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Reflection zone plates for 2D focusing and spectroscopy of hard X-rays

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Abstract. We propose the use of reflection zone plates (RZP) for two main applications: focusing and spectroscopy. A RZP combines three optical properties - reflection, imaging and dispersion - in one optical element. Having been successfully implemented into an X-ray fs-beamline at BESSY II, RZPs could be an important device in upcoming new high brilliance X-ray sources such as Free Electron Lasers (FEL). The reduction of the number of optical elements in a beamline would allow the highest possible transmission and satisfy the purpose of conserving the unique properties of FELs such as their high coherence.

1. Introduction

Reflection Zone Plates combine the imaging and focusing properties of lenses with the dispersive character of diffraction gratings. Such optical elements are of high interest for application at present and planning synchrotron facilities. Their implementation allows the reduction of the number of optical elements in a beamline. Less optical components mean less intensity loss. Higher intensity at the experiment normally means more significant results.

Therefore, the development of RZPs has been of high interest in the synchrotron community for several years. Takano et al. [1] described the successful application of one-dimensionally focusing (1D) RZPs for hard X-rays at Spring-8. Erko et al. [2, 3] reported about design, fabrication, and application of two-dimensionally focusing (2D) RZPs for the soft and hard X-ray ranges. Promising results were reached for soft X-rays at BESSY II [2].

The fabrication of 2D RZPs for the hard X-ray range is a technological challenge. The minimum zone size in lateral dimension for these devices is in the range of a few dozens of nanometers. These demands can be fulfilled by using e-beam writing. The creation of the third dimension of the RZPs is more complex. The distance between the upper and the lower level is in the range of only about ten nanometers. Standard procedures for the generation of the second structure level are reactive ion etching (RIE) processes. The lateral precision for the etching process should be less than 1 nm. Lateral variations of more than 1 nm can already cause intensity losses of about 10%. Therefore, high precision processes are on demand. Beside RIE, additive processes like metal sputtering can deliver high precision. For the investigation of the best fabrication method, very precise RIE processes were compared with sputtering processes with shorter durations.

The goal of this investigation is design, fabrication, and characterization of 2D RZPs for the hard X-ray range. At the beginning, new software for the calculation of zone plate structures has been developed and tested. Using the calculated ASCII-data, the RZP patterns could be written onto a

sample via e-beam writing. After the fabrication, the RZPs were characterized at the KMC-2 beamline of BESSY II (Germany). RZPs can be designed in on- and off-axis mode, which is defined by the section of the zone structure that is fabricated and thus illuminated. On-axis means that the section contains the center of the zone plate (optical axis), whereas in off-axis zone plates the center is out of the illuminated region. This causes a separation of the specular reflection and the diffraction orders and thus the energy dispersion.

2. Fabrication

The minimum zone size of hard X-ray RZPs is in the range of a few dozens of nanometers. Therefore, it is necessary to use modern nanopatterning technologies like e-beam writing to realize them. As mentioned above, to produce sufficient data files for the e-beam writing, the software for high resolution zone structure calculation was developed.

This software uses flexible parameters for the source-center distance, the center-detector distance, the energy of incident beam, the incident angle, and the resolution. The calculations were done for the Ni K edge energy of 8333 eV for both off-axis and on-axis RZPs with imaging distances of 10 cm and 50 cm.

Figure 1 shows the steps of the best fabrication process of such RZPs. Starting with a plane super polished silicon substrate of sufficient thickness, a positive e-beam resist (AR-P 671.02, Allresist GmbH) was spin coated onto the surface and patterned by e-beam writing (step 1). The exposed resist was then developed in an organic solution (AR600-56, Allresist GmbH) (step 2). In the following reactive ion etching procedure remaining rests of the developer were removed (step 3). To create the actual structure of the zone plate, the comparison between sputtering and RIE clearly showed that sputtering is much faster and equally precise. So, the final RZPs were structured by sputter-coating the sample with a Ni layer of approximately 10 nm (step 4). The rest of the resist was then lifted off the substrate (step 5), which leaves the zone plate structure formed by the Ni pattern on Si. A gold finishing (step 6) ensures high reflectivity in the hard X-ray range.

