ESRF	**Experiment title:** Hard X-ray focusing by multilayer Laue lenses with focal lengths > 10 mm	**Experiment number**: MI 1168
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Report:

Multilayer Laue lenses (MLL) are a promising novel approach to achieve small hard x-ray spot sizes [Maser2004]. The working distance of the microscope has to be in the millimeter range rather than in the micrometer range to allow for convenient sample handling and generic experiments. However enlarging the distance reduces the numerical aperture of the lens and thus enlarges the theoreticaly achievable spot size, if the thickness of the multilayer stack is kept constant. Primary objective of the experimental session was the evaluation of an improved version of crossed multilayer Laue lenses.

To offer both, a long focal distance and an improved spot size, it is necessary to produce a lens with a large aperture. Two different MLL designs with about 50 µm of total deposition thickness, being one of the largest so far, were produced and tested. A major challenge in the deposition process is its duration of several days. The process has to be monitored and well understood to ensure stability for such a period of time. Even small deviations from the zone plate law can lead to a significant reduction in focusing capabilities. Subsequently, a bar is cut out of the coated wafer and a thin section simillar to a TEM H-bar lamella is prepared by focused ion beam milling; this is the actual, onedimensionally focusing lens. Two of these parts are then glue-bonded perpendicularly to each other in order to obtain a point focusing device. The formation of the lamella and the relative assembly of both lenses is also a crucial step for the focusing capabilities of the system.

The resulting device is attached to a single mount and can be used instead of existing focusing optics in an x-ray microscope.

Basic MLL characterization

The nanofocusing lenses at ID13 were replaced with MLLs for the experiments in the scope of the proposal. Two different MLL designs have been tested at an x-ray energy of 15.25 keV:

Deposition	Focal length	Aperture	Zones	Smallest zone width
PS6056	15.3 mm	48 µm	50-2500	11 nm
PS6359	9.5 mm	53 µm	512-7000	5 nm

We used the coherent diffraction imaging technique of ptychography [Hoenig2011] as core method to characterize the lenses. The sample is scanned as in scanning transmission x-ray microscopy with an overlap of the illuminated parts on the sample and far-field diffraction patterns are recorded. Those patterns enable to calculate a reconstruction which gives high resolution phase and amplitude information about the sample as well as about the illumination within the sample plane. Using wave propagation, this information can be used to calculate the beam shape at the focus position, if the sample was slightly defocused.

For these measurements an x-ray beam energy of 15.25 keV was used and a MAXIPIX pixel detector was placed at a distance of 1.50 m from the sample to acquire images. Beam spot sizes of about 50×50 nm^2 for lenses made of PS6056 have been reconstructed. Figure 1 shows an example of the reconstructed and propagated amplitude in the focal plane. We have increased the precision of the relative alignment of both individual MLLs compared to previous experiments leading to decreased side lobes of the focus.

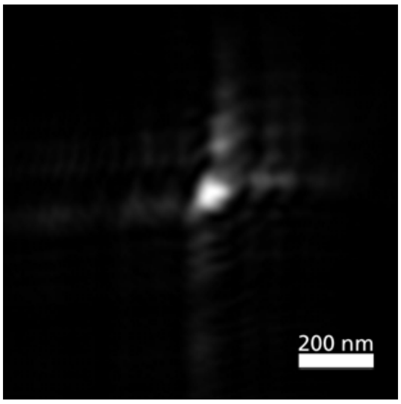


Figure 1: Amplitude of the beam within the reconstructed focal plane.

Further measurements were made testing a type of wedged MLL. This is an improved geometry of the MLL, where every zone is tilted with respect to the optical axis according to the local Bragg condition [Conley2008]. So far, MLLs have only been studied experimentally in tilted geometry, where the entire stack is tilted and only a small part of the zones may comply with their respective Bragg angle.

Tilting series of the lenses have been performed and the diffraction patterns have been acquired by a PCO high resolution x-ray camera at a distance of about four times the focal length. Several lenses were made from deposition PS6359 using different parameters to realize the wedged geometry. A MLL with a flat design acts as reference. Hence, diffraction patterns show the same characteristics except for changes induced by the wedged design. A comparison of the local diffraction efficiencies shows an increased overall diffraction efficiency by about 50 %. A remarkable increase of about 200 % of the local diffraction efficiency was achieved particularly for the smaller zones having zones widths around 5.5 nm (see figure 2).

