A 2D waveguide beam for X-ray nano-diffraction

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The Micro- and Nanofocus X-ray Scattering (MINAXS) beamline of PETRA III is equipped with two consecutively arranged endstations, the last of which is the Nanofocus endstation [1]. First in-beam commissioning of the experimental equipment was successfully performed end of 2010 using two-dimensionally confining (2D) hard X-ray silicon waveguides with cross-sections of (50 nm × 50 µm) to (50 nm × 2 µm) for nanobeam generation. The first high resolution nanodiffraction experiment on a nanocrystalline TiN hard coating was performed in April 2011 to verify the resolution of the nanodiffraction setup and to reveal the local gradients across the blasted TiN coating [2]. In this experiment a beam out of a 2-dimensionally confining waveguide was used for diffraction experiments for the first time. The method used to generate the sub-µm beam is based on X-ray waveguides and is performed in a two-step process. First, the source is imaged onto the waveguide position using compound refractive lenses (CRL) installed in two lens exchangers ca. 20 m upstream of the endstation. The so generated focal spot is then coupled into a 2D X-ray waveguide by front-illuminating its entrance and the nanocollimated beam exiting the waveguide is used for experiments [2,3]. The high width-to-height aspect ratio of the waveguide cross-section was chosen to compensate for the high over-illumination of the waveguides by the (at the time) relatively large size of the prefocused beam. A vertical beam size of 237 nm was measured at the exit of the smallest waveguide with a vertical beam divergence of 1.4 mrad. A scheme of the most important components constituting the experimental setup at the Nanofocus endstation is shown in figure 1. The positioning of both, the waveguide and the sample is each based on a hexapod, giving the ability of tuning all 6 degrees of freedom (3 linear, 3 rotational) with only one device. Both hexapods are equipped with top-mounted 2D-piezo-positioners providing a translational reproducibility of down to 10 nm. In order to perform a high resolution X-ray diffraction experiment a flat, thin sample is placed normal to the beam direction and very closely to the exit of the waveguide (typical distance <100 µm). It is then scanned across the waveguide beam while diffraction images are recorded by means of a 2D detector.

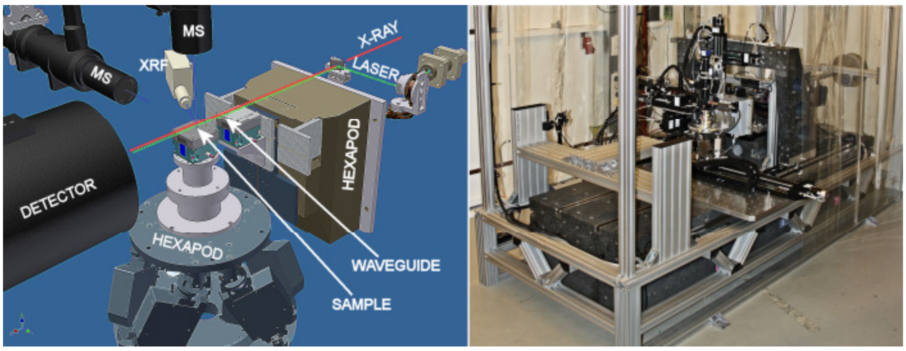

Figure 1: Left: schematic arrangement of the most important components that constitute the experimental setup at the Nanofocus endstation (MS = microscope, XRF = X-ray fluorescence detector). Owing to their small size, the positions of the waveguide and of the sample are only indicated with arrows. Right: photograph of the current setup at the Nanofocus endstation of the MINAXS beamline. The size of the granite table is 2.5 m by 0.8 m. The beam direction is from right to left and the detector is removed for a better visibility of the setup.

