ESRF	**Experiment title: X-ray Microfocussing - part II - applications: study of micron sized systems)**	**Experiment number**: IH-HC-766
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Shifts:	**Local contact(s)**: C. Mocuta	*Received at ESRF:*

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Report:

The microdiffraction setup implemented and tested in the experiment BLC-2175 (X-ray energy of 10keV, focused beam size of 3 x 5 μm^2 (horiozontal x vertical) using a circular Fresnel Zone Plate) has been used to study the diffraction of single micro-objects. In the first test experiment to characterize the microdiffraction possibilities at ID01, Nb microstructures have been studied. Nb(110) on Al_2O_3 is a well-characterized system typically used as buffer layer for the growth of rare earth magnetic layers. Microstructures of these systems are of high interest for applications such as magnetic data storage.

The sample studied in this experiment was grown by molecular beam epitaxy and patterned by electron litography by Arndt Remhof, Ruhr-Universitaet Bochum, Germany (three different areas of 1mm^2 of the sample were patterned with different dot size). They consist of a thin film of 30 nm Nb(110) deposited on Al_2O_3 a-plane and covered by Cr to prevent oxidation. Fig. 1 (a)-(c) shows optical microscopy images of small regions of each patterned area. The sample was mounted on a goniometer head and aligned parallel to the x-ray beam. The patterned areas have been optically prealigned in the center of rotation of the diffractometer using a microscope mounted on top of the sample and focused on the center of rotation of the diffractometer. The exact alignment of the Nb cube has been performed using two translations of the diffractometer parallel and perpendicular to the incoming x-ray beam. Fig. 1(d) and (e) show diffraction images at the Nb(220) Bragg position (θ=31.835°), i.e. x-ray intensity changes with the lateral sample position for the 3 x 3 and 2 x 2 μm^2 cubes. The single 3 x 3 μm^2 cubes are well resolved in both lateral directions, while the 2 x 2 μm^2 cubes are well resolved only along the direction parallel to the incoming beam (x direction). The individual 1 x 1 μm^2 cubes could not be resolved at all. For better comparison, Fig. 1 (f) shows line scans in x direction measured for all samples.

For both the 2 x 2 and the 3 x 3 μm^2 cubes, the cubes in y direction are less well resolved than in x direction, even if the ideal projected beam size on the sample surface (i.e. the illuminated surface area) at Bragg angle would be 5 x 5 μm^2. This is believed to be due to a slight misalignment of the sample, which is slightly out of the focal point. The effective beam size at the sample position is thus about 6 x 11 μm^2. With this beam size, the larger cubes can still be resolved, while the beam covers too many of the 1 x 1 μm^2 dots, and so the scattering of the single dots cannot be separated anymore.

Fig. 2 shows radial and angular scans of the Nb(110) reflection, performed on the non-structured film and the structured areas. The measurements of the 3 x 3 μm^2 and 2 x 2 μm^2 cubes was performed such that

the intensity is dominated by the scattering of one cube, while for the 1 x 1 μm^2 cubes, it averages over several cubes.

