High energy X-ray nanofocusing by silicon planar lenses

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Abstract. Optimizing the lens design and improving the technological process, we manufactured X-ray planar compound refractive lenses with vertical sidewalls up to 70 microns deep. The lens surface roughness in the order of 20 nm was attained. The minimal thickness of the material between two individual lenses of 2 μm was realized. The optical tests of the new planar lenses were performed at the ESRF BM05 and ID15 beamlines. The technological breakthrough allows reaching the nanometer focusing. The resolution below 200 nm was measured in the energy region of 15-80 keV. The best resolution of 150 nm was demonstrated at 50 keV X-rays.

1. Introduction

In the recent years there is a great demand for nanometer X-ray beams at energies higher then 20 keV. A number of new applications such as surface and interface scattering, high pressure magnetic scattering, and depth strain analysis using powder micro diffraction were developed. It turned out that X-ray planar refractive lenses made by microfabrication technology are very promising candidates for nanoprobe applications [1-6]. Si microfabrication technologies are well developed and fabrication of lenses with very small parabola radius is straightforward. Microelectronics technology allows to make lens-chips consisting of different integrated lens systems (ILS) with dozens of parallel compound refractive lenses [5-6]. The ILS was proposed in order to simplify the use of refractive lenses in the energy tunable experiments. The design feature of the ILS is that it consists of arrays of refractive lenses aligned in parallel. All lenses in ILS have fixed focal distance that was achieved by varying the numbers of individual lenses in each compound refractive lens. Each lens array is optimized for certain energy but number of arrays taken together allows to cover considerable energy range. To choose the desirable working energy, one can switch from one lens array to another by parallel displacement of ILS in vertical direction.

2. Lens design and manufacturing

The lenses were manufactured by microfabrication process consisting of two main steps, i.e. pattern generation in the hard mask on the silicon surface and pattern transfer into the silicon. The pattern generation was performed by photolithography. A SiO_2 layer (1 μm thick) was used as a highly selective hard mask for pattern transfer into the silicon substrate. The etch process was based on the time multiplexed etching technique, the Bosch process, where the sidewall passivation layer is

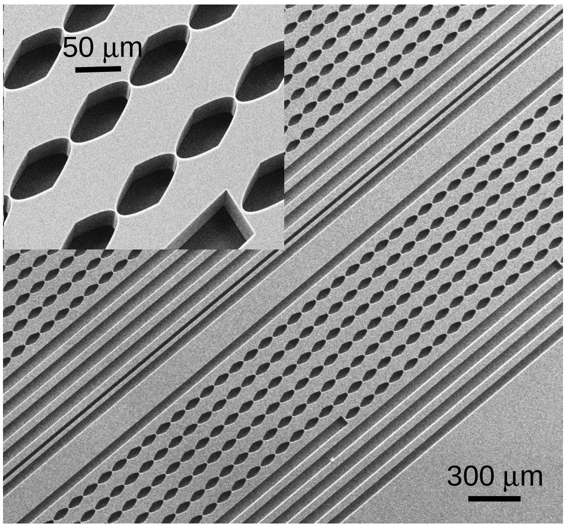

Figure 1. SEM image of ILS and individual lenses (insert)

deposited in a separate step from silicon etching. C_4F_8 and SF_6 were used as the passivation and etch gases, respectively. The mask undercut was regulated by polymer deposition and for some lenses corrections for undercut were done. The scalloping amplitude and pitch were controlled by duration of etching and passivation steps. To reduce the scalloping amplitude and mask edge originated roughness some chips after deep etching were thermally oxidized and then grown SiO_2 was removed in etching solution.

The current ILS design comprises 10 lens arrays optimized for energy range from 10 to 55 keV with the step of 5 keV. Each of these lenses has focal distance of 10cm. The length of the individual lens was 100 µm and their aperture was 50 µm. The web thickness of individual lenses on the optical axis is 2µm. The lens surface roughness was attained in the order of 20 nm. The structures are 50 µm deep. Parameters of the nanolenses are summarized in Table 1. In parallel with ILS, the test structures like bi-lenses are implemented on the Si chip. They can be used for the diagnostics of the beam and verification the focusing properties of nanolenses. SEM image of nanolenses is shown in Fig. 1.

