Nanometer thin films as XRF reference samples

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An ideal reference sample for the calibration of the sensitivity of micro-XRF set-ups has negligible absorption of exciting and fluorescent radiation, a high degree of uniformity and homogeneity and emits a multitude of non overlapping X-ray fluorescence lines over a broad spectral range with suitable, preferably comparable 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. Therefore activities were started to develop FSTF into functional reference materials and first 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. Various deposition techniques assure 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 showing a very good reproducibility of the elemental depositions. The statistical evaluation of micro-XRF mapping data, obtained with beam sizes of 1.5 µm to 100 µm and beam energies up to 26 keV, showed that mass deposition heterogeneities are below 1% rms for most elements even at 1.5 µm resolution. Recently, very thin membranes have been tested as supports, since they can withstand very high radiation intensities and absorption is further reduced due to the smaller thickness. XRF measurements indicate that these samples are equivalent in terms of homogeneity with the polymer foil based samples.

First applications of these FSTFs aimed at assessment of absolute lower detection limit and characterization of depth resolution and sensitivity of confocal set-ups.