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X-ray Full Field Microscopy at 30 keV

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Abstract. In our X-ray full field microscopy experiments, we demonstrated a resolution better than 260 nm over the entire field of view of 80 µm x 80 µm at 30 keV. Our experimental setup at PETRA III, P05, had a length of about 5 m consisting of an illumination optics, an imaging lens and a detector. For imaging, we used a compound refractive lens (CLR) consisting of mr-L negative photo resist, which was fabricated by deep X-ray lithography. As illumination optics, we choose a refractive rolled X-ray prism lens, which was adapted to the numerical aperture of the imaging lens.

1. Introduction

Hard X-ray full field microscopy allows high resolution transmission imaging of samples, which are opaque for visible light. This is often required in materials science and microstructure technology. Today most of the X-ray microscopes installed at synchrotron radiation sources or with X-ray tubes are based on Fresnel zone plates. A reasonable resolution can only be achieved for energies up to about 20 keV, due to the limitation in fabrication of high aspect ratio zone plates with an outer ring widths in the submicrometer range. Microscopes, using refractive lenses for hard X-rays, have been built up at synchrotron beam lines [1] and tube sources [2, 3]. Due to several reasons, full field microscopes for hard X-rays were strongly limited in resolution or required large distances between the optical components. Here we realized an X-ray full field microscope for 30 keV, with higher resolution and shorter setup length, using compound refractive lenses (CLR) out of mr-L negative photo resist. In our very first experiment we achieved a resolution better than 260 nm over a field of view of 80 µm x 80 µm with a setup of around 5 m length. Our results show that this experimental setup has a high potential to revolutionize the field of hard X-ray full field microscopy, opening up new possibilities for studying materials in research and industry.

2. Microscope setup

The microscope was setup at beamline P05 at PETRA III. A sketch of the setup is shown in Figure 1. The microscope consists of an illumination optics, an adjustable diaphragm, an objective lens and a detector. A biconcave parabolic compound refractive lens was used as imaging optics and a rolled X-ray prism lens for the illumination optics. As detector we choose a high resolution system using a LSO-scintillator and a magnifying optics for imaging the fluorescent light on the CMOS-detector.

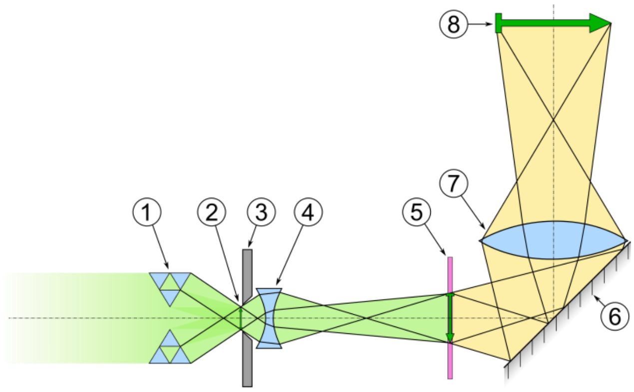

Figure 1: Setup of an X-ray full field microscope with refractive lenses for synchrotron beam lines, consisting of 1) condenser lens, 2) sample, 3) diaphragm, 4) objective lens, 5) scintillator, 6) 45° mirror, 7) microscope objective for visible light, 8) CMOS-detector; the path of the X-rays until the scintillator is drawn in green, the path of the visible light starting at the scintillator is drawn in yellow

In order to achieve the optimum performance of the microscope the magnification factor needs to be adapted to the achievable resolutions in the different imaging planes. For a theoretical resolution of the objective lens of 60 nm and a resolution of the scintillator of 1.5 µm the magnification in the X-ray part of the microscope has to be at least 26. This ensures that all details resolved by the objective lens are also visible on the scintillator. Additionally, selecting only the necessary X-ray magnification for highest resolution, allows the length of the microscope to be as small as possible.

In the same way, the size of the pixels of the CMOS-detector defines the magnification of the visible light objective in front of the detector. For resolving one unit of line and space at least 2.5 pixels, better 4 pixels or more, are needed. Therefore, a magnification of 20 is needed to image 1.5 µm line and space on 4 pixels with a size of 7.3 µm.

For an objective lens with a focal length of 90 mm the sample distance was set to 95 mm and the image distance was 2500 mm which results in the selected magnification of 26. In combination with an illumination lens, which had an illumination distance of 2.5 m, the overall length of the microscope was about 5 m.

3. Objective lens

A compound refractive lens was used as imaging lens. The lens had a focal length of 90 mm and an entrance aperture of 71 µm. It was fabricated by deep X-ray lithography out of mr-L negative photo resist [4] at KIT/IMT. The lithography was done at the X-ray lithography beamline Litho 2 at ANKA. Deep X-ray lithography allows fabricating submicron structures with very high precision [5].

