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Nanodiffraction at MINAXS (P03) beamline of PETRA III

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Abstract. The Nanofocus endstation of MINAXS (Micro- and Nanofocus X-ray Scattering, also referred to as P03) beamline at PETRAIII of DESY (Hamburg, Germany) has been equipped and is operated by the University of Kiel in the framework of BMBF-Verbundforschung funded by the German federal ministry of research. It was put in user operation beginning of 2012 and it provides a nanobeam generated by using two-dimensionally confining X-ray waveguides or elliptically curved mirrors in crossed geometry (KB mirrors). for nanodiffraction experiments. Sample experimental data for both nanobeam types is shown.

1. Nanodiffraction endstation of MINAXS beamline

The Micro- and Nanofocus X-ray Scattering beamline (MINAXS, also referred to as P03) is equipped with two consecutively arranged experimental endstations: the Microfocus endstation [1, 2] and the Nanofocus endstation [3]. The Nanofocus endstation described here begins at 97 m from the undulator source and is the last part of the beamline. The setup at this endstation was developed and installed within a cooperation between the University of Kiel and DESY and it is operated by the University of Kiel. The design objective of the Nanofocus endstation is to routinely provide a monochromatic X-ray beam with a cross section of about 100 nm for nanodiffraction experiments, i.e. small-angle and wide-angle X-ray scattering measurements (SAXS and WAXS) with high spatial resolution (< 100 nm) at an energy range of 8 to 23 keV. Originally 2D X-ray waveguides were intended to be the nanobeam defining elements. Meanwhile a set of nanofocusing mirrors (Kirkpatrick-Baez mirrors, KB-mirrors) is also available for high-flux nanobeam generation in user experiments. A schematic layout of the MINAXS beamline showing the position of the Nanofocus endstation is displayed in figure 1 a). Two concepts to generate the nanobeam at the Nanofocus endstation are presented.

The first one is based on two-dimensionally confining X-ray waveguides [4] and is performed in a two-step process. First, the source is imaged onto the waveguide position using compound refractive lens (CRL) transfocators positioned at 77 m and 82 m, respectively. The so generated focal spot illuminates the input aperture of the X-ray waveguide and the exiting nanocollimated, spatially and coherently filtered beam is used for experiments. This concept is shown schematically in figure 1 b) and it is the method used for the first nanodiffraction experiments performed at the Nanofocus endstation. The vertical beam size at closest sample position was around 450 nm (vertical divergence of 1.4 mrad) out of a 2D waveguide with a cross-section of 50µm × 50 nm (horizontal × vertical) and the total flux in the so generated beam was 3×10^7 /sec [3]. The X-ray waveguides were fabricated and provided by the group of Prof. Tim Salditt, University of Göttingen [5].

The second concept – again a two-step process – uses superpolished, nanofocusing KB-mirrors [6]. The focal spot is a demagnified image of the intermediate source generated using the CRLs in a short focal length lens configuration. As the intermediate source is smaller than the undulatur source a decreased demagnification ratio for the mirror system is obtained. If the undulator source was imaged directly the smallest beam size at a working distance of 100 mm would be about 1 µm. Using the intermediate source concept, however, a spot size of 100 nm × 200 nm is achievable at the same large working distance, required to accommodate extended sample environments. Each 100 mm long mirror has a shape error of 0.9 nm (peak-to-valley) and a roughness of 0.1 nm (rms). The mirror system, fabricated by JTEC of Osaka, Japan is currently in its final stage of commissioning, and a spot size of 370 × 760 nm² was achieved at a total flux of 4 × 10^9 /sec, while a beam size of 1.5 × 1.5 µm² is routinely available at a total flux of 3 × 10^{10} /sec. Final improvement of the mirror alignment is ongoing and a beam position feedback system will be installed in order to compensate for the fluctuations of incident beam position, currently constricting the optimum mirror alignment.

