ESRF	**Experiment title:** First Real Space Characterization of Residual Stress Depth Profiles in Nanocrystalline Coatings	**Experiment number**: MA-368
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Preliminary Report – 4 Months after the Experiment:

The aim of the experiment was to characterize simultaneously structural and residual stress-depth gradients in nanocrystalline coatings. For that purpose, the users prepared CrN, TiAlN, TiCN, CrAlN model coatings on Si(100) and steel substrates using magnetron sputtering. The crystal size in the coatings was in the range 5-70 nm. The coating/substrates composites were cut into small sections using FIB technique with a resulting thickness of about 20µm.

The samples were characterized in transmission using scanning WAXS (see Fig. 1). The beam was directed perpendicular to the interface and the samples were moved in the beam using x-y-ϕ motors (Fig. 1).

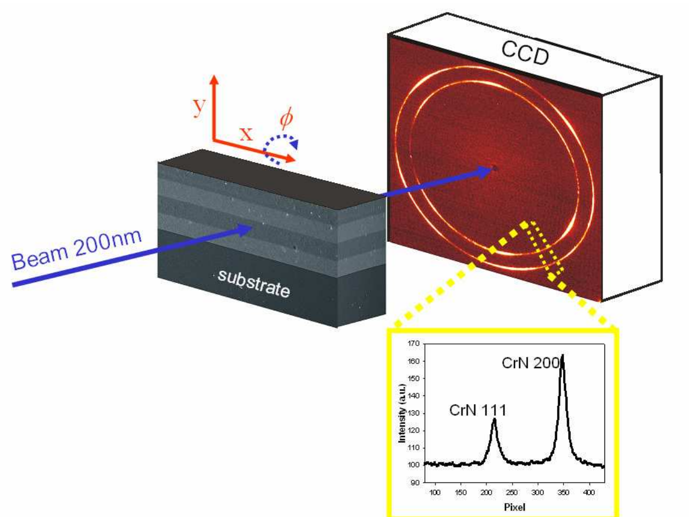

Fig. 1 A schematic sketch of a scanning WAXS setup used for the characterization of depth-gradients in a multilayered coating.

By scanning the samples across the cross-section, it was possible to collect Debye-Scherrer rings and evaluate peak position, peak broadening and intensity as a function of coating depth.

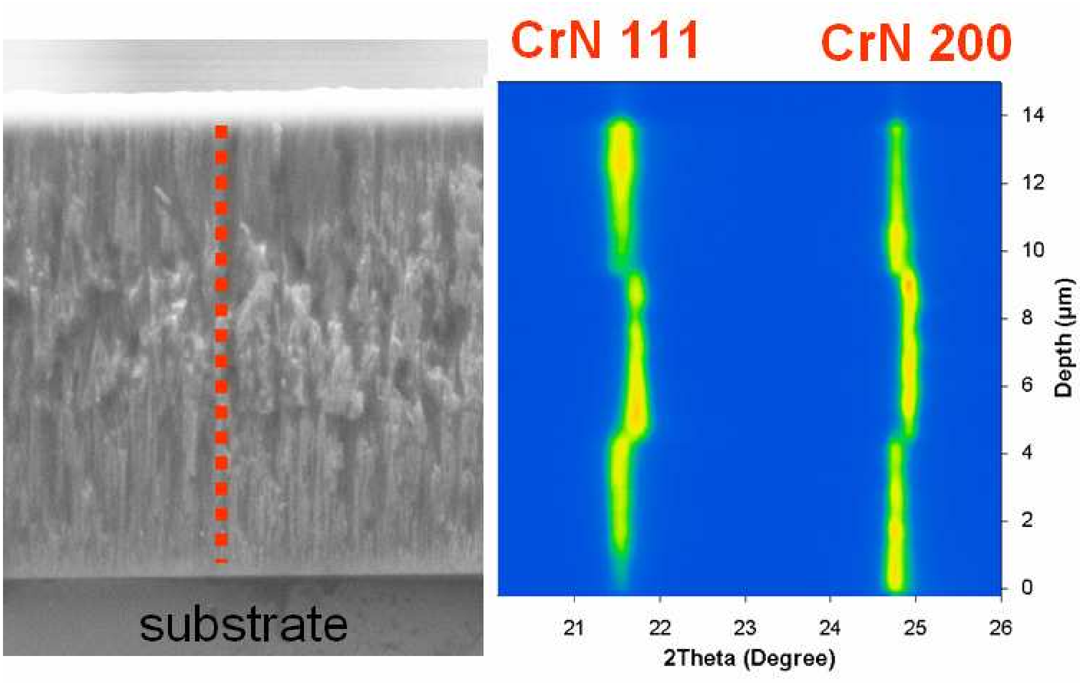

Fig. 2 Positions of CrN 2θ reflections as a function of coating depth evaluated from Debye-Scherrer rings.