However, lithography allows only the fabrication of linear structures; rotational symmetric lenses cannot be produced by this technique. To solve this problem, the refraction for horizontal and vertical direction is separated and effected by different surfaces, which are fabricated by two separate lithographic exposures. An alternating crossed arrangement of horizontal and vertical lens elements however allows 2D-imaging. In order to avoid astigmatism, the number of lens elements for focusing in one direction is increased by one, with the first and the last element having only half of the refractive power than the other elements. Thus, the principal planes for both directions match each other and the focal length for both directions is equal.

The lens we used in our experiment consists of 249 biconcave parabolic lens elements in total, which means 124 biconcave parabolic lens elements in horizontal and 125 vertical direction. The radius of curvature *r* in the apex of each parabola is 6 µm. The distance *d* between the two surfaces of one lens element is 7 µm, the distance *a* between two lens elements is 30 µm (see Figure 2). The total length of the lens, which is a result of the number of lens elements *N*, the length *L* of the lens elements and the distance *a* between two lens elements, is limited to 60 mm due to the current fabrication process at KIT/IMT. Therefore, the aperture of the lens is geometrically limited to 70 µm, if the aperture of all lens elements is equal.

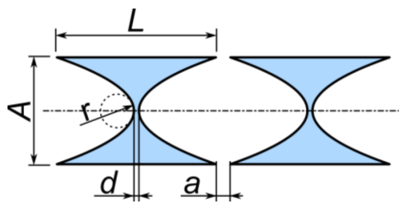

Figure 2: Layout of a parabolic compound refractive X-ray imaging lens (r – radius of curvature in the apex, A – aperture, d – smallest distance between to surfaces of one lens element, a – distance between two lens elements, L – length of one lens element)

The absorption limited aperture of the lens, also called effective aperture [6], is 78 µm. This value is larger than the geometrically possible aperture, which is limited because the overall length of the X-ray lens is limited. Optimizing the apertures of the lens elements to the application is beneficial for achieving best results in each application. An objective lens for full field microscopy has to be optimized in order to achieve a homogeneous and high image quality. Thus, it is important that the lens has a high numerical aperture for all points of the entire field of view.

The result of the optimization is a lens with an aperture varying from 71 µm in the entrance plane, to 66 µm in the middle and to 82 µm in the exit plane [7]. Due to this shape of the lens the resolution increases over the entire field of view. Especially at the borders of the field of view the gain is significant, resulting in a more homogeneous resolution and brightness of the image. For the optimized lens a theoretical resolution of 60 nm in the center and 78 nm at the borders of a field of view of 100 µm x 100 µm was calculated. The average transmission of the lens is 61 %.

To fabricate a lens with horizontal and vertical elements, which are tilted by 90° with respect to each other, two X-ray lithographic exposures with +45° and -45° are needed. The absorber mask used for the exposure consists of a titanium membrane with 2.7 µm thickness, carrying gold absorbers with 20 µm thickness. As the total size of the membrane is 23 mm x 63 mm, the membrane is fragile and avoiding bending of the membrane is very challenging. Due to the 45° exposures bending of the membrane of the absorber mask is directly imaged to the substrate in the lithography step, which results in an incorrect alignment of the lens elements on the substrate. For the lens we used in our experiment we found that the maximum deviation from the ideal position was ± 7 µm. Therefore, the size of the usable aperture was reduced by 14 µm and consequently a theoretical resolution of only 80 nm in the center of the field of view would have been achievable.

4. Condenser lens

Synchrotron beamlines offer a nearly parallel beam. Using imaging lenses for sample illumination results in a small focal spot size, which is sufficient for illuminating a very small area on the sample but not for illuminating an expanded field of view homogeneously (see Figure 3a). Illuminating a larger area by defocusing results in a significantly decreased number of photons on each point of the field of view. Additionally, defocusing reduces the angular variance in illumination directions. While the overall angular spread is constant, each individual point in the field of view suffers from a reduced angular spread (see Figure 3b). So, for illumination an expanded field of view, imaging lenses are not the best choice at synchrotron beamlines. Thus we choose a rolled X-ray prism lens (RXPL) [8] as illumination optics, which provides an homogeneous illumination over an extended field of view (see Figure 3c).

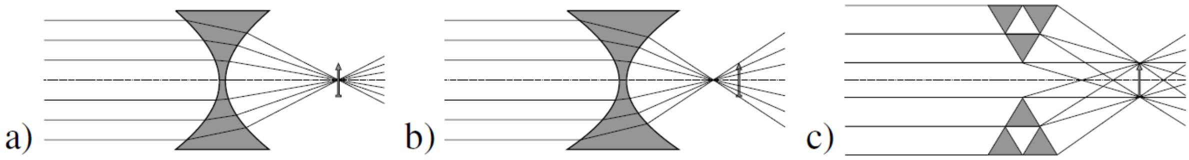

Figure 3: Schematics of illumination optics with parallel incoming rays: a) using an imaing lens focused on the sample, b) using an imaging lens not focused on the sample, c) using an prismlens

Rolled X-ray prism lenses consist of rolled up structured foil, which results in a huge number of layers each containing multiple prisms (see Figure 4). The prisms just change the direction of an incoming parallel beam, not its divergence. Besides the refractive index, the direction of the outgoing ray is determined by the number of prisms in one layer, which can be chosen freely by choosing the shape of the foil before rolling it up. Thus, the incoming beam is cut into sections by each row illuminating a defined part of the sample under a defined direction. As a result each point of the sample is illuminated from multiple directions, which is advantageous in full field microscopy. Additionally, the photon flux on the sample is increased.

