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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 taylored 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. This contribution aims to demonstrate the comprehensive analytical capabilities of cross-sectional X-ray nanodiffraction (CS-nXRD) for this purpose.

The method relies on the scanning of thin film cross-sections with sub-30nm spatial resolution, while recording the corresponding film thickness-dependent diffraction patterns. 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 DC 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.

Complementary analysis by electron microscopy and CS-nXRD using in-situ sample environments for mechanical and/or thermal loading further enhances the analytical potential and various examples illustrating these method's possibilities will be presented.

Keywords

nanodiffraction
synchrotron XRD
residual stress