Low Cost Rolled X-ray Prism Lenses to Increase Photon Flux Density in Diffractometry Experiments

H. Vogt[a], A. Last[a], J. Mohr[a], F. Marschall[a], K.-U. Mettendorf[b], R. Eisenhower[b],
M. Simon[c]

[a] *Karlsruhe Institute of Technology (KIT), Institute of Microstructure Technology (IMT),*
Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

[b] *Bruker AXS GmbH, Östliche Reinbrückenstraße 49, 76187 Karlsruhe, Germany*

[c] *PI miCos GmbH, Freiburger Strasse 30, 79427 Eschbach, Germany*

ABSTRACT

At the Institute of Microstructure Technology (IMT) of the Karlsruhe Institute of Technology (KIT) a new type of refractive X-ray optics has been developed. Due to its comparably easy fabrication method and the large aperture the so called Rolled X-ray Prism Lenses (RXPL) have the potential to be used with X-ray tubes in industrial environment as a low cost alternative to existing optics. The lens itself is build out of a micro structured foil, which is cut into shape and rolled around a winding core to form a refracting element for X-rays. The resulting refractive structure can be used as illumination optics. Diffractometry experiments with a NIST 1976a sample where performed and showed an up to 18-fold enhanced integrated intensity compared to that acquired with a steel-tube collimator.

INTRODUCTION

The Institute of Microstructure Technology (IMT) at Karlsruhe Institute of Technology (KIT) produces multiple types of refractive X-ray optics. They are fabricated using the LIGA process (Saile 2009), which yields high aspect ratios and high surface quality. Figure 1 shows multiple examples of such refracting structures (from a to c): a compound refractive lens, a kinoform lens which allows for larger apertures and less absorption (Nazmov, Shabel'nikov et al. 2004) and a refractive prism lens (Simon, Reznikova et al. 2008).

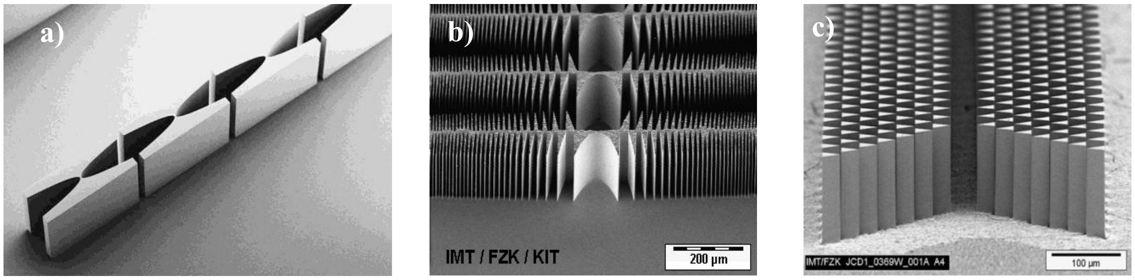

Figure 1: Different types of refractive collecting lens types for X-rays: a compound refractive lens (a), a kinoform (b) and a prism lens (c)

Applications for those optics range from illumination to imaging applications. Whereas in X-ray microscopy applications both types of optics are required to produce high-resolution images, in material analysis applications, like X-ray diffractometry, no imaging properties of the optics are required. For this type of application the prism lens is the most useful refractive optics, since it offers the possibility for comparatively large apertures and low absorption.

Nevertheless this lens type has three down-sides: First the aperture is limited by the height of the structures: the high columns are becoming unstable when exceeding a certain aspect ratio. Secondly to create a 2D refracting lens, two of these lens sets need to be aligned tilted to 90° and third the structures are fabricated using a high-aspect ratio process, which is cost-intensive and time consuming.

To improve this situation a new kind of refracting lenses was developed at our institute: the so called Rolled X-ray Prism Lenses (RXPL) [Simon, Reznikova et al. 2010]. Figure 2a shows a sketch with a cut-out of such a lens and Figure 2b a photograph of such a lens.

