CHARACTERIZATION OF NANOPARTICLES WITH X-RAY SPECTROMETRY UNDER GRAZING INCIDENCE CONDITIONS

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Reliable and specific metrology and methodology are needed in order to analyze the impact of nanoparticles to the environment, especially for the analysis of dose response relationships. Besides quantity the knowledge of the elemental composition and chemical properties of aerosols from -in many cases- unidentified sources are essential for the assessment of potential risks which may emerge from exposure of industrial goods (e.g. wafers) or humans to aerosols. For these reasons, the characterization of nanoparticles needs further development with respect to sampling techniques combined with sensitive chemical and elemental analysis and quantitative metrology as well.

X-ray spectrometry, in general, is a non-destructive analysis method for the elemental composition of various sample systems. At synchrotron radiation facilities ideal beam conditions are achieved and, thus, more sophisticated analytic techniques can be applied. High spectral resolution enables studies of the chemical state of the elements [1] while the low divergence of the beam allows for dedicated geometries, such as a grazing incidence arrangement. Grazing Incidence X-Ray Fluorescence analysis (GIXRF) has the potential to effectively contribute to the characterization of nanoparticles deposited on flat surfaces with respect to elemental compositions, and potentially even depth profiles. Based on Total-Reflection X-ray Fluorescence (TXRF) analysis, which offers lower levels of detection in the pg to fg range [2], in GIXRF the incident angle of the excitation radiation is tuned between 0° and about threefold the critical angle of total-reflection. Therewith, the intensity of the X-ray standing wave field (XSW) at a given height above the surface can be considerably modified. A particle of a specified diameter deposited on the flat surface will be affected by the intensity of excitation radiation varying with the angle of incidence and hence yields a correspondingly varying X-ray fluorescence signal. Thus, in addition to information on the elementary composition of the particles, which is inherent to XRF, some information on the deposited size fraction can be obtained.

For the development of advanced models for quantitative X-ray spectrometry (XRS), well-characterized sample systems and instrumentation are a pre-condition. PTB employs well-characterized beamlines, reliable XRS instrumentation and absolutely calibrated X-ray detectors for the non-destructive investigation of bulk and layered samples at BESSY II [3]. On clean wafer-surfaces size-preselected TiO_2 and NaCl nanoparticles were sampled by using a newly developed tunable impactor or an electrostatic sampler. The impactor produces a characteristic deposition pattern while electrostatic sampling led to an even distribution with deposition densities up to 10^5 particles/mm^2. Quantitative results concerning the absolute mass of the deposited particles were obtained.

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