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Multilayer Laue Lenses (MLL) with 45 mm Focal Length as Optics for In-situ Nanoindentation Experiments

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In the past decade research activities on Multilayer Laue Lenses (MLLs) have shown encouraging results in hard X-ray microscopy. Calculations have shown their potential to reach diffraction limited resolutions in the order of 1 nm [1, 2].

The increasing demand of in-situ and operando experiments require working distances in X-ray microscopes of at least several millimeters and up to several centimeters. This space becomes necessary to contain the experimental setup, stages and chambers as well as the samples. We have developed a monolithic 2D focusing MLL optics based on two crossed MLLs with low stress multilayers of the Mo/C/Si/C system [3, 4]. This multilayer configuration allows to deposit multilayer stacks with a total thicknesses of up to 100 µm. With this large aperture we designed an MLL with an extraordinary long focal length and a corresponding long working distance of several centimeters and diffraction limited focal spot sizes well below 100 nm. A measurement with an MLL based on this multilayer system has demonstrated the capability to realize high diffraction efficiencies close to theoretical predictions [5].

The design of the long focal length MLLs is based on the Fresnel zone plate law and contains zones 4000 to 12500 with a focal length of 45 mm at an X-ray energy of 12 keV with equivalent zone widths between 9.6 nm and 17 nm. This results in a total thickness of slightly more than 100 µm. Figure 1 shows a cross section SEM image of a FIB (focused ion beam) milled lamella. Working distance, i.e. space between the order sorting pinhole and the focus position, is for this type of MLL in the order of 25 mm at an X-ray energy of 12 keV. The diffraction limited focal spot size of this design is in the order of 50 nm, which is sufficient for many experimental setups and techniques. First focusing measurements with lenses based on this design have been successfully performed at the ESRF beamline ID13. The mounting of the monolithic crossed MLL housing, the pinhole and the sample is shown in Figure 2 in their respective positions as they were used for the measurement.

This ESRF ID13 setup was also used to perform in-situ indentation measurements on 15 µm CrN thin film on Si(100) substrate, which was loaded stepwise using a wedge diamond indenter [6]. At each indentation step, WAXS frames were collected using an Eiger 4M detector. The data were used to evaluate multiaxial stress fields in the loaded film as a function of the applied load. In Figure 3a, a scanning electron micrograph from the film cross-section showing the indenter imprint with a few cross-sectional cracks is presented. In Figure 3b, a cross-sectional distribution of in-plane stresses evaluated for the indenter load of 1 N can be seen. The resolution was limited by the roughness of the interfaces as well as the temporary setup in the first iteration of these measurements. The results indicate very complex variation of compressive intrinsic stresses with a maximum in the film center, caused by the

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variation of process parameters during the film deposition. Due to the indentation, additional anisotropic stress is initiated into the film, whose form and magnitude correlate with the cracks shown in Figure 3a. The in-situ approach allows to reveal microstructure origins of the indentation-induced cracks in the film and to determine the magnitude of stress responsible for particular crack patterns.

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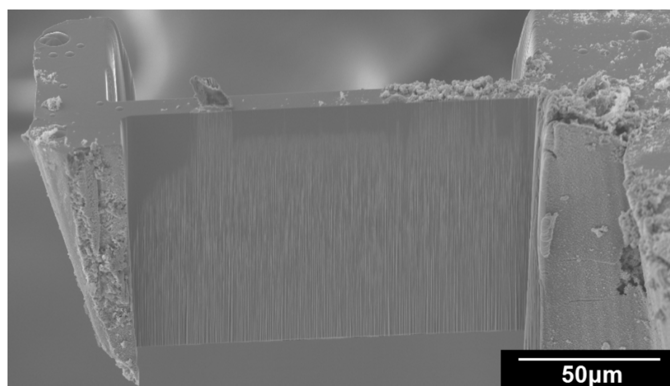


Figure 1. FIB-milled MLL deposition. The lamella is tilted by 37 degrees to the line of sight.

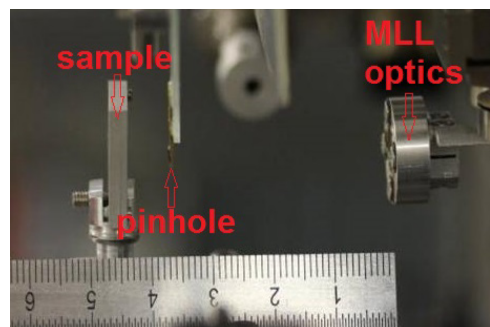
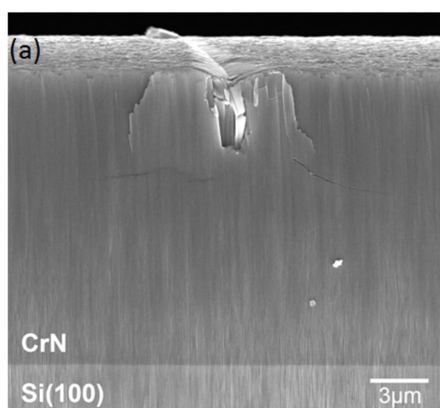


Figure 2. Photograph of the MLL setup with sample in focal plane and an absolute scale. The working distance was limited by the small opening of the available pinhole (diameter approx. 20 µm).



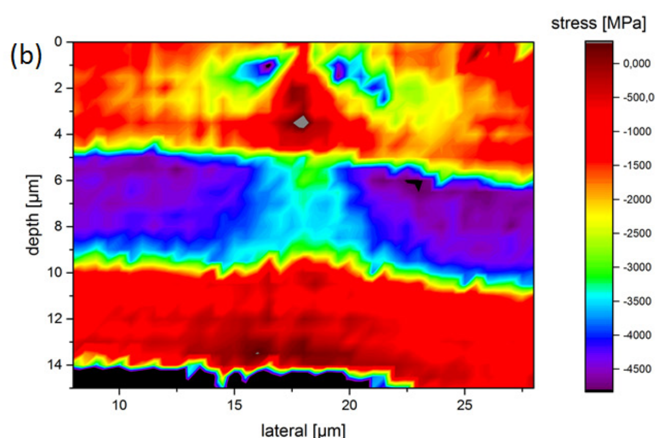


Figure 3. SEM cross-section image of a CrN thin film on Si(100) substrate with cracks formed due to in-situ indentation (a). Cross-section distribution of in-plane stress evaluated from X-ray nanodiffraction data (b)

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