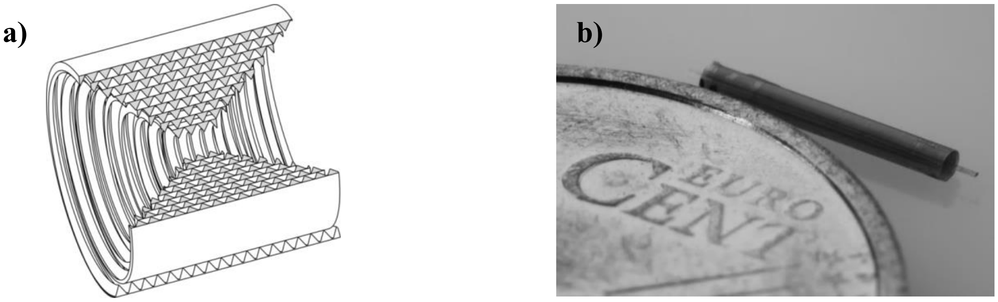

Figure 2: Principle sketch of a Rolled X-ray Prism lens (a) and a photograph of a produced lens (b)

The lens consists of a polyimide foil with triangular shaped corrugations on one side, which is cut and rolled around a glass-fiber to form a refracting element for X-rays. The lens aperture is not limited to the stability of the single refracting elements, since they are not free-standing. The absorption is low due to the low Z-number material, the lens has point focusing properties and for fabricating the lens no high-aspect ratio process is required.

With all these properties, the lens shows a promising potential to become a low-cost alternative for X-ray illumination applications.

FABRICATION

To fabricate a Rolled X-ray Prism lens three major steps are necessary: First the foil needs to be produced, second: the layout calculation and foil cutting and third the rolling process to form the actual lens.

FOIL FABRICATION

The foil is fabricated using a molding process. The mold itself is a structured silicon wafer. Figure 3a to f shows the principal steps to create the structure on the wafer.

An oxidized wafer is coated with a UV photoresist by spin-coating (Figure 3a). Then the photoresist is structured by UV-lithography using a chromium mask with a parallel line pattern, followed by an etching process which transfers this pattern into the silicon oxide (Figure 3b). After removing the photoresist, V-shaped grooves are realized by anisotropic KOH etching of the (100)-silicon wafer to form the final mold (Figure 3c). The wafer is oxidized again to prevent the gold layer deposited in the next step to amalgamate with the silicon. To produce the foil a thin gold separation layer is deposited on the wafer and a liquid polyimide precursor (FUJIFILM™ Durimide® 32) is spin-coated on top. After backing a thermal release tape (Nitto Denko™ Revalpha) is glued onto the foil to be able to peel the foil of the wafer without tearing it (Figure 3f). To remove the foil from the thermal release tape the tape is heated to neutralize its adhesion properties and the foil, around 3 µm thick with 7.1 µm high triangular shaped corrugations on one side, is released. Figure 3g right shows an SEM picture of the structures on one side of the foil.

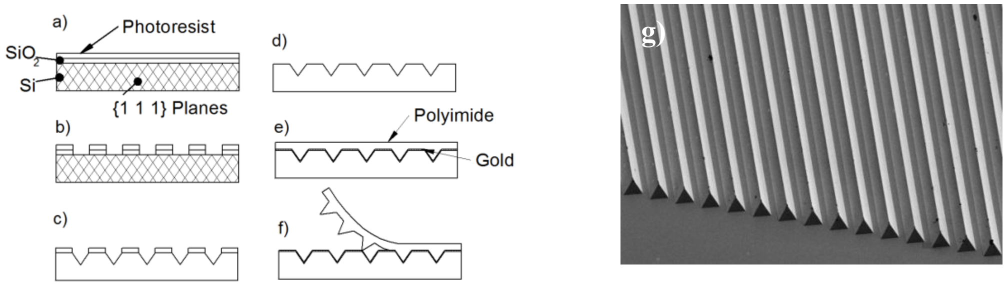

Figure 3: Principal steps to create a mold wafer for RXPL-Foils (a-f) and SEM picture of the structures on the foil (g)

