Stacked Fresnel Zone Plates for High Energy X-rays

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Abstract. A stacking technique was developed in order to increase focusing efficiency of Fresnel zone plates (FZP) at high energies. Two identical Si chips each of which containing 9 FZPs were used for stacking. Alignment of the chips was achieved by on-line observation of the *moiré* pattern. The formation of *moiré* patterns was studied theoretically and experimentally at different experimental conditions. To provide the desired stability Si-chips were bonded together with slow solidification speed epoxy glue. A technique of angular alignment in order to compensate a linear displacement in the process of gluing was proposed. Two sets of stacked FZPs were experimentally tested to focus 15 and 50 keV x rays. The gain in the efficiency by factor 2.5 was demonstrated at 15 keV. The focal spot of 1.8 μm vertically and 14 μm horizontally with 35% efficiency was measured at 50 keV. Forecast for the stacking of nanofocusing FZPs was discussed.

Keywords: Fresnel zone plates, focusing, stacking, high energy.
PACS: 41.50.+h, 07.85.Tt, 07.85.Qe, 42.79.Ci

INTRODUCTION

Over the last years, there is a strong demand for hard x-ray focusing above 20 keV [1-2]. Efficient focusing of hard x rays using diffractive optics such as Fresnel zone plates is limited by the ability to manufacture diffracting structures with a small outermost zone width and a large thicknesses imposed by the weak interaction of x rays with matter. With increasing photon energy, the required thickness of phase shifting material increases. In order to achieve a small period and high aspect ratios one can attempt a multiple FZP setup [3-4]. We propose to use Si-based FZPs made by Micro Electro Mechanical Systems (MEMS) technology bringing together Si-based microelectronics with micromachining technology [5]. In addition to a high accuracy and reproducibility, the MEMS technology makes possible the realization of number of FZPs on-a-chip providing a reasonable energy tunability of microscopy setups at multipurpose beamlines. In order for stacked zone plates to behave as one, they have to be positioned laterally and longitudinally within reasonable proximity. Alignment of the two zone plates was done by observing the *moiré* pattern from the two zone plates in real time and maximizing the fringe spacing by moving one of the zone plate. We have developed a technique of an angular alignment to compensate a linear displacement in the process of gluing. Two types of Si chips were used for stacking to focus 15 and 50 keV X-rays. The stacked sets were experimentally tested and the measured focal spot and efficiency are in excellent agreement with calculations that underlines the good quality of the stacked system. A forecast for the stacking of nanofocusing Fresnel zone plates was discussed.

FZP MANUFACTURING

Silicon based FZPs were manufactured using microelectronics and MEMS microfabrication technologies, including the following main processes: electron beam- and photolithography, deep plasma etching and anisotropic wet etching of silicon. Two types of Si chips have been fabricated (see Table 1). The first type of Si-chips contains FZP structures which are 9 μm thick that corresponds to optimum efficiency at the energy range around 7.5 KeV. The structures on the Si-chip of second type are 30 μm deep and they are optimized for use at energies around 25 keV. The aspect ratio is more than 50, demonstrating the state-of the-art of the modern Si microfabrication technology. Each chip is 18cm wide and 25 cm height, and all of them are identical.

TABLE 1. Si chip parameters.

Chip number	Membrane thickness*	Zone height	Energy range**, keV	Maximum efficiency
Chip 1	12 μm	9 μm	6 – 12	30% at 7.5 keV
Chip 2	90 μm	30 μm	17 - 40	32% at 23 keV

*Initial thickness of Si membranes and remaining thickness in the FZP area after etching are 3 μm (chip1) and 60 μm (chip 2).
Where energy range is energy range with focusing efficiency greater than **20%.

Each chip contains 9 different FZP elements (5-cicular and 4-linear) designed as medium and long focal distance optical elements. The main parameters of the FZPs are listed in Table 2. Scanning electron microscope image of FZPs 22 and 32 are shown in Fig.1.

TABLE 2. FZP parameters.