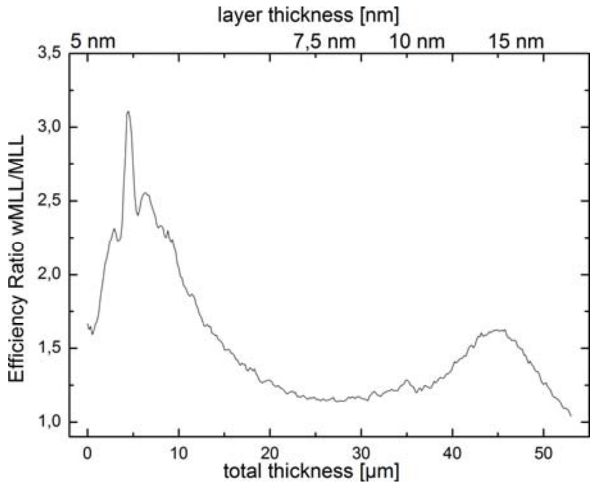


Figure 2: Increase in local diffraction efficiency from tilted to wedged MLL.

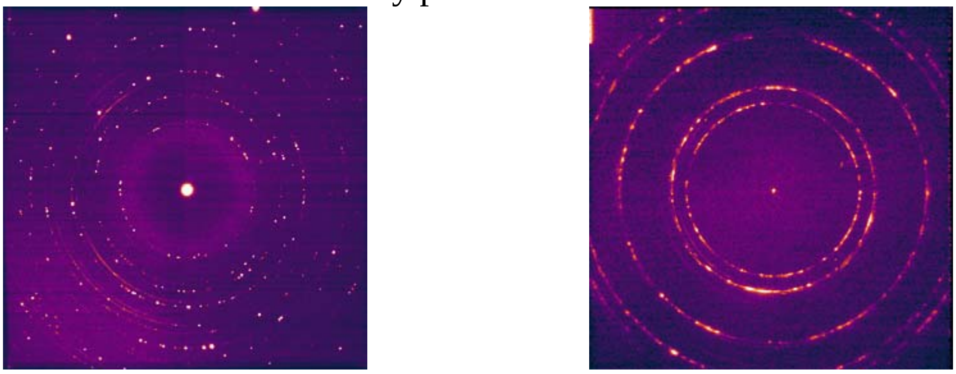
X-ray holography

Diffraction patterns for a reconstruction method based on holography [Lubk2013] have been acquired along with the ptychographic measurements. Further enhancement of the analysis of this method is intended to lead to an additional approach of beam characterization of x-ray optics. The data will be processed by the Triebenberg Laboratory of the Institute of Structural Physics of the Technische Universität Dresden.