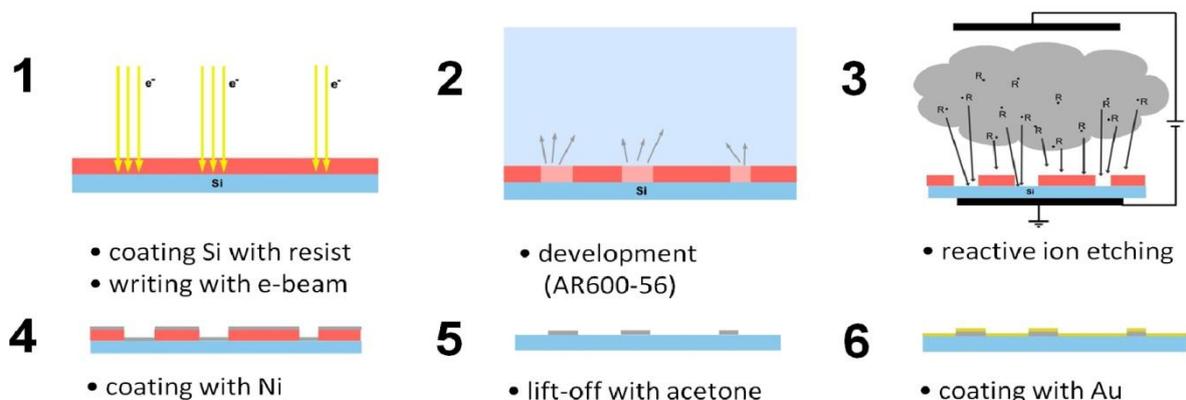


Figure 1. Flow chart of the fabrication process including RIE.

3. Setup and Results

The fabricated RZPs were characterized in two directions: mechanical properties and optical features.

The mechanical properties were determined with SEM and AFM; their optical properties were characterized directly at the KMC-2 beamline.

The structure's lateral dimensions were measured at LEO 1050 SEM. It

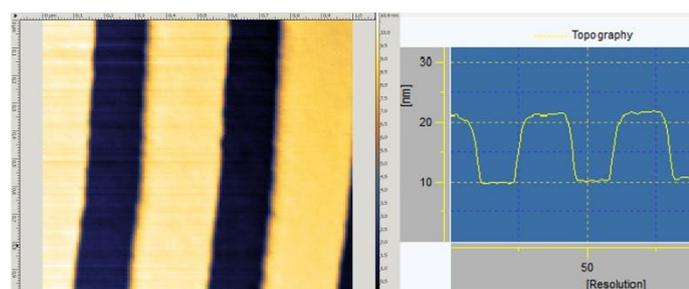


Figure 2. AFM image of a RZP with a topography spectrum.

allows the determination of lateral distances with nanometer accuracy. The minimum zone size of the RZP was measured as 40 nm, which matches well with the value of the design. For our RZPs, a maximum deviation between design parameters and measured dimensions of less than 5 nm was found. The surface roughness and the distance between the two levels of the structures were measured with an AFM (see figure 2). The measured roughness at the silicon as well as at the nickel surface was determined to be less than 1 nm over the whole RZP area. The distance between the lower and upper level of the RZPs used for measurements at the beamline was determined to be $10 \text{ nm} \pm 1 \text{ nm}$.

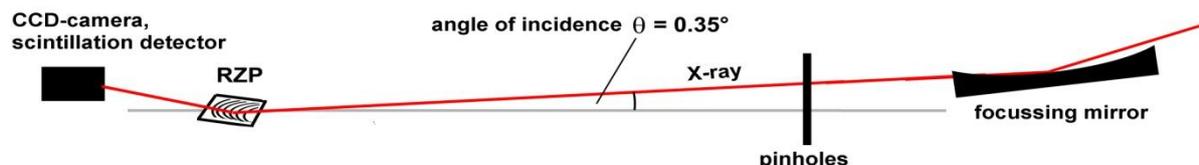


Figure 3. Scheme of the experimental setup.

Finally, the RZPs were tested at the KMC-2 beamline at BESSY II. The synchrotron radiation beam went through pinholes (200 μm and 500 μm) to create a well defined point source (figure 3).

At first, a combination of off-axis zone plates with an imaging distance of 50 cm was examined. It consists of two zone plates; an inner one (A) and an outer one (B) (see figure 4,a). The zone plates were designed to project their 1st (inner one) and 3rd (outer one) diffraction order into the focal spot.

Figure 5 shows a camera picture of the specular reflection, the combined focal spot of A and B and the 1st diffraction order of B, which is out of focus and therefore appears blurred. Different wavelengths of the incoming light caused different vertical positions of the focal spot, which proves that off-axis RZPs can be used as spectroscopic elements even in the range of hard X-rays (see figure 6).