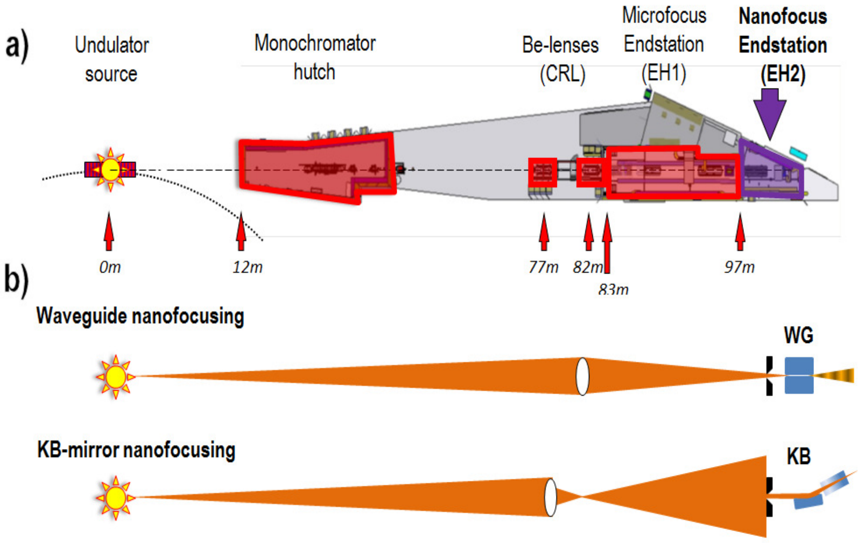
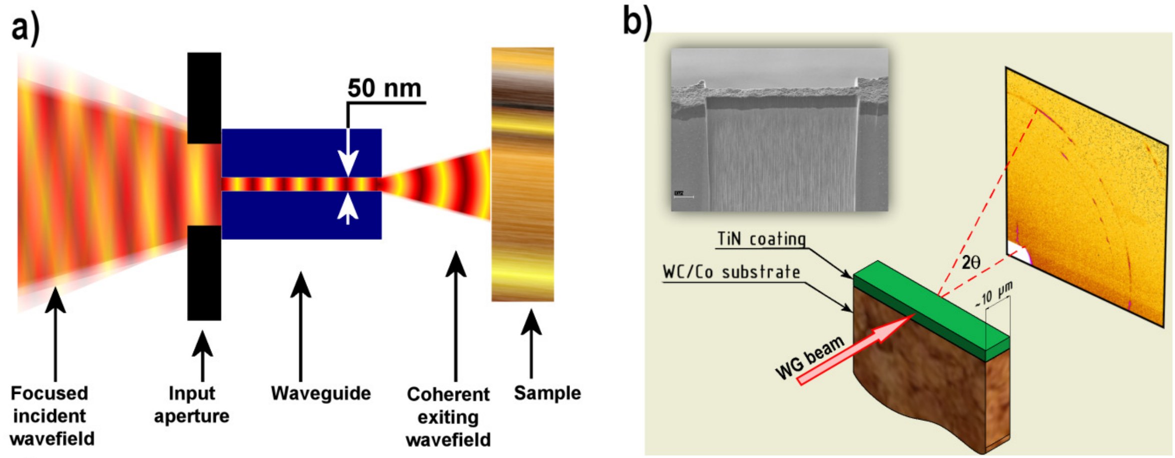


Figure 1. a) Schematic layout of the MINAXS beamline showing the arrangement of its two experimental endstations, EH2 being the nanofocus endstation. **b)** For nanofocusing at EH2 two focusing geometries exist, each using Beryllium lenses for pre-focusing or creating a virtual source. The nanobeam is then generated with a X-ray waveguide (WG) or a Kirkpatrick-Baez mirror system (KB), respectively.

2. First nanodiffraction experiments

2.1. *Nanodiffraction with 2D X-ray waveguides*

The first nanodiffraction data using a 2D-waveguided nanobeam was recorded from a nanocrystalline TiN coating as used in protective hard coatings on metal cutting and turning tools. In these coatings unfavorable tensile in-plane residual stresses occur and can be turned into compressive ones by using post-deposition surface treatment like blasting in order to increase lifetime and performance of the coated tools. The aim of the experiment was to verify the resolution of the setup and to reveal the local gradients across the blasted coating. A 6 µm coating was deposited on WC/Co substrates using a CVD process and consequently blasted using Al_2O_3 particles. A 10 µm thick slice was prepared perpendicular to the interface and scanning nanodiffraction data were recorded in transmission WAXS geometry (12.8 keV beam energy) by scanning the waveguide beam vertically across the slice with a step size of 200 nm. The sample was positioned at a distance of about 100 µm from the exit of a 50 µm × 50 nm waveguide, giving a horizontal beam size of 430 nm at the sample position. The setup is shown schematically in figure 2 a-b). In figure 2 c) the distribution of the experimentally observed Debye-Scherrer ring intensities as a function of the diffraction angle and of the vertical scanning position is shown in a compositional image. At depths down to about 7 µm TiN (111) and TiN (200) reflections with varying intensities indicate a presence of a strong crystallographic texture in the coating. The relative changes of the peak positions (marked by arrows) indicate the presence of compressive strains close to the blasted coating surface. Although the coating was thinned to a 10 µm slice and a part of the macroscopic (I order) strains probably relaxed, the changes in the peak positions indicate that a portion of strain was still preserved. Further details can be found in [3].