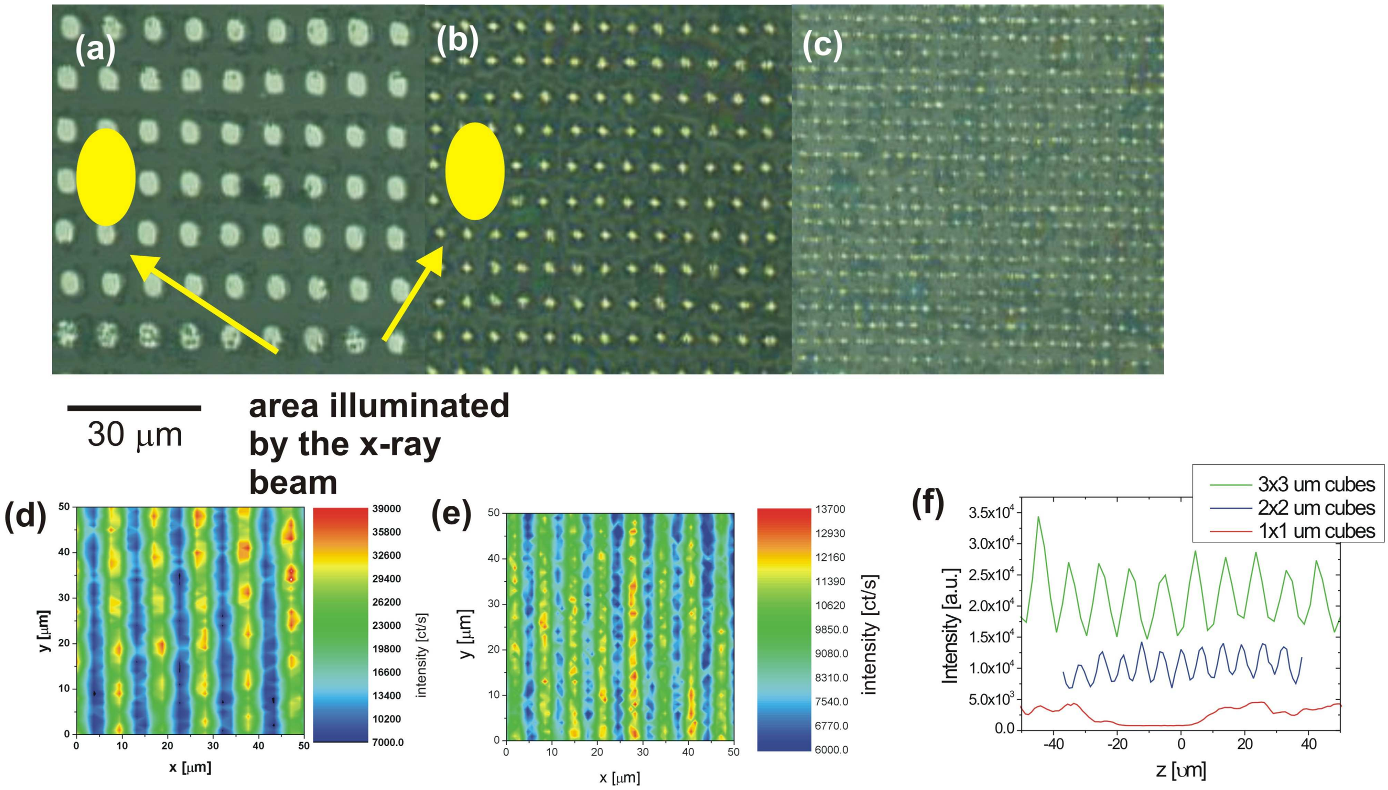

Fig. 1: *(a) -(c) optical microscopy images of the structured Nb areas with object sizes of 3 x 3, 2 x 2, and 1 x 1 μm^2,with a horizontal spacing of 10, 6 and 4 microns respectivly. The x and y directions are parallel,respectively perpendicular to the incoming x-ray beam; x-ray scattering map of the (d) 3 x 3 and (e) 2 x 2 μm^2 areas measured at the Nb(220) reflection, and (f) selected line scans of the areas (see text).*

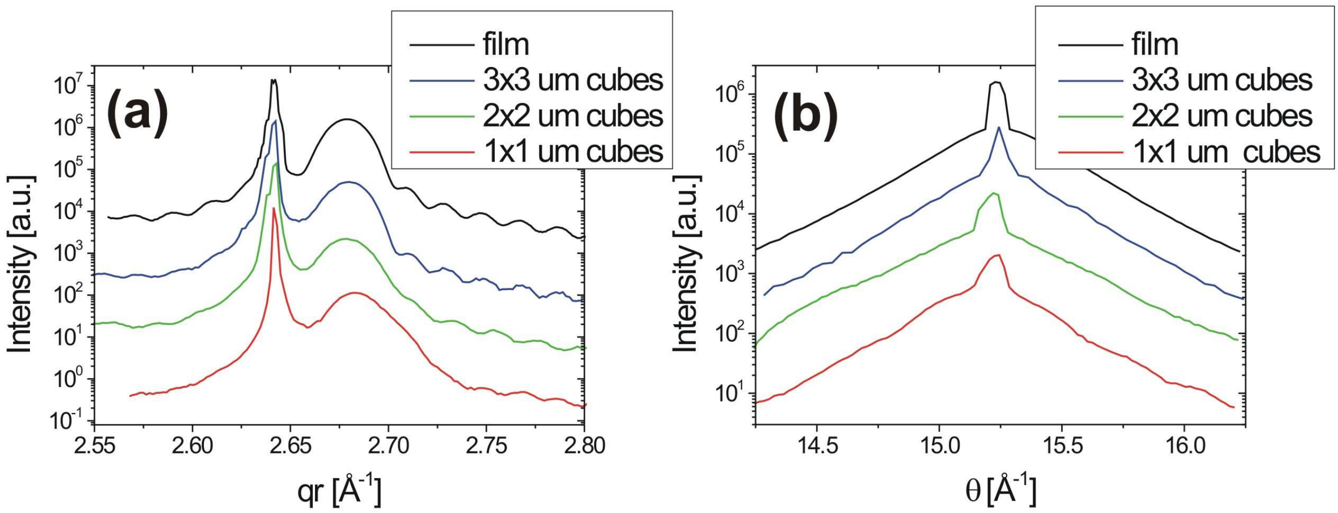

Fig. 2: *(a) Radial- and (b) angular- scans at the Nb(110) reflection. Scans ar performed on the original film and the structured areas.*

The radial scan of the unstructured Nb film is characterized by the sharp sapphire Bragg peak and the broad Nb (110) film peak. The Laue oscillations of the Nb peak indicate the high crystallinity and the smoothness of the Nb film. The 2-component peak observed in the angular scan is characteristic for Nb on sapphire. The sharp component has been attributed to a long-range order of the lateral domains [1.2] The typical shape of the radial and angular scans has also been observed for the structured areas, only with

reduced intensity due to the reduction of the amount of scattering material. Only for the 1 x 1 μm^2 cubes a deformation of the Bragg peak has been observed in the radial scan. This has been attributed to the worse quality of these structures; their size is close to the limit of the structuring method and are thus more irregular than the larger cubes. Additionally, a slight shift of the illuminated area during the scan (of the order of 1 μm) might contribute to the deformation of the Bragg peak. Such a shift is estimated following the alignment of the sample in the center of rotation of the diffractometer within 50 μm.

Conclusion:

With the setup used for this micro-diffraction test experiment at ID01, the diffraction of single micro-objects of 3 x 3 μm^2 size and 10 μm distance separation can be well resolved. The scattering of 2 x 2 μm objects at 6 μm distance from each other can also be resolved, but in this case a strong overlap of the scattering of neighbouring dots in direction perpendicular to the incoming x-ray beam has been observed. The scattering from 1x1 μm objects with a distance of 4 μm cannot be separated.

The diffraction of objects of micrometer size can be measured at ID01 provided that they are separated by at least 5-6 μm. Further improvement of the beam size, stability, and the positioning of the objects (e.g. the reproducibility of the motors used for the prealignment of the sample) are necessary in order to extend the experimental possibilities at ID01 to smaller objects or a higher object density.

References:

[1] Gibaud et al, J. Phys.: Condens. Matter **7** (1995) 2645-2654
[2] Wolfing et al, J. Phys.: Condens. Matter **11** (1999) 2669-2678