FZP number	Focal length F (cm) at 8 keV	FZP aperture A (μm) and length L (μm) for linear	Outermost zone width Δr_n (μm)	Number of zones, N
33 circular	50	A = 194	0.4	122
11 linear	100	A = 387; L = 1000	0.4	242
12 circular	100	A = 387	0.4	242
21 circular	150	A = 582	0.4	364
31 linear	150	A = 582; L = 1000	0.4	364
13 linear	200	A = 775; L = 1000	0.4	484
23 circular	200	A = 775	0.4	484
22 linear	240	A = 930; L = 1000	0.4	582
32 circular	240	A = 930	0.4	582

EXPERIMENT AND DISCUSSION

Stacking was performed at the Microoptics test bench (MOTB) located in the 2nd experimental hutch (55m from the source) of the ESRF BM5 beamline [6]. White radiation from the bending magnet (source size vertically 80 μm and 250 μm horizontally) was collimated by primary and secondary slits and passed through a double crystal monochromator provided an X-ray beam of selected fundamental energy. The FZP chips were mounted on the stages with all necessary angular and linear movements. Two chips with zone plates were joined together in a way that the front side of the first chip is faced to the backside of another one. Therefore, corresponding zone plates are separated from each other by approximately 500 micrometers (substrate thickness). Alignment of the two zone plates is achieved by a straightforward X-ray phase contrast imaging technique by looking at the high resolution CCD camera image of the beam. When you look through one FZP at another, you see a *moiré* pattern, always appears when two repetitive patterns overlap. A slight motion of one of the objects creates large-scale changes in the moiré pattern. Increasing the spacing in the *moiré* pattern indicates a correct trend in the alignment and when fringe

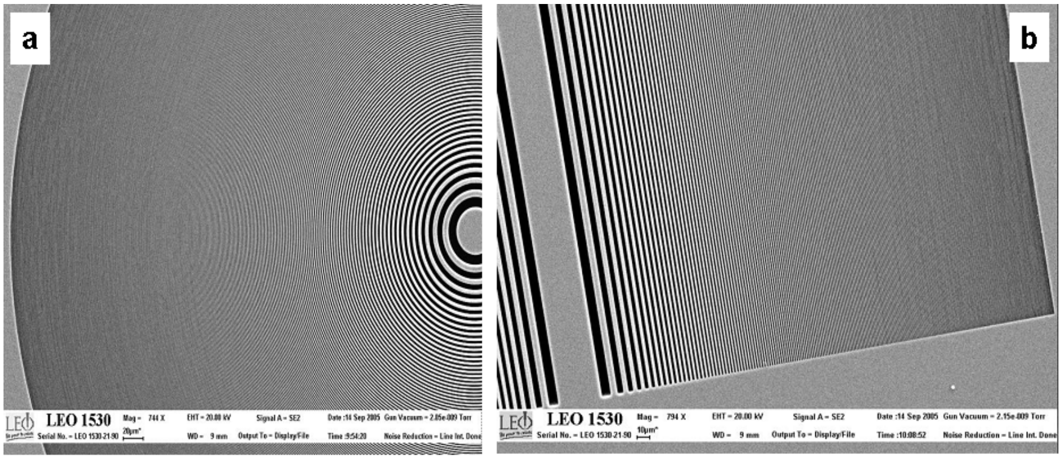

FIGURE 1. SEM images of circular FZP 32 (a) and linear FZP 22 (b). Aperture is almost 1mm.

spacing is infinite the stacking alignment is perfect. The precise and smooth vertical movement of the MOTB table allowed checking alignment of all FZP structures on the chip including linear ones. Alignment was done in the energy range 10-18 keV depending on FZP.

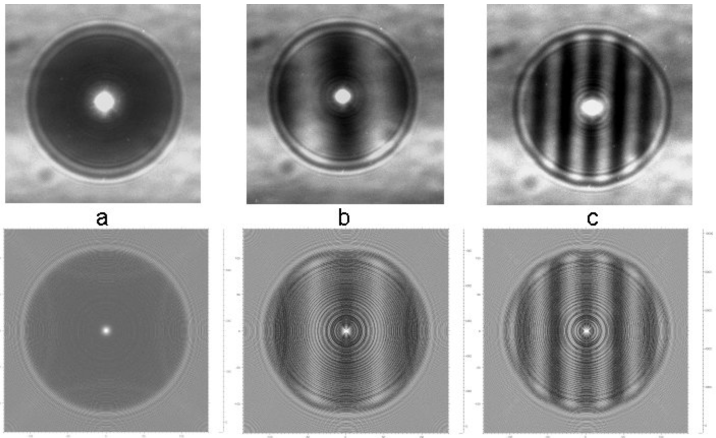

FIGURE 2. Experimental (upper raw) and theoretical (lower raw) *moiré* pattern recorded at 12 keV for FZP 33 at 76 cm; a-aligned, b-lateral displacement 1micron , c lateral displacement 2 microns.