The aim of the experiment reported here was to verify the resolution of the nano-diffraction setup and to reveal the local gradients across the blasted, nanocrystalline TiN coating. TiN coatings on WC/Co substrates deposited using chemical vapor deposition (CVD) possess usually not-favorable tensile in plane residual stresses which can be turned to compressive ones by using post-deposition surface treatment like blasting (Klaus et al., Bartosik et al.). A polycrystalline ca. 7 µm thick TiN coating was deposited on WC/Co substrates using a chemical vapour deposition process and was blasted subsequently using Al2O3 particles with a diameter of approximately 50 µm. From the coating/substrate composite, a 10 µm thick slice was prepared by cutting the composite perpendicular to the interface, by mechanical polishing using a diamond saw and finally by focused-ion beam milling. The slice was characterized in transmission WAXS geometry (energy 13 keV) by scanning the waveguide beam vertically across the horizontally aligned coating and the substrate using a vertical step size of 0.2 µm. The sample was positioned at a distance of about 100 µm from the 50 µm × 50 nm waveguide. For every measurement step, one quadrant of a Debye-Scherrer ring was collected at the sample-detector distance of 120.4 mm. In figure 2 the distribution of the experimentally observed intensities as a function of the diffraction angle and the vertical scanning position is presented in a compositional image constructed from the individual Debye-Scherrer rings. At vertical positions down to about 7.5 µm, one can recognize TiN (111) and TiN (200) reflections. The varying intensity along the TiN reflections indicates a presence of a strong crystallographic texture in the coating. The relative changes of the peak positions (marked by arrows) can be interpreted as the presence of compressive strains which are obvious especially close to the blasted coating surface. Though 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 in the coating. At vertical positions of about 7.5-8 µm, a WC (100) reflection can be observed together with a higher fluorescence background.

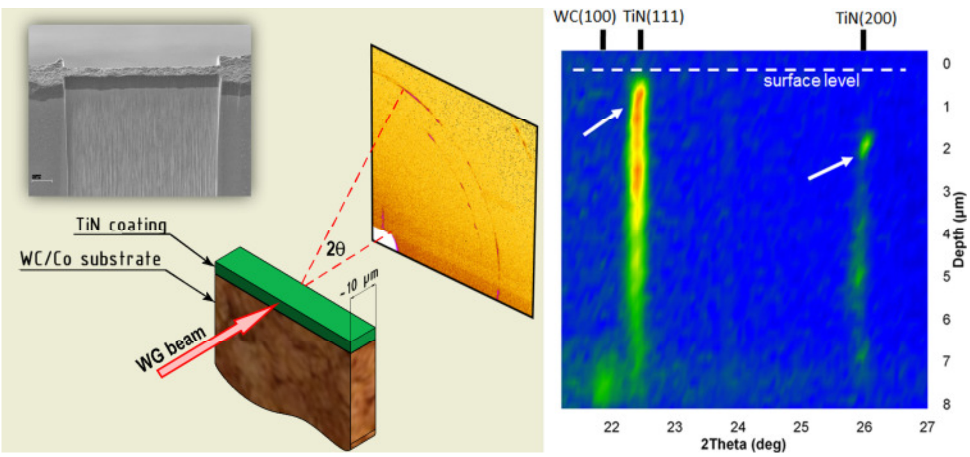

Figure 2: Left: schematic setup of the nanodiffraction experiment on a TiN coating deposited on a WC-Co substrate (inset shows a TEM image of the actual sample). Right: compositional image of the experimentally observed intensities recorded in the nanodiffraction experiment. The intensity modulations of the TiN (111)/(200) and substrate WC (100) reflections indicate the presence of a strain gradient within the microstructure of the coating.

In future, smaller waveguides with a less elongated cross-section, i.e. a width-to-height aspect ratio close to 1 can be used because of the currently ongoing implementation of a Kirkpatrick-Baez mirror optics at the Nanofocus. The authors would like to thank the MINAXS beamline technician Kai Stassig (DESY) for his support throughout the construction phase of the MINAXS Nanofocus endstation, Thorsten Kracht, Maria-Teresa Nunez-Pardo-de-Vera and André Rothkirch of DESY IT-group for continuously great help. The authors also thank the German Federal Ministry of Education and Research (BMBF) for the funding within the “Verbundforschung” grants 05KS7FK3 and 05K10FK3.

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