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Probing Nanoparticles Deposited on Flat Surfaces by X-ray Spectrometry at Grazing Incidence

Falk Reinhardt[1], Burkhard Beckhoff[1], Harald Bresch[2] und Stefan Seeger[2]

[1]Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin
[2]Bundesanstalt für Materialforschung und -prüfung, Unter den Eichen 87, IV.24, 12205 Berlin

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The strive for advancement in quantitative and qualitative nanoparticle analysis is motivated by multiple questions about potential risks caused by handling of or exposition to nanoscaled particles (interaction with the environment). Among those questions the verification and quantification of the dose-response relationship is a prominent one. Furthermore, improved knowledge of the interactions of nanoparticles with all kind of material surfaces is necessary for evaluating the use of the particle's properties. To answer those questions analysis of nanoparticles has to be developed further with respect to both sampling procedures and analytic techniques aiming on elemental compositions.

The further development of analytic techniques also involves the examination of deposited nanoparticles with synchrotron radiation. Depending on the particle number concentration, size-preselected nanoparticles were sampled on clean and flat silicon wafers either by use of a cascade impactor or by use of an electrostatic sampler. The deposition density on the wafer was approximately 10^5 particles/mm^2 or less. Deposition with the electrostatic sampler led to an approximately evenly distributed deposition.

PTB employs monochromatized synchrotron radiation of well-characterized beamlines, reliable X-ray spectrometry (XRS) instrumentation and absolutely calibrated X-ray detectors for the non-destructive investigation of bulk and layered samples in its laboratory at BESSY II (Beckhoff, 2008).

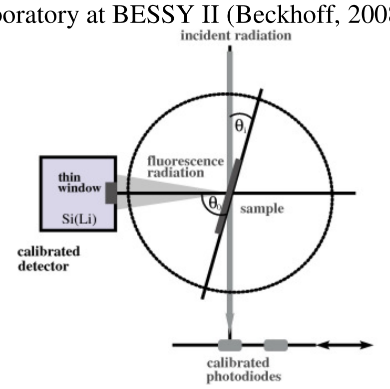

Figure 1: Sketch of grazing incidence XRS beam geometry

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 analysis (TXRF), which offers lower levels of detection in the pg to fg range, 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 is modified. A particle of a given 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, information on the deposited size fraction can be obtained (Fig. 2).

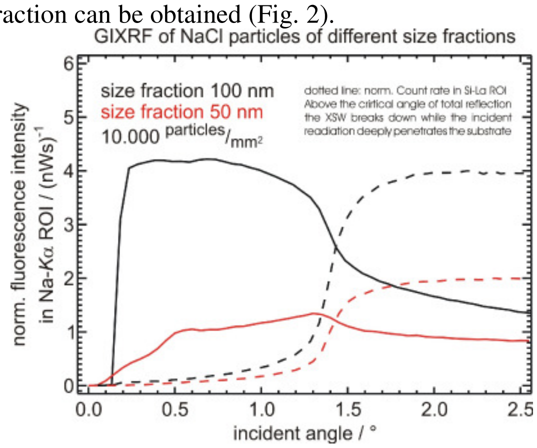

Figure 2: Angular dependence of detected fluorescence radiation dependent on incident angle.

Different specimens were prepared by depositing Zn compounds and NaCl particles with size fractions down to 10 nm on silicon wafer substrates by use of a differential mobility analyzer (DMA). Those particles serve as model systems for current and future reference-free quantitative analysis of size fractioned aerosol particles.

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Beckhoff, B. (2008). *J. Anal. At. Spectrom.* 23, 845.