In order for two zone plates to behave as one, they have to be positioned laterally and longitudinally within reasonable proximity. For lateral alignment Δx, a typical requirement is $\Delta x < 1/3\ \Delta r_n$, where Δr_n is the outermost zone width. We have studied theoretically and experimentally the behavior of the *moiré* pattern recorded at different distances from the stacked FZPs. The best pattern visibility and contrast was observed at distance corresponding FZP 1st order focusing. Figure 2 depicts *moiré* patterns recorded during horizontal alignment with different displacements between zone plates. The fringe spacing is $c = r_1^2/\Delta x$, where r_1 is the radius of the first zone. When zone plates are aligned with accuracy Δr_n, the spacing between fringes is equal lens aperture and for final lateral alignment intensity monitoring in the focus is required. A compact micro-mechanical motion system (piezo-based Y-Z stage) was used to execute such precise alignment with 30 nm step size (Fig. 3a). For two zone plates to behave effectively as one, the second element must be placed within the focal depth of the first one. Longitudinal alignment d must satisfy the condition $d < \Delta r_n^2/\lambda$. The influence of the separation between FZPs on focusing efficiency in a stacking set was studied. Fig. 3b shows intensity in the focal spot with the splitting between two FZPs. It is clearly seen from this graph that splitting up to 1.5 mm is acceptable that is in good correspondence with requirements to longitudinal alignment (FZP depth of focus for 12 keV is 1.56 mm).

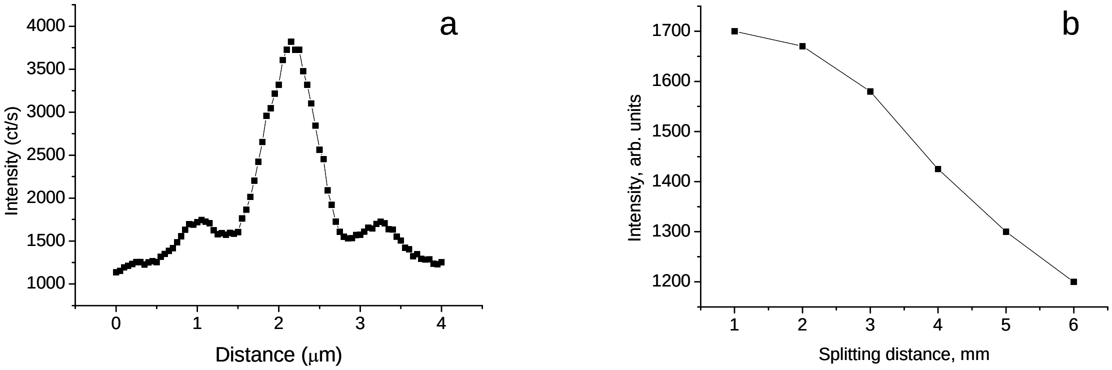

FIGURE 3. Intensity in the focus measured: (a) during FZPs lateral displacement with 30 nm steps and (b) with separation between two FZPs 33 at 12 keV.

To provide the desired stability and to use focusing elements at different beamlines, contrary to double zone plate holder approach [2], we have bonded FZP chips. Slow solidification speed epoxy glue was used. We stacked two sets of FZPs: one is optimal for 15 keV and second for 50 keV X-rays. It was found that long-run gluing process introduces lateral misalignment in the order of 1 or 2 μm. To compensate this displacement we have proposed the technique of angular alignment. Simple estimation shows that compensation tilt angle must be less then the ratio $\Delta r_n/h$. For example for 50 keV stack $\Delta r_n/h = 0.013$ and lateral displacement up to 6 μm for 500 μm chip separation can be easily corrected with tilt. It was shown that lateral displacement in the order of 1 μm can be compensated by sub-degree tilting, that is easily achievable with standard beamline equipment. Situation is more favorable for 15 keV stacking set, where displacement in the order of 25 μm can be corrected. It should be emphasized that with angular alignment technique all 9 FZPs in the stack can be corrected and successfully used.