SHAPE CALCULATION AND CUTTING

To derive the actual lens shape, the number of refracting elements in a certain distance to the optical axis needs to be calculated, which is necessary to refract a ray to a desired working distance. This is done using a ray-tracing program. In this simulation the absorption as well as the "shadowing" of the single layers can be calculated in advance. This shadowing occurs due to the fact, that every layer of a certain number of prisms has an entrance acceptance angle, in which rays are guided through the prism row without leaving it. Figure 4 shows a principle sketch to illustrate this behavior: the dotted line represents the shallowest ray; the dashed line represents the steepest ray which may enter the row of refracting elements in point P_3' respectively P_3 and is guided successfully through the layer, leaving it at P_1' respectivelyP_1. Every ray which has an angle between the shallowest and the steepest ray is guided through the layer, every other ray will be absorbed or misguided by reflection at the backside of the next inner layer. This behavior is called shadowing.

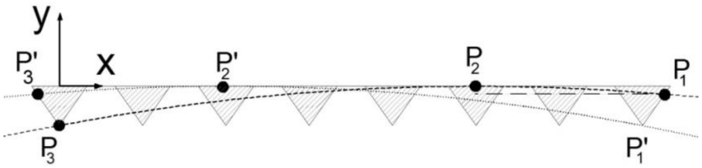

Figure 4: Principle sketch to illustrate the shadowing effect of a row of refracting elements

The shape calculation yields a necessary number of refracting elements in a certain distance to the optical axis. For example to produce a lens for illuminating a small point on the optical axis, the principle structure in Figure 5a is obtained: the number of prisms increases with growing distance to the optical axis.

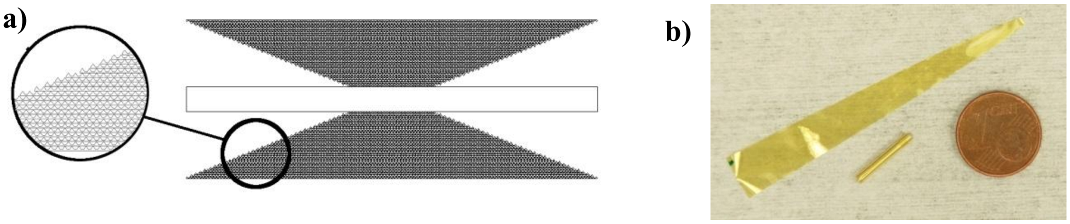

Figure 5: Resulting structure of an RXPL after layout calculation in rolled state (a) and foil layout after calculating and cutting the un-rolled state (b)

This shape is the *rolled* layout. To retrieve the *un-rolled* layout the formula of the Archimedean spiral is used, see e.g. (Weisstein 2013). Figure 5 right shows a photograph of the parabola-like resulting foil layout.

The cutting itself is done using a laser-ablation system. This yields high contour accuracy and undamaged foil edges, avoiding mechanical forces on the structured foil.

ROLLING

The third and last step is the rolling process. The rolling is done using a mechanical aid. This aid and the principal steps which are used to produce the RXPL are shown in Figure 6.