Demonstration of nano x-ray diffraction measurements with MLL

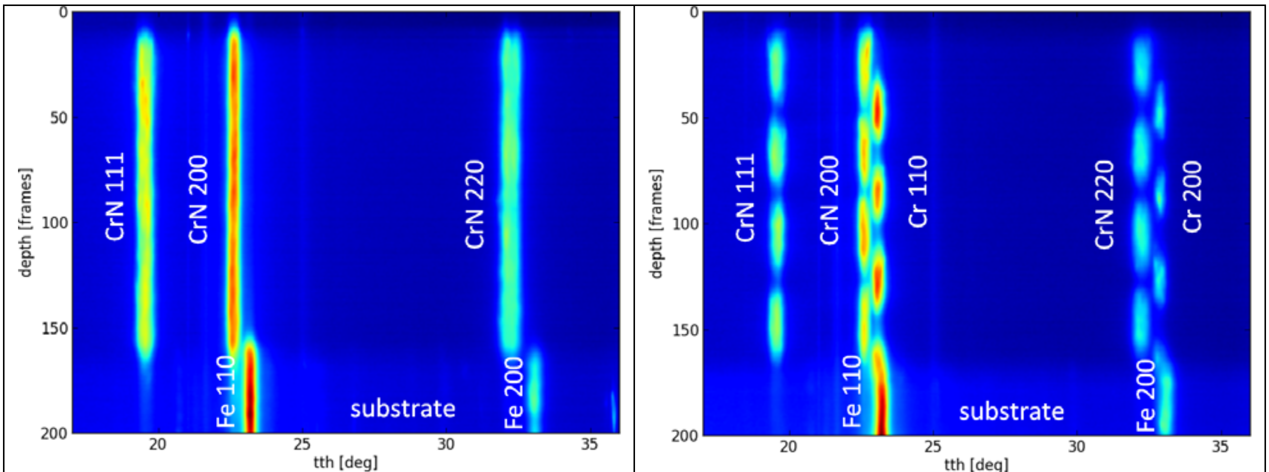
The experimental set-up of ID 13 already allows x-ray diffraction studies with high spatial resolution using the focus of KB mirrors, a Fresnel zone plate or a pair of nanofocusing lenses (NFL) as a small probe. E.g., this kind of measurement can be used to obtain local strain mapping. As crossed MLLs allow for smaller focal sizes, an enhanced spatial resolution can be expected applying those lenses. Some beam time was used to study the capabilities on a test sample. A suitable specimen was kindly provided by the group of Prof. Dr. J. Keckes, Montanuniversität Leoben, Austria, which recently studied hard coatings at ID13 with focal sizes of 250 nm and 100 nm [Keckes2012].
The experimental set-up was modified in comparison to the configuration used for ptychography, using the pair of crossed MLL made from PS6056 at 15.25 keV: The far field detector was replaced by the FReLoN CCD camera and an additional x-ray shielding was installed to suppress scattered radiation originating from the slit system. A beam stop

downstream the specimen blocks direct light onto the detector. The distance from the x-ray focus to the center of the detector was determined to be 70.45 mm. Basic calibration of the system was done with an Al_2O_3 powder. Vertical line scans with a length of 10 µm at a step size of 20 nm across the multilayer of the hard coating have been acquired at different positions. An example of a pattern for each specimen is shown in fig. 3. Debye-Scherrer rings are clearly visible. The evaluation of this data is currently in progress. The next paragraph summarizes initial results of the evaluation by the group in Leoben. An effect of the enhanced resolution might not be seen here, because the multilayer specimen was just coarsely aligned with respect to the optical axis due to time constraints, i.e. the focused beam and the original surface of the substrate are not sufficiently parallel.



(a) Al_2O_3 reference sample (b) CrN Hard coating

Figure 3: Nano-XRD patterns acquired with the FReLoN CCD detector.



(a) CrN monolayer (b) CrN-Cr multilayer

Figure 4: Depth-resolved diffraction scans from 3µm thick CrN monolayer (left) and from CrN-Cr multilayer (with 0.5 µm CrN and 0.25 µm Cr sub-layers) (right) deposited on steel. The data were obtained by an 360° azimuthal integration of the respective Debye-Scherrer rings.

Diffraction data from 3 µm thick monolithic CrN and multilayered CrN/Cr coatings on steel were collected with a spatial resolution of 50 nm are presented in fig. 4. Up to now, it was not possible to characterize such thin structures and resolve individual sublayers as it was done in the CrN/Cr sample using the present setup. The overlap of the reflections the CrN and Cr reflections in Fig. 4 (b) is partly due to the fact that the interfaces were not parallel to the incident beam. A better alignment would allow a more distinct separation of the individual sub-layers; this is however a very time consuming procedure. The results indicate a presence of residual stress in CrN phase in both coatings which is reflected by the CrN 220 reflection splitting. In the next step the data will be further treated in order to evaluate depth profiles of residual stresses in the individual phases and the evolution FWHM as a function of the azimuthal angle and depth. It is expected that the final results will provide very valuable information on the evolution of microstructure and strains during magnetron sputtering of thin films and coatings.

Summary

We have demonstrated a stable setup of a crossed pair of Multilayer Laue Lenses, which was used to conduct nanodiffraction experiment with hard coatings. A wedged MLL was tested and demonstrated increased local and global diffraction efficiency compared to an MLL in tilted geometry MLL.

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