The performance of the stacked FZPs for 15 keV has been tested at MOTB at the BM05. High resolution X-ray CCD camera (pixel size 0.65 μm) was used to evaluate the size of the focal spot and FZP efficiency. FZP 33with focal distance of 95 cm at 15 keV was chosen. Efficiency about 32% was measured. The gain in the efficiency compared with the single FZP is in the order of 2.5. The measured focal spot in the vertical direction was around 2.5 μm that is a good agreement with the demagnification factor.

Optical properties of the second set of stacked FZPs were evaluated at the ID15 beamline at the ESRF. 50 keV X-rays were selected by using a horizontally deflecting Si crystal monochromator in Laue geometry. The set was placed at the distance 60 meters from the source which is 30 μm vertically and 250 μm horizontally. A focal distance for FZP 33 was 3.3 m. The FZP alignment was done using a CCD camera with pixel size 1.5 μm. But the lateral size of the microbeam was measured by scanning a 35μm thick Au knife-edge across the beam vertically and horizontally, recording the intensity by PIN diode detector. Focal spot of 1.8 μm vertically and 14 μm horizontally was measured. The measured focal spot is in excellent agreement with calculations and underlines the good quality of the stacked system. Gain in the focal spot compared with the flat beam in slits the same size was measured as 450.

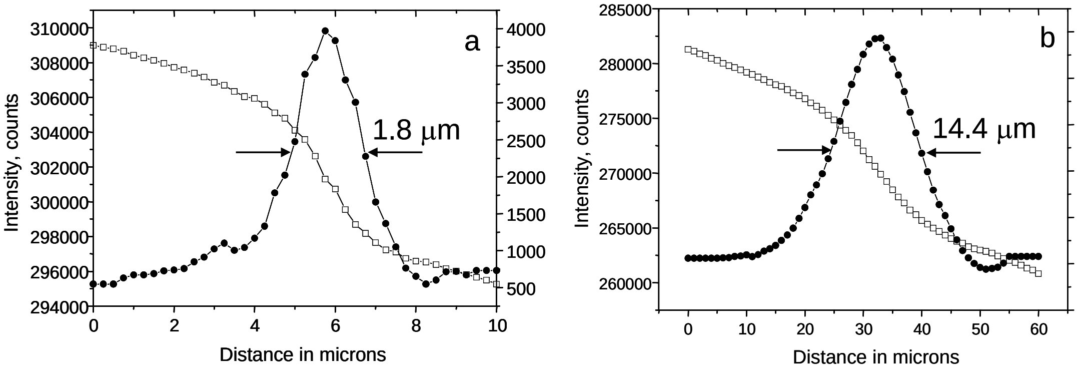

FIGURE 4. Vertical (a) and horizontal (b) knife scans

CONCLUSION

Si-based FZP structures manufactured by means of MEMS technology have been used for stacking. *Moiré* interference pattern was used for the on-line alignment. The formation of the *moiré* pattern was studied experimentally and theoretically during lateral and longitudinal alignments. Intensity monitoring was proposed for fine lateral adjustment within the first *moiré* fringe. Two sets of Si chips have been stacked and glued for focusing 16 and 50 keV X-rays. Technique of angular alignment to compensate a linear displacement was proposed. Intensity gain in the order of 2.5 was achieved compared to single FZP. The measured focal spot and efficiency are in excellent agreement with calculations and underlines the good quality of the stacked system. Our estimations show that stacking of three and more FZPs is straightforward with efficiency more then 20% above 100 keV. The proposed technique can be extended to nanofocusing FZP. To achieve the spatial resolution of 50 nm in the energy range 6-15 keV, a longitudinal proximity in the order of 20 μm is required. Lateral alignment should be done within 15 nm, but with the proposed angular alignment technique lateral displacement in the order of 100 nm in the process of solidification can be compensated. For example the tilt angle of ~0.14^{o} is needed to compensate 50 nm lateral misalignments at 12 keV. From these simple estimations it is clear that stacking of nanofocusing FZPs is feasible

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