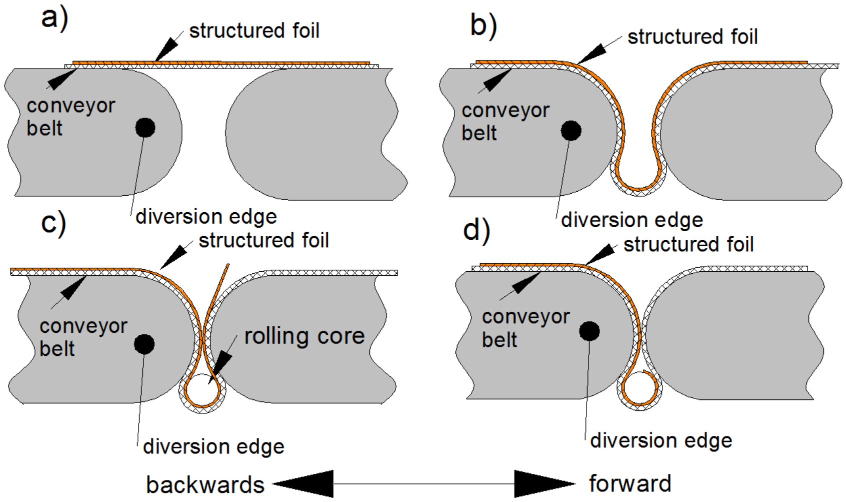

Figure 6: Principal steps to roll an RXPL foil

The cut foil is placed on top of a conveyor belt, which consists out of a 7 µm thick Kapton® foil (Figure 6a). In the next step the conveyor belt and the structured foil is pushed into a loop between two grinded metal edges, which are called diversion edges (Figure 6b). A glass-fiber 125 µm in diameter is placed in the loop and the distance of the diversion edges is reduced until it equals the thickness of two times the conveyor belt height plus two times the structured foil thickness (Figure 6c). Then the conveyor belt is fed backwards and the jutting end of the structured foil is fed into the loop. Then the distance between the diversion edges is further reduced by the height of the structured foil, so that the end cannot leave the loop, when feeding the conveyor belt in forward direction (Figure 6d). The forward motion of the conveyor belt is continued until the whole structured foil is rolled around the glass-fiber. Figure 7 shows two different lenses for different working distances and energies: the smaller one has a diameter of about 800 µm and the larger one is approximately 2 mm in diameter.

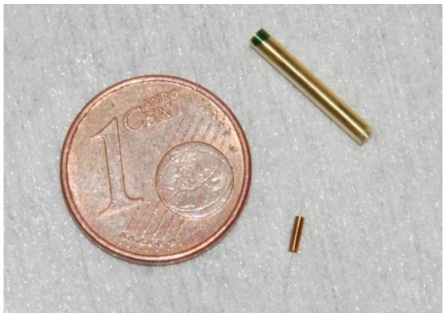

Figure 7: Two RXPLs with 800 µm and 2 mm diameter for different working distances and energies.

The resulting structure within the lens can be inspected by creating a radiographic picture of the lens. Figure 8 shows such a picture with enlarged cut-outs at different locations within the lens.

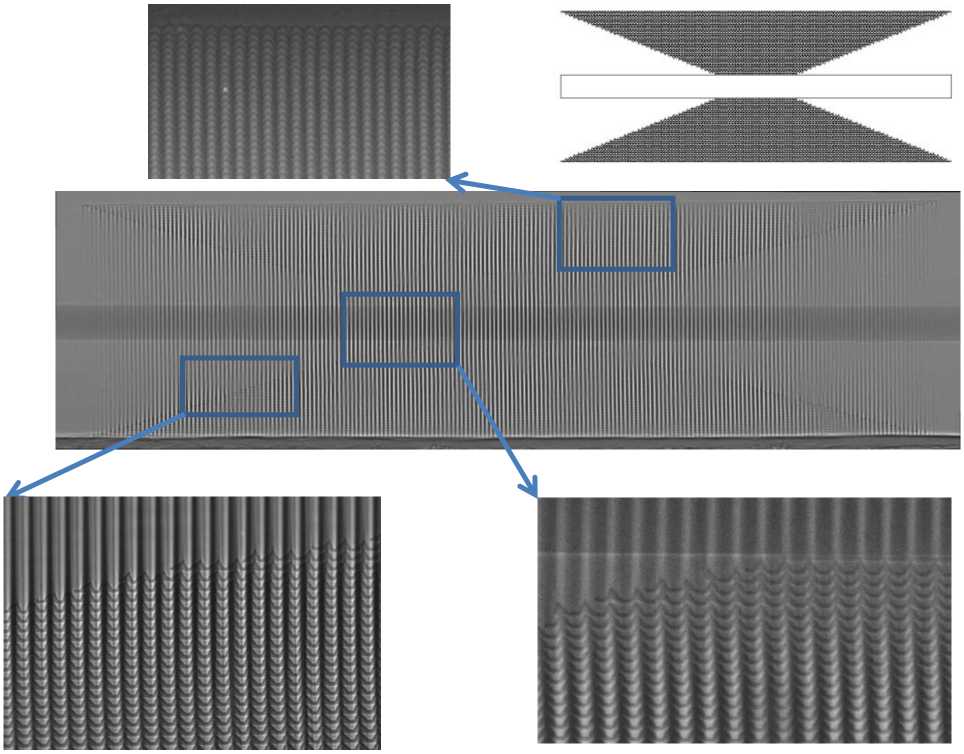

Figure 8: Radiographic picture of an RXPL with cut-outs at different positions within the lens and principle lens layout

When comparing the principle layout in the upper left corner of Figure 8 with the full-lens radiographic picture in the middle the similarity becomes apparent. The cut-outs show high structure quality at different locations within the lens.