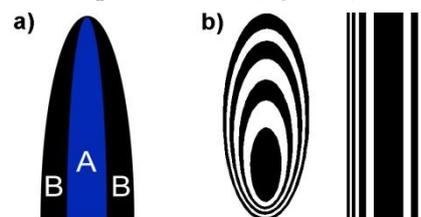


Figure 4. Design of the examined zone plates: (a) a combination of 2 off-axis RZP, (b) an elliptical on-axis RZP and a sagittal 1D RZP.

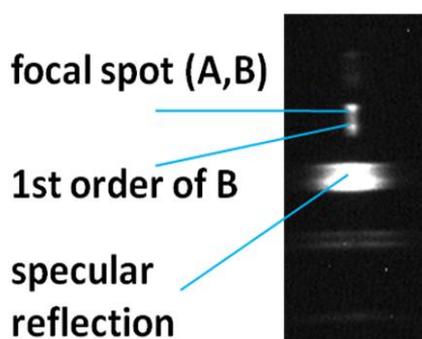


Figure 5. Camera image of the diffraction pattern and reflection of the off-axis RZP.

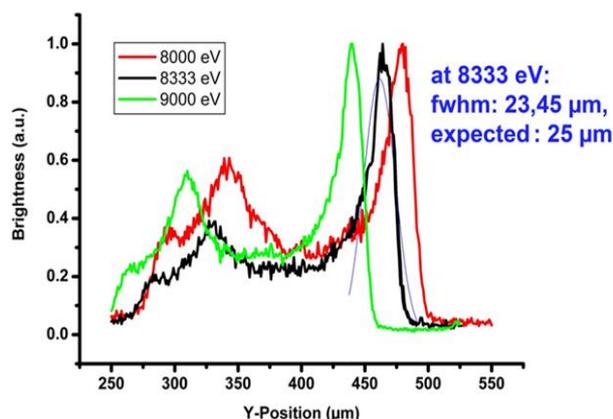


Figure 6. Dispersive property of the off-axis RZP: the vertical position of focal spot changes with the energy.

In a second experiment, on-axis RZPs were tested. This time, two different pinholes were used as light sources; with diameters of 500 μm and 200 μm . As an additional device for comparison, a one-

dimensional RZP with the same focal length for the same photon energy of 8333eV, which focuses the beam only in sagittal direction, was created (see figure 4,b). The distance between the RZPs and the focal plane was 10 cm. By changing the pinhole size, the imaging properties of the RZPs were shown (see figure 7). The source size is depicted with the expected reducing factor of 1/100, which results in spot sizes 100 times smaller than the pinhole sizes (see figure 8).

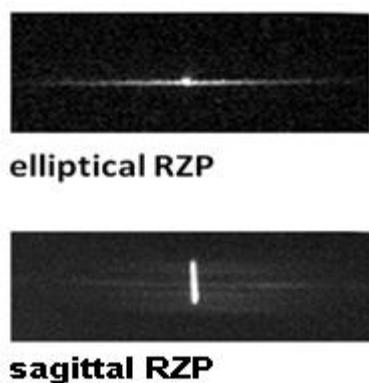


Figure 7. Camera images taken for on-axis two-dimensional and sagittal 1D RZPs.

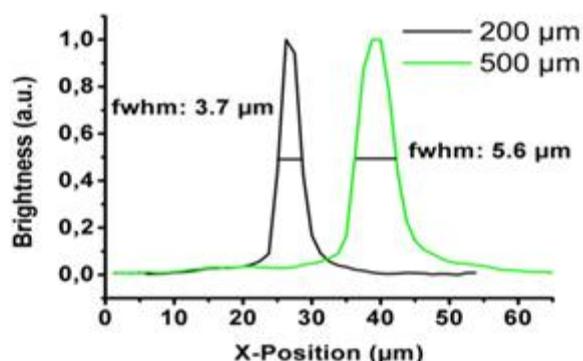


Figure 8. Influence of pinhole diameter on the size of the focal spot.

4. Conclusions

During this investigation Reflection Zone Plates of different characteristics for the hard X-ray range have been successfully fabricated and characterized. The focusing and imaging properties of on-axis as well as off-axis RZPs were studied. It has been shown, that RZPs work as focusing elements for hard X-rays in both modes. The dispersive characteristics of the off-axis RZPs were proven clearly on the basis of the vertical shift of the focal spot at different photon energies. The roughness and precision of the RZP surfaces was kept below 1 nm during the fabrication process.

RZPs have proved themselves to be excellent optical elements for focusing hard X-rays. In the off-axis mode, RZPs can be used as spectroscopic elements which offer new and interesting opportunities for time-resolved investigations as planned for new radiation sources like XFEL.

Acknowledgements

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