In Fig. 2, an example of the peak position analysis in CrN graded nanostructure with a thickness 15µm is presented. The coating was prepared by varying bias voltage during the deposition applying -40, -120 and -40V for every 5µm thick "sublayer". The aim was to check the sensitivity of the synchrotron approach. The results (Fig. 2) demonstrate that using the position-resolved WAXS it was possible to resolve how the lattice parameter changes as a function of the depth. The results from Fig. 2 were produced by analyzing a small section of Debye-Scherrer rings. The peak shifts is caused by both the composition changes as well as by the residual stresses.

By analyzing the shape of the Debye-Scherrer rings, it was possible to resolve residual stress-depth gradients. For that purpose, lattice parameter was evaluated from Debye-Scherrer rings as a function azimuthal angle and the stresses were calculated using the $\sin^2\phi$ method. As an example, in Fig. 3 a residual stress profile across the thickness of the CrN coatings is presented. The results document a relatively high compressive stress of about -1.8 GPa at the interface, an exponential decrease of the stress at depths of 15-10µm, a sudden increase of the stress in the range 5-10µm and again a decrease of the stress in the range 5-0µm. It is obvious that the higher bias voltage of -120V caused an increase of the stresses at the depths of 5-10µm. It is interesting that at depths 0-5 and 10-15 µm the residual stress decreases always exponentially and at depth 5-10 µm the stress state seems to be constants. This documents different growth mechanisms in the case application of -40 and -120 V bias.

Since the thickness of the samples (from Figs.1-3) across the beam direction was only 20µm, the residual stress state was modified by FIB machining. For that reason, the users used finite-element modeling to reconstruct residual stress state in the films in order to obtain the original biaxial residual stress-depth profile.

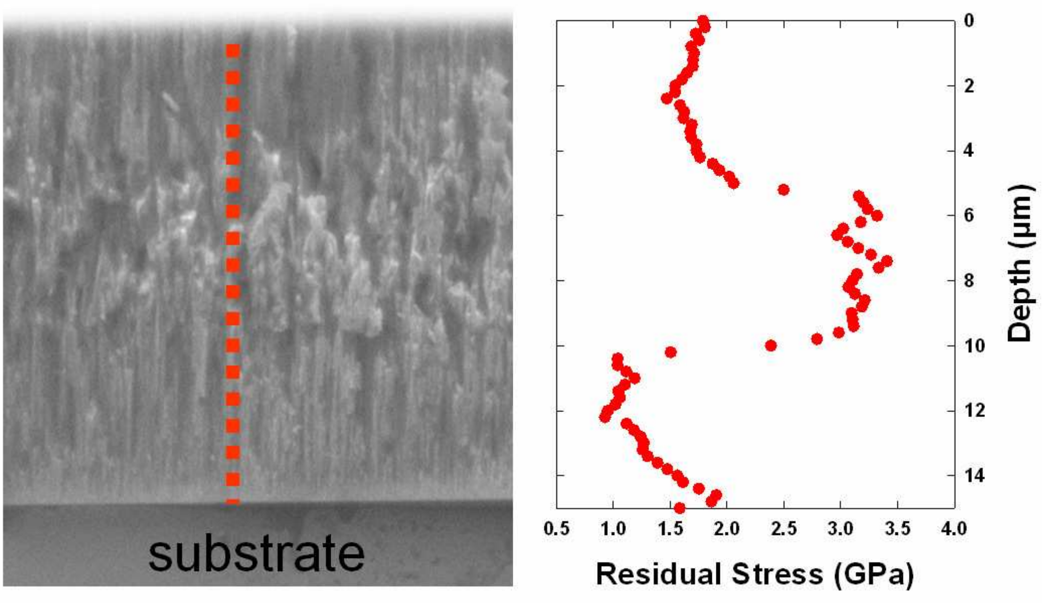

Fig. 3 A residual stress depth-profile across a 15µm thick CrN coatings prepared using varying bias voltage.

In summary, the approach demonstrated above was successfully used to analyze also other multilayered and graded thin films and coatings. The results provided for the first time information about the residual stresses, crystallite sizes and texture as a function of depth. The results will be submitted to high-rank journals in next months.