Characterization of nanomaterials with combined GIXRF and XRR approach

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Grazing Incidence XRF (GIXRF) is a Total Reflection X-Ray Fluorescence Analysis (TXRF) related technique. As the penetration depth of the incident X-ray beam in the total-reflection regime is very small, i.e. in the order of only a few nanometers, the XRF spectra represent the elemental concentrations of this near surface region. Furthermore, varying the angle of incidence in the grazing incident regime and collecting XRF spectra at several angle positions results in angle dependent intensity curves for each element. These curves are correlated to the layer thickness, depth distribution and mass density of the elements in the sample. But the evaluation of these measurements is ambiguous with regard to the exact distribution function for the implants as well as for the thickness and density of nanometer-thin layers.

In order to overcome the ambiguities of GIXRF the method is combined with X-ray reflectometry (XRR), which is an established technique for the characterization of single- and multi-layered thin film structures. Both techniques use similar measurement and data evaluation procedures. Moreover, the physical properties for the calculation can be derived directly from the same sample model, thus making the approach straightforward. This combined analysis reduces the uncertainties of the parameters of the individual techniques. The software JGIXA was specifically developed for the simultaneous calculation and fitting of GIXRF and XRR data, but also allows separate evaluation. The quality of the fit is determined via a total chi-square value of all available measurement data. Global optimization algorithms perform the refinement of the sample parameters.

Software and data evaluation approach were benchmarked by characterizing metal and metal oxide layers on Silicon as well as Arsenic implants in Silicon. The results of the different optimization algorithms have been compared to test the convergence of the algorithms.

The combined measurement and evaluation of GIXRF and XRR data can improve the obtained information, as it reduces uncertainties and ambiguities of the individual techniques [1][2].

An Empyrean System, which is a commercially available platform for X-ray Diffraction applications, offers optics and detectors for XRR measurements. In order to allow for combined measurements, an Amptek SDD was added to this system and the acquisition of XRF spectra synchronized to the XRR scan. A comparison of measurements, which were performed with different incident beam monochromators and mirrors, which are available for the Empyrean System, as well as a comparison to data obtained from a table-top spectrometer, which was specifically developed for combined GIXRF and XRR measurements, will be presented.

[1] Ingerle, D., Pepponi, G., Meirer, F., Wobrauschek, P., & Streli, C. (2016). JGIXA — A software package for the calculation and fitting of grazing incidence X-ray fluorescence and X-ray reflectivity data for the characterization of nanometer-layers and ultra-shallow-implants. *Spectrochimica Acta Part B: Atomic Spectroscopy*, *118*, 20–28. https://doi.org/10.1016/j.sab.2016.02.010

[2] Ingerle, D., Meirer, F., Pepponi, G., Demenev, E., Giubertoni, D., Wobrauschek, P., & Streli, C. (2014). Combined evaluation of grazing incidence X-ray fluorescence and X-ray reflectivity data for improved profiling of ultra-shallow depth distributions. *Spectrochimica Acta. Part B: Atomic Spectroscopy*, *99*(100), 121–128. https://doi.org/10.1016/j.sab.2014.06.019