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1D Silicon Refractive Lenses for Surface Scattering with High Energy X-rays

F. Bertram¹, O. Gutowski¹, J. Patommel², C. Schroer¹ and U. Ruett¹,a)

¹DESY Photon Science, Notkestrasse 85, 22603 Hamburg, Germany
²Technical University Dresden, D-01062 Dresden, Germany

a)Corresponding author: uta.ruett@desy.de

Abstract. At the high energy X-ray beamline P07 at PETRA III, 1D focusing down to 4 micrometer vertical beam height while preserving a horizontal beam width of 0.5 mm was established by refractive lenses etched into a silicon wafer. A single wafer with 8 different lens structures can cover the full energy range between 50 and 120 keV. For surface diffraction on ultrathin films a factor of 4 in intensity can be achieved compared to the already established Al-compound refractive 2D-lenses.

INTRODUCTION

Surface X-ray diffraction studying crystal truncation rods of a 2 dimensional system is usually performed in grazing incident geometry, with an incident angle smaller than the critical angle of the substrate. Rotating the sample while preserving the angle of incidence on the surface provides images of the crystal truncation rods in the reciprocal space. High energy X-rays of energies above 50 keV can be employed for structural investigations of surface layers, when restrictive sample environments are used and a fast overview of the reciprocal space is necessary for time resolved experiments [1]. Here, full advantage of the small Bragg angles due to the large Ewald’s sphere is taken. A drawback is the reduced flux compared to the usual X-ray range of about 10 keV and the small critical angle (usually < 0.05°) requiring a well focused beam and a very smooth surface of the substrate.

The focusing of high energy X-rays can be easily done by Al compound lenses with a small radius of about 50 micrometer. At P07 at PETRA III a focus of 2.5 micrometer vertically and 30 micrometer horizontally can be achieved at 80 keV with 62 lenses accepting 0.3 × 0.3 mm² of the incoming beam corresponding to 10% of the total beam. An overview of the outline of the experimental station is shown in Fig. 1.

For studying of ultrathin films, an increased horizontal beams width would be beneficial for better integration over the surface, and also utilize a large beam size. Al-1D compound refractive lenses are only commercially available with a radius of 0.2 mm, so about 300 lenses would be required to focus a X-Ray beam of 80 keV photon energy at P07.

Therefore, structures of refractive lenses etched into a silicon wafer of 1 mm thickness especially adapted to the setup at P07 were tested, as an alternative to the Al-1D lenses. We present the characterization of the lenses at different energies and a comparison of real data from a test system of iron oxide thin films on MgO.

SILICON COMPOUND REFRACTIVE LENSES

Silicon compound refractive lenses are already successfully used for nanofocusing of hard X-rays [2, 3]. Calculations have been performed to adapt the lenses to the geometrical needs at P07 taking into account that the distance between lenses and sample position can be varied between 4.3 and 7.5 m. A 1 mm thick silicon wafer could be etched up to 0.9 mm in depth corresponding to a horizontal beam acceptance of 0.9 mm, respectively. The foreseen wafer allows to manufacture structures up to a length of 85 mm. So the amount and size of lenses was calculated to take full advantage of the material and maximize the acceptance of the lenses. It was found that 8 different structures are necessary to cover the required energy range of 50-120 keV, see Tab. 1. Due to problems with etching the depth for the test structures was
FIGURE 1. Outline of the P07-DESY hutches at PETRA III. The compound refractive lenses are located in the secondary optics hutch. The distance to the sample can be adjusted between 4.3 and 7.5 m.

FIGURE 2. Picture of the lens structures etched into a silicon wafer. 8 different structures can be used to focus photon energies between 50 and 120 keV.

limited to 0.5 mm. The wafer is shown in Fig. 2. In use the wafer is standing upright and the beam travels along the wafer through the etched structures. At 50 keV almost the full vertical beam size can be focused to a few micrometers. But even at 100 keV the aperture for the silicon refractive lens is larger than for the 2D Al-CRL with the usual radius of 50 μm.

TABLE 1. Description of the lens structures. N is the number of lenses in the structure. R is the lens radius, D the aperture.

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RESULTS

In the first place the performance of the lenses was characterized by a test structure of a 1mm wide line of Pd on a sapphire wafer. Here, the fluorescence of the Pd was detected at an angle of 90 degrees to the surface while translating the wafer vertically through the focused beam, with an angle of 0.05 degree with respect to the beam. The detected intensity reflects almost directly the vertical shape of the incident beam.
It was found that the lenses are not completely uniform horizontally due to problems of the etching process. Still a vertical beam size of less than 4 micrometers was achievable with an efficiency of better than 50%. The gain in intensity compared to 2D-lenses usually used at the beamline is about 4, even with these prototype lenses.

To test the performance with a real system, a sample was chosen with 2 different epitaxially grown layers: an thin film of magnetite (Fe₃O₄) on top of an ultrathin film of nickel oxide (NiO) grown on magnesia (MgO) [4]. Here, the diffraction pattern shows Crystal Truncation Rods (CTR) as well as diffractions spots because of the already 3D ordered film. The intensity along the CTR is modulated by the thickness of the film, see Fig. 3.

![1D lens 2D lens](image)

**FIGURE 3.** On the left the 2D data of the sample are shown for comparison of the 2D and 1D focusing lenses. On the right a line cut along the crystal truncation rod as seen on the 2D data on the left is shown. Additionally, a sketch of the sample is given. The resolution of the data is higher for the 2D lenses, and the contrast also seems to be higher. It is clearly visible that the 1D lens data provide more intensity, so that the weak film reflections become more pronounced and can be studied more precisely.

A comparison of the 2D lenses and the 1D lenses shows, that there is a significant gain in intensity for surface diffraction. Especially the intensity of the structural reflections of the ultrathin film becomes detectable because of the increased integration over a larger area of the surface. But it has to be mentioned that the resolution of scattered signal from the CTRs suffers. This is even more important, if thick films are studied.

**SUMMARY AND OUTLOOK**

The etched silicon 1D lenses are a very successful solution for focusing high energy X-rays. A proven significant gain in intensity was achieved comparing the scattered intensity of the very same sample in the very same setup. The production of the lenses should be further improved to allow focusing of at least 0.9 mm horizontally and to decrease the vertical beam size further. The lenses are easily to handle and simple to align. They can be moved in and out reproducibly for quick setup changes in less than a minute. They are more robust than individual CRL put together to a long structure, since they always stay together as they are and a new structure is simply picked by translating the wafer through the beam.

The lenses were already successfully used at 80 keV for in situ surface diffraction. They are also very important for studies of depth dependencies in samples. Here, the sample surface is aligned parallel to the beam and studied at different distances to the sample surface. Especially when nanostructures are investigated, where a good integration over a larger volume is required, the 1D lenses outclass the 2D lenses, because of improved intensities. Quick changes of both lens systems are of great advantage for synchrotron users to optimize the beam shape to the requirements of the experiment.
ACKNOWLEDGMENTS

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REFERENCES


