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Comprehensive Thin Film Analysis by Cross-sectional X-ray Nanodiffraction

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All thin films and engineered surface layers inherently exhibit marked through-thickness property gradients. The complexity of these gradients varies greatly, ranging from the simplest case of a nucleation layer at a substrate interface, to intricately tailored multilayer architectures comprising many different materials, phases and microstructures at multiple levels of hierarchy. In order to understand the overall functional properties of these structures it is of paramount importance to characterize these through-thickness gradients, ultimately making it possible to properly attribute certain aspects of application performance to the time-dependent deposition parameters.

It is the aim of this contribution to demonstrate the comprehensive analytical capabilities of cross-sectional X-ray nanodiffraction (CS-nXRD) for this purpose. Using recent advances in X-ray focussing optics and 2D X-ray detector equipment, it has become possible to scan thin film cross-sections with sub-30nm spatial resolution, while recording the corresponding film thickness-dependent diffraction patterns. Contained within this combination of high resolution real space scanning and extensive reciprocal space analysis is the possibility to access a vast amount of information during each growth stage of a thin film.

Using the example of a TiN – SiO_x multilayered thin film, the various parameters accessible through CS-nXRD will be presented. The film was deposited using magnetically unbalanced reactive pulsed direct current magnetron sputtering from one Si and two oblique Ti targets, alternately switching between them, resulting in a zigzag-like film morphology. The evolution of (I) phase composition, (II) crystallographic texture, (III) grain size, (IV) micro-stress/defect concentration and (V) macro-stress *within each of the sublayers* was characterized and could be attributed to various and time-dependent growth mechanisms, as well as the corresponding deposition conditions.

Further, complementary analysis by electron microscopy and CS-nXRD using *in-situ* sample environments for mechanical and/or thermal loading can provide an even more complete picture, where the nano-scale behavior of thin films during critical application conditions comes into reach of real experimental characterization. For this purpose mechanical testing devices dedicated to *in-situ* CS-nXRD have been developed and various examples will be presented, illustrating the potential of this approach.

Primary author: Dr TODT, Juraj (Erich Schmid Institute for Materials Science, Austrian Academy of Sciences)

Co-authors: Mr MEINDLHUMER, Michael (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben); Mr GRUBER, David (Montanuniversität Leoben); Prof. DANIEL, Rostislav (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at the Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben); Prof. MITTERER, Christian (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at the Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben); Dr BURGHAMMER, Manfred (European Synchrotron Radiation Facility); Dr KRYWKA, Christina (Institute of Materials Research, Helmholtz Zentrum Geesthacht); Prof. KECKES, Jozef (Department of Materials Physics, Montanuniversität Leoben)

Presenter: Dr TODT, Juraj (Erich Schmid Institute for Materials Science, Austrian Academy of Sciences)

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