EXPERIMENTAL RESULTS

The RXPLs where tested at synchrotron sources as well as in a tube diffractometry experiment. Figure 9 shows for example the results obtained at ANKA, TopoTomo beamline in Karlsruhe, Germany. The lens was designed for a working distance of 150 mm for 9.9 keV. The graph in Figure 9a is the maximum intensity on a CCD detector (PCO 4000) for different distances between CCD and lens exit aperture. Figure 9b shows a cut perpendicular to the optical axis through the focus at the maximum intensity distance.

The working distance of 145 mm is below the desired one of 150 mm. The Full-Width-Half-Maximum (FWHM) of the peak perpendicular to the optical axis in vertical direction lies closely to the theoretical minimum of around 10 µm due to the height of the prisms. The FWHM in horizontal direction is larger (17 µm) due to the shape of the source. The overall spectral intensity enhancement, which is the ratio between the mean intensity on the detector within the FWHM area of the peak with the lens and the mean intensity within the FWHM area without the lens, was around 60.

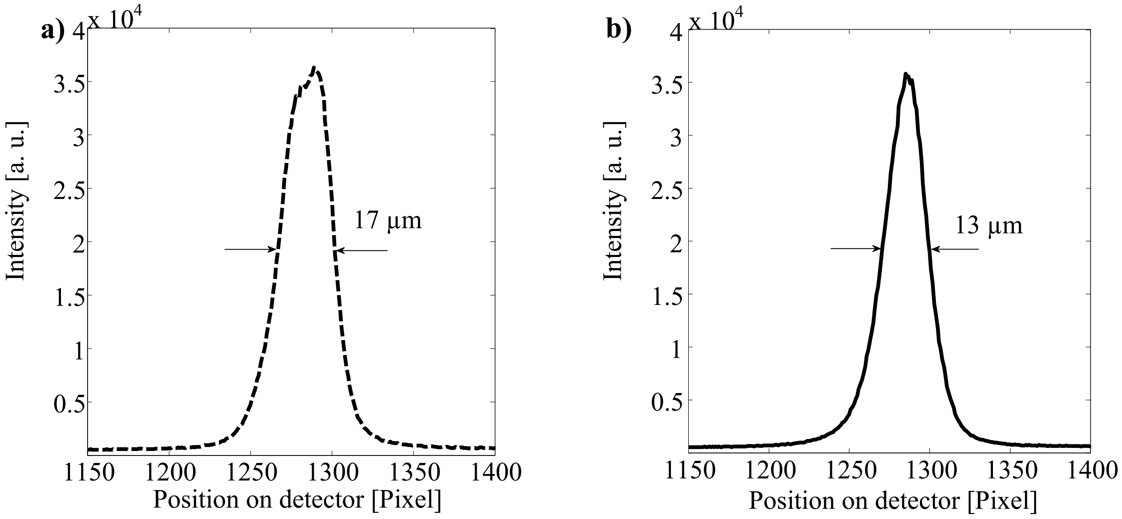

Figure 9: Results obtained at 9.9 keV at a synchrotron source: maximum intensity on detector versus distance between lens exit aperture and detector plane (a) and cut perpendicular to the optical axis through the focal spot at the position of maximum intensity along the optical axis (b)

The diffractometry experiment was performed in an existing Bruker D8 Discover setup. Figure 10 shows the principal setup.

