

**ULTRATHIN MULTI-ELEMENT LAYER STACKS – A NEW TYPE OF
REFERENCE SAMPLES FOR μ -XRF AND TXRF
(ORAL PRESENTATION)**

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Ideal reference samples for the calibration of micro-XRF set-ups with high sensitivity should have negligible absorption of exciting and fluorescent radiation, a high degree of uniformity and homogeneity and emit various non-overlapping X-ray fluorescence lines over a wide spectral range with suitable intensity. Free standing thin films (FSTF) can meet these requirements. After the discontinuation of NIST SRM 1832/1833, suitable reference materials are no longer commercially available.

Further, the high sensitivity of modern XRF and TXRF instruments leads to a demand of reference samples with very low mass fractions in the range of ng/mm² or less with homogeneous distribution of the reference element on the sample. In this range standard droplet samples reach their limits due to agglomerations in the drying process of the droplet.

Therefore a project was started to develop FSTF into functional reference materials, and test samples have been produced by depositing stacks of thin metal films by physical vapour deposition (PVD) at AXO Dresden. We used polymer foils and silicon nitride membranes as supports. The deposition technique assures very homogeneous layers and a large degree of flexibility regarding the choice of elements and mass densities.

The samples were characterized by AAS and ICP-OES at the University of Hamburg and by micro-XRF at the synchrotron radiation sources HASYLAB, Hamburg, and ANKA, Karlsruhe as well as LANL, Los Alamos, and show a very good reproducibility of elemental depositions. The statistical evaluation of micro-XRF mapping data obtained with beam sizes of 1.5 μ m to 100 μ m and photon energies up to 40 keV showed that lateral mass deposition heterogeneities are below 1% (rms) for most elements even at 1.5 μ m spatial resolution. Possible applications of these FSTFs are calibration of the energy scales of EDXRF instruments, assessment of absolute lower detection limits and characterization of depth resolution and sensitivity of confocal set-ups.

Further, reference samples with layer type depositions in the atomic monolayer and sub-monolayer range ($< 10^{15}$ Ni- atoms/cm²) are developed and tested to meet the demands of the decreasing lower limits of detection of TXRF in terms of technical feasibility.