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X-ray nanodiffraction characterization of strains and microstructure in nanostructured thin films

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Nanocrystalline thin films exhibit typically inhomogeneous depth gradients of microstructure, strain and physical properties varying at the nanoscale. Currently it is not trivial to reveal how those gradients relate to the macroscopic film functional properties. One of the main reasons is the lack of experimental techniques which can provide thin film thickness-dependent data with sub-micron resolution.

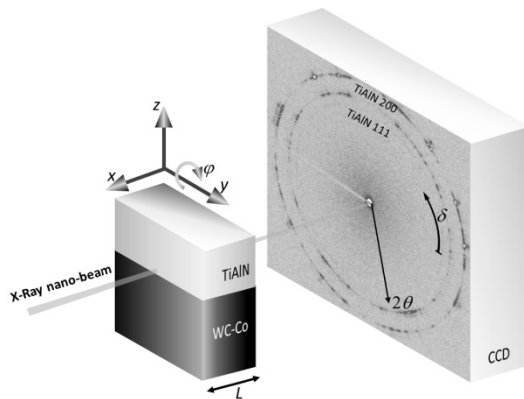


Figure 1. A schematic setup of the cross-sectional X-ray nanodiffraction experiment carried out using a monochromatic X-ray beam. The thin film on the substrate (with the thickness $L=50\ \mu\text{m}$ in the beam direction) was moved along the y axis with a step of 100 nm and the diffraction data were collected using a CCD detector.

In order to reveal gradients of phases, crystallographic texture, crystallite size and strains in thin films, we have developed a cross-sectional synchrotron X-ray nanodiffraction technique (Fig. 1) which operates with the X-ray beam diameters down to 50 nm [1-3]. The aim of this contribution is to discuss the methodological aspects of the new approach and its applications at the beamlines ID13 at ESRF in Grenoble and at P03 at Petra III in Hamburg. On the examples of

hard nitride and metallic thin films (Fig. 2), it will be demonstrated that the new approach can serve as an effective tool to characterize the inhomogeneous thin film properties with 50nm resolution. In this way it is possible to correlate the gradient properties of thin films with the deposition conditions and functional behavior.

Additionally, results from in-situ X-ray nanodiffraction coupled with indentation will be presented. The new in-situ design is suitable to reveal microstructural changes and stress behavior under the indenter in nanomaterials. In this way, it is possible to study deformation behavior in materials with size effects.

Additionally, results from mechanical tests obtained from bending experiments on micro-cantilevers performed in scanning and transmission electron microscopes will be presented to illustrate variability and anisotropy of mechanical properties in graded nanostructured films. The results will be correlated with the X-ray nanodiffraction data. Finally, it will be demonstrated that both complementary approaches can serve as an effective tool to analyze depth gradients of structure-property relationship in nanocrystalline thin films.

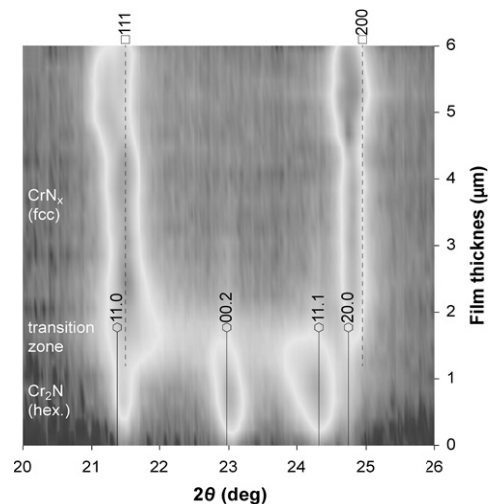


Figure 2. A result from the cross-sectional X-ray nanodiffraction experiment on a graded CrN_x $6\ \mu\text{m}$ thin film indicates the distribution of phases as a function of the depth.

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