Table 1. Lens parameters

	Energy, keV	Focal distance, mm	Focal depth, mm	Number of lenses	Total lens length, µm	Effect. aperture, µm
1	10	106.6	5.15	12	620	23.38
2	15	103.5	2.33	28	1436	27.58
3	20	99.3	1.3	52	2660	30.63
4	25	101.0	0.89	80	4088	33.71
5	30	100.5	0.65	116	5924	35.69
6	35	99.2	0.52	160	8168	3678
7	40	99.7	0.43	208	10616	37.73
8	45	99.5	0.38	264	13472	38.06
9	50	100.1	0.34	324	16532	38.15
10	55	100.1	0.32	392	20000	37.84

3. Experiment and discussion

Focusing properties of nanolenses were studied at the ESRF BM5 (Micro optics test bench end station) and ID15 beamlines. The complete energy tunability test and the influence of fabrication drawbacks or inaccuracies on focusing properties were performed at the BM5 beamline using X-ray energies from 10 to 30 keV. The focal properties of nanofocusing refractive lenses at energies higher then 50 keV were studied at ID 15 beamline.

At BM05 the lateral size of the microbeam was measured by scanning an Au wire of 200 µm in diameter across the beam vertically, recording the transmission radiation by a PIN-diode detector. All lenses on the chip have shown the focal spot (FWHM) around 200-300 nm. All compound refractive lenses in the ILS focus X-rays at designed distance with accuracy better then 2% when changing the X-ray energy. In order to study the influence of fabrication inaccuracies on focusing properties of nanolenses, we have tested lenses on three Si chips varying the scalloping amplitude and pitch. The measured optical properties of lenses on these chips are presented in Table 2. The best resolution (FWHM) and gain in flux density were measured for the lenses with 20 nm roughness.

Table 2. Summary of the experimental results at BM05

Chip	Energy, keV	Focal distance, mm (theoretical)	Focal distance, mm (measured)	Focal spot FWHM, nm	Transmission, %	Gain
1[a]	15.0	25	26.75	240	20	22.7
	17.5	34	35.50	280	27.1	21.7
2[b]	15.0	25	25.00	180	16.7	25.8
	17.5	34	33.75	220	24.8	35
3[c]	15.0	25	25.00	170	18.6	25.2
	17.5	34	34.00	140	24.6	30

[a] Scalloping amplitude 30 nm and scalloping pitch 400nm; [b] Scalloping amplitude 25 nm and scalloping pitch 250nm; [a] Scalloping amplitude 20 nm and scalloping pitch 400nm

At high energies the lateral size of the microbeam was measured by scanning a 35μm thick Au knife-edge across the beam vertically, recording the intensity by PIN diode detector. Results of the measurements are summarized in Table 3. We would like to stress that 150 nm is the best ever measured focal spot at 50 keV.

Table 3. Summary of the experimental results at ID15

Energy, keV	Focal distance, mm	Number of lenses	Focal spot FWHM, nm (theory)	Focal spot FWHM, nm (experimental)
50	106	324	70	150
60	124	392	80	250
70	172	392	110	250
80	225	392	150	200

4. Conclusion

We have designed, manufactured and experimentally tested the planar refractive lenses for hard X-ray nanofocusing. Planar refractive lenses were designed as Integrated Lens System which combines the advantages of compound refractive optics with the precise alignment possibilities of planar fabrication technology. The lens optical properties as size of the focal spot, gain were performed at ESRF BM05 and ID15 beamlines. The resolution below 200 nm was measured in the energy region of 15-80 keV. The resolution of 150 nm was demonstrated at 50 keV energy, to our knowledge this is the best measured resolution within high energy X-rays. As a next step we would like to realize two-dimensional focusing with a specially developed nanopositioning system. In the future we would like to optimize the lens design in the way to have better focal distance matching for vertically and horizontally focusing lenses.

5. References

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