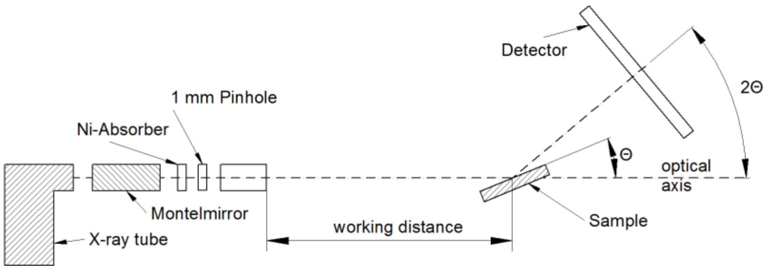

Figure 10: Principal setup diffractometry experiment

To create a semi-monochromatic source and to reduce divergence of the X-rays emitted by the X-ray tube (Siemens KFL-CU-2K) a Montel mirror (Incoatec Montel-p) and a Ni-absorber foil was placed between the tube and the RXPL. The lens itself was then mounted on a tilt-stage to be adjusted to the beam. The sample used was a NIST-1976a corundum powder sample and the diffractogram was acquired using a VANTEC 500 detector. To acquire a figure of merit the diffractogram was once acquired using an RXPL and once using a steel tube collimator, which yielded the same focal spot diameter on the sample. Figure 11 shows the resulting diffractograms: the diffractogram acquired using the steel tube collimator (a) and the diffractogram acquired using the RXPL lens (b). The difference is apparent: the diffractogram with the RXPL shows additional lines as well as fewer speckles, but also more background towards the beam axis. In Figure 12 the integrated intensity values are plotted: the diffractogram with the lens shows an up to 18-fold enhanced intensity compared to that with the steel tube collimator for same acquisition times. The peak-shift between the integrated intensities results from a shifting of the position of the focal spot on the sample.

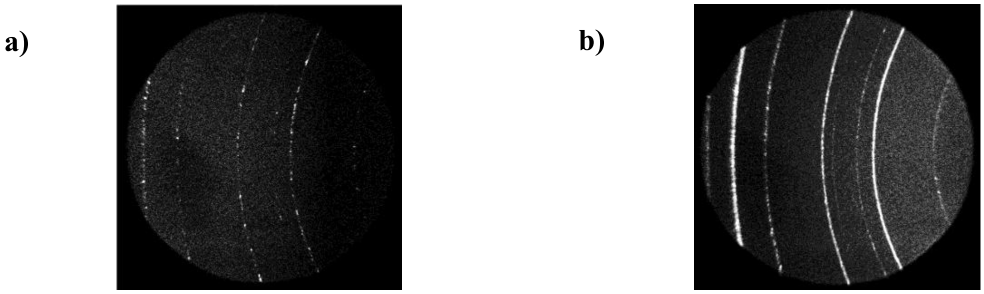

Figure 11: Two-dimensional diffractogram patterns acquired using a steel-tube collimator (a) and an RXPL (b)

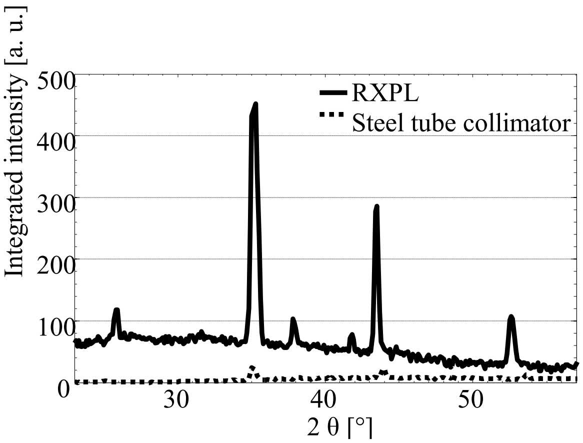

Figure 12: Integrated intensities derived from diffraction patterns in Figure 11

CONCLUSION

RXPLs offer an alternative to existing illumination optics at synchrotron sources as well as for tube sources. The fabrication process does not require any time consuming and cost intensive high-aspect ratio processes. Therefore RXPLs can be a low-cost alternative to other X-ray illumination optics. Experiments at synchrotron sources show the potential of the lenses (spectral intensity enhancement of 60) and diffractometry experiments show promising results (integrated intensity enhancement 18-fold compared to steel-tube collimator).

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