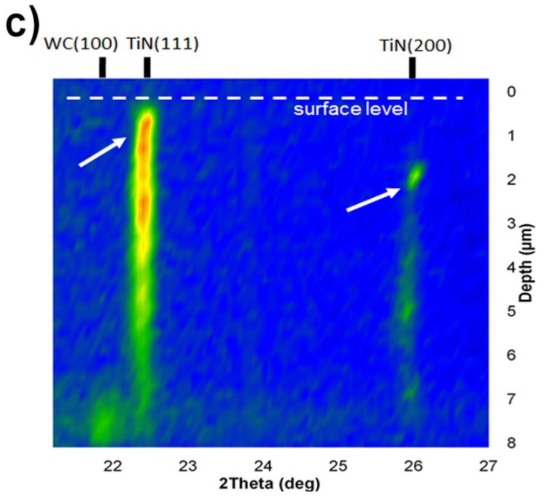


Figure 2. a) Schematic layout of an X-ray waveguide based diffraction experiment: the prefocused X-ray wavefield is coupled into the waveguide, a spatially and coherently filtered wavefield propagates along the waveguide and the exiting beam illuminates a sample placed closely (typically 100 µm) to the waveguide exit. **b)** Nanodiffraction experiment on a TiN coating deposited on a WC-Co substrate (TEM image of the sample in inset) **c)** Compositional image of the Debye-Scherrer ring intensities recorded in the nanodiffraction experiment indicating the presence of a strain gradient within the microstructure of the coating. [3]

2.2. *Nanodiffraction with KB-mirror optics*

Two cross-sections of Norway spruce with a thickness of 17 µm were prepared by micro-cryotome and focused ion beam milling techniques. The aim was to gain first experimental values with weakly scattering material and structural details of the cell wall with high spatial resolution. The samples were scanned with a nanobeam of 370 nm by 760 nm parallel to the cell wall axis with a step size of 500 nm in two-dimensional meshes. Each diffraction image contains local information on the structural properties of the cellulose fibrils, which are different in each cell wall layer and which determine the mechanical stability. From the recorded data e.g. the content of oriented cellulose could be determined, as shown in Fig. 3. The data analysis also showed that despite the not yet finalized mirror alignment different cell wall layer were clearly resolved.

3. Conclusion and future plans

Nanodiffraction data recorded at the Nanofocus Endstation of MINAXS is shown. A 2D-waveguide beam was used on a TiN coating on WC-Co substrate revealing strain gradients across the coating with sub-micron resolution. In future, smaller waveguides with a symmetric cross section will be used because of the ongoing implementation of a KB-mirror optic. Providing a nanofocused beam on its own (target value 100 nm by 200 nm) the mirror optics will also enable the use of waveguides with symmetrical sub 100 nm cross sections. First nanodiffraction data using the KB nanobeam were also successfully recorded from weakly scattering thin sections single wood cell walls using a beam size of 370 by 760 nm. In the near future nanodiffraction experiments will be combined with various in-situ parameters (tension, pressure, temperature) and in grazing incidence geometry.

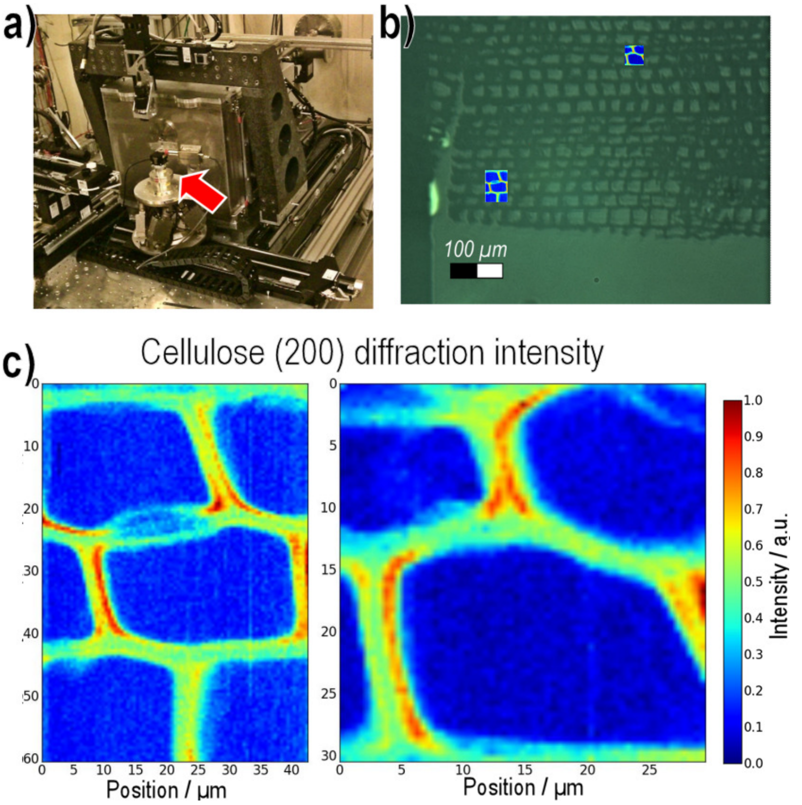


Figure 3. a) Photograph of the KB-mirror setup. The arrow indicates the sample position, mounted on top of a precise hexapod. The KB mirror system is included in the steel tank behind the sample. **b)** Microscope image of a thin cross-section of wood prepared by cryotome and focused ion beam milling techniques. The image was recorded in situ with the in-line microscope. The microscope image is superimposed with the regions that were scanned in the experiment. **c)** The nanobeam was scanned across the parallel aligned cell walls with a step size of 500 nm and the information contained in each diffraction image was used to map the cellulose content (shown here) in the different cell wall layers.

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