens which has a diameter of 0.5 mm and outermost-zone width of 0.1 µm, respectively. Thus, the position accuracies of an FZP as a condenser lens and an FZP as an objective lens are 0.05 µm and 0.025 µm, respectively.

For the representative-zone width, the number of dots Q_i around the circles was optimized by test exposure. Furthermore, the relationship between the representative-zone width and exposure time was optimized by test exposure of each set of representative-zone widths. The exposure time of writing each outer circle with radius R_i of representative-zone width is assumed to be an element of sets A, B, C and D. The sets of the representative-zone width for an FZP as a condenser lens and as an objective lens are shown in Table 2. The relationships between the representative-zone width and exposure time for a condenser lens and for an objective lens are shown in Figs. 1(*a*) and 1(*b*), respectively.

For a circular constituent of a zone which has a radius R_k, the most closely representative-zone width was found from the elements $\{a_i\}$ in set A, $\{b_i\}$ in set B, $\{c_i\}$ in set C and $\{d_i\}$ in set D, and the number of dots Q_k around the circle was calculated by the equation $Qk = (R_k/R_i)Q_i$, where R_i is the radius of the outermost circle in the representative zone.

3. Pattern replication of Fresnel zone patterns

To replicate a fine-pattern width of 0.1 µm, ZEP-520 positive tone resist was employed. 0.3 µm-thick resist was spin-coated on the FZP substrate. Pre-baking was carried out on a hot plate for 240 s at a temperature of 453 K. After the FZP pattern had been generated on the CAD system with our computer program, EB writing exposure was carried out with an electron acceleration voltage of 30 kV. Development was carried out in *o*-xylene for 5 min at a temperature of 295 K and rinsing was carried out in isopropyl alcohol for 30 s at room temperature. After Au was coated on the resist pattern of the objective-lens FZP by utilizing a Hitachi E-1030 ion sputter system, the resist pattern of the FZP was observed by utilizing a Hitachi S-900 scanning electron microscope (SEM).

For EB writing of an FZP for an objective lens which has a diameter of 0.5 mm and an outermost-zone width of 0.1 µm, an EB current of 10 pA and an objective aperture of 50 µm for EB writing were employed. SEM photographs of the inner-zone patterns and the outermost-zone patterns are shown in Figs. 2(*a*) and 2(*b*), respectively. Though the EB writing time is found to be approximately 30 min for an outermost zone and approximately 2 h for an entire plate, outermost zones and inner zones were patterned within CD control of 25% and 10%, respectively. Therefore, it seems that there is no problem with drifting during the EB writing time which would lead to zone distortion and misregistration. Therefore, the original program is effective for the FZP pattern replication.

For EB writing of a larger-area FZP for a condenser lens which has a diameter of 1 mm and an outermost-zone width of 0.2 µm, EB current is very important due to the effect of electromagnetic lens aberration. The EB current was optimized to decrease the radius of the electron-beam spot without affecting the electron stability. An exposure beam current of 5 pA was employed. SEM photographs of the inner-zone pattern and the outermost pattern are shown in Figs. 2(*c*) and 2(*d*), respectively. Though the EB writing time is found to be approximately 4 h for an outermost zone and approximately 16 h for an entire plate, outermost zones and inner zones were patterned within CD control of 25% and 10%, respectively. Thus, it seems that there is no problem with drifting during the EB writing time which would lead to zone distortion and misregistration. Therefore, our original program combined with a decreased EB current to avoid disturbing the electron-beam stability is effective for the replication of the FZP pattern.

