Methods for Nanoscale Strain and Stress Mapping in Thin Films

Jozef Keckes*
*jozef.keckes@unileoben.ac.at
Department Material Science, Montanuniversität Leoben, Austria.

Thin films possess complex gradients of residual strains and stresses, which vary laterally and also across film thickness. These originate (i) from self-organized film growth, (ii) from intentionally varying deposition conditions and/or (iii) from inhomogeneous thermal and/or mechanical loads applied during service. In order to optimize functional properties of thin films, it is necessary to apply nanoscale characterization approaches.
In the first part, results from cross-sectional synchrotron X-ray nanodiffraction (CSnanoXRD) [1] will be presented. The experiments were performed at the ID13 beamline of ESRF using monochromatic X-ray beams with a diameter down to ~30 nm in order to analyze *depth gradients* of microstructure and stresses in nanocrystalline thin films. The gradients are correlated with the varying deposition conditions, providing an opportunity to optimize the time-dependent synthesis process and to perform knowledge-based design of the films.
Secondly, results from dark field X-ray microscopy (DFXM) collected at the ID06 beamline of ESRF will show *lateral variation* of strain and mosaicity within individual grains of thermally cycled Cu thin films [2]. It will be shown that the thermal history results in the homogenization of grain interior and strain accumulation at grain boundaries.
Finally, results from the ion-beam layer removal (IRL) method [3] will be used to reveal *stress gradients* across heteroepitaxial and nanocrystalline thin films. Focused-ion beam techniques allow determining stress gradients with a spatial resolution down to ~10 nm and reveal stress concentrations even in amorphous materials.

[1] Keckes, J.; Daniel, R.; Todt, J.; Zalesak, J.; Sartory, B.; Braun, S.; Gluch, J.; Rosenthal, M.; Burghammer, M.; Mitterer, C.; et al. 30 nm X-ray focusing correlates oscillatory stress, texture and structural defect gradients across multilayered TiN-SiOx thin film. *Acta Mater.* **2018**, *144*, 862–873, doi:10.1016/j.actamat.2017.11.049.
[2] Hlushko, K.; Keckes, J.; Ressel, G.; Pörnbacher, J.; Ecker, W.; Kutsal, M.; Cook, P.K.; Detlefs, C.; Yildirim, C. Dark-field X-ray microscopy reveals mosaicity and strain gradients across sub-surface TiC and TiN particles in steel matrix composites. *Scr. Mater.* **2020**, *187*, 402–406, doi:10.1016/J.SCRIPTAMAT.2020.06.053.
[3] Massl, S.; Keckes, J.; Pippan, R. A direct method of determining complex depth profiles of residual stresses in thin films on a nanoscale. *Acta Mater.* **2007**, *55*, 4835–4844, doi:10.1016/J.ACTAMAT.2007.05.002.