The condenser we choose for our experiments had a working distance of 2.5 m, an aperture of 2 mm and a theoretical transmission of 88 %. The lens was specifically designed for illuminating the entire field of view with a divergence tailored to the objective lenses acceptance angle. Thus, we achieved a high brightness of the image and consequently a short exposure time of a few seconds.

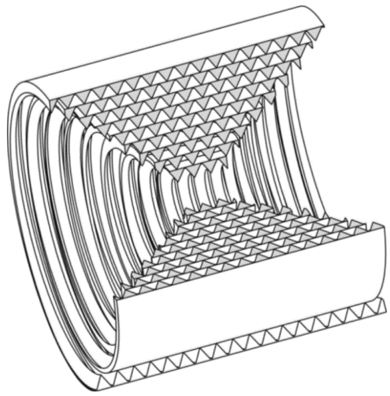

Figure 4: Schematic of a rolled X-ray prism lens

However, rolling up a foil with 10 µm thickness is a very complicated process, and a total avoidance of fabrications defects is highly challenging. We found, that the illumination is not yet completely homogeneous. However our experiment proves that this can be easily compensated for, by dividing the image by its flat field.

5. Experimental results

Our very first experiments for full field microscopy at 30 keV took place at PETRA III, P05 [9]. The 6.8 m long granite optical bench with several high precision parallel kinematic stages mounted on sliders is an excellent basis for full field microscopy. The distance between condenser lens and scintillator was around 5 m for an X-ray optical magnification of 26. As detector we used an 11 µm thick LSO-scintillator combined with a 45°-mirror, a 10-fold or 20-fold microscope objective, and a Nikon D4 camera.

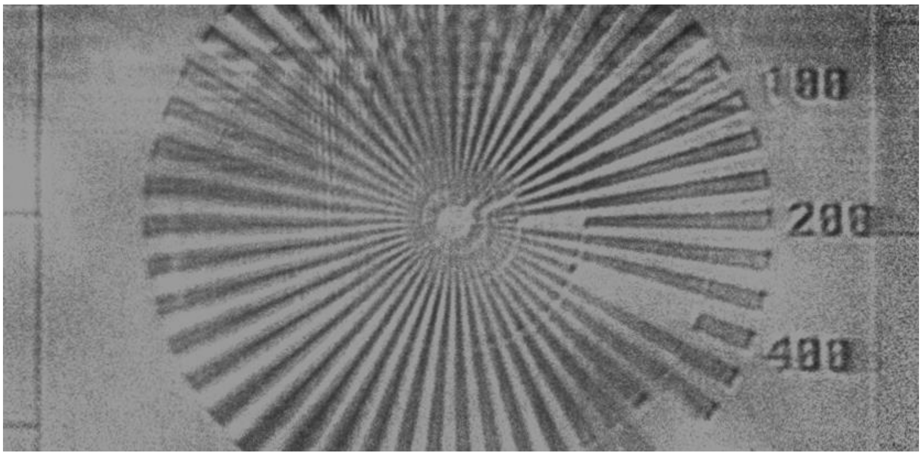

Figure 5: X-ray micrograph of an Xradia nano test pattern at 30 keV divided by its flat field (30 µm outer diameter of Siemens star)

With our setup we have been able to demonstrate a resolution better than 260 nm over the entire field of view of 80 µm × 80 µm, which means that the smallest visible structure details with a width of 130 nm had a periode of 260 nm. Figure 5 shows a detail of a micrograph of an Xradia nano test pattern divided by its flat field. The exposure time for this image was 30 s. The gold layer of the sample was only 500 nm thick, so the transmission was 97.5 % at 30 keV, which is challenging but sufficient for absorption contrast imaging.

6. Conclusion

Our concept based on polymer compound refractive lenses as objective lenses and rolled prism lenses as illumination optics allows building up fast and compact high energy microscopes with high resolution at synchrotron beamlines for ambient conditions. With our microscope, using compound refractive lenses out of mr-L resist, structure details down to 130 nm line and 130 nm space are visible. So, the resulting resolution is 260 nm with a field of view of 80 µm × 80 µm. Our theoretical calculations show that resolutions of below 60 nm are achievable with an optimal set of lenses. As several improvements are currently implemented in the lens fabrication process, we expect approaching this limit in future experiments. The results demonstrate a high potential to make hard X-ray full field microscopy available for materials analysis.

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