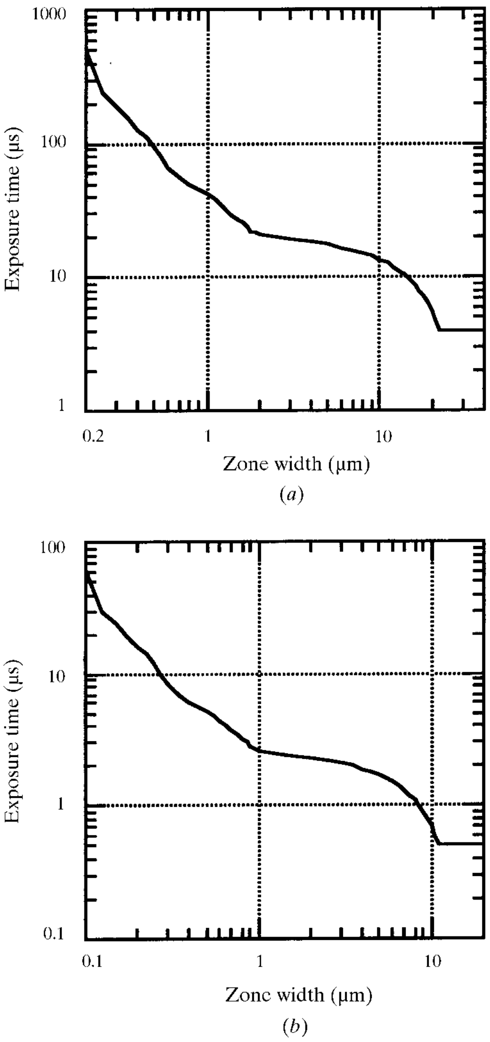

Figure 1
Relationship between the representative-zone width and the exposure time (*a*) for an FZP as a condenser lens, and (*b*) for an FZP as an objective lens.

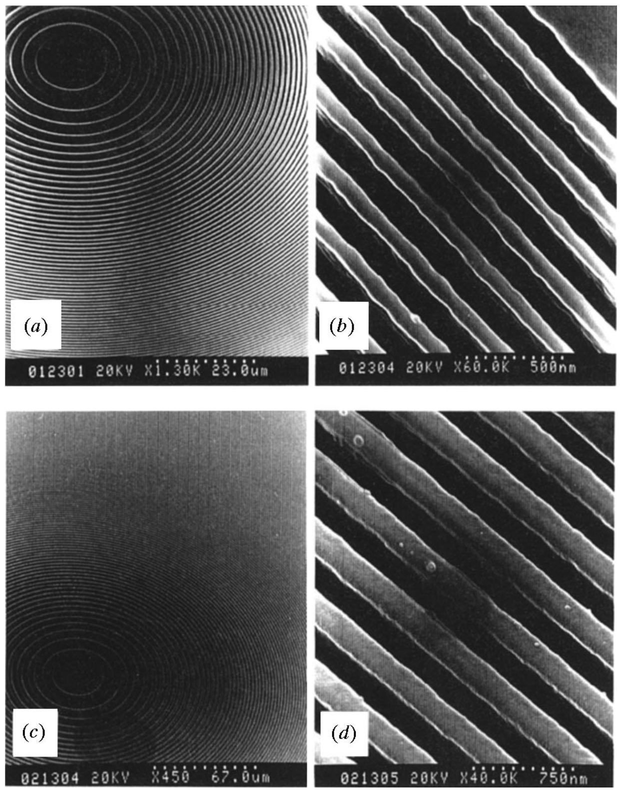

Figure 2
SEM photographs of an FZP as an objective lens with a diameter of 0.5 mm and outer-zone width of 0.1 µm [(*a*) inner-zone patterns and (*b*) outermost-zone patterns], and those of an FZP as a condenser lens with a diameter of 1 mm and outer-zone width of 0.2 µm [(*c*) inner-zone patterns and (*d*) outermost-zone patterns].

4. Conclusions

We have confirmed the effectiveness of 30 kV EB writing of FZP patterns for an objective lens which has a diameter of 0.5 mm and outermost-zone width of 0.1 μm, and FZP patterns for a condenser lens which has a diameter of 1.0 mm and outermost-zone width of 0.2 μm. In addition, we have shown that our original computer program, combined with a decreased EB current to avoid disturbing the electron-beam stability, is effective for replicating a large-field FZP with an outermost zone with fine patterns by utilizing a very compact EB writing tool.

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