Quantitative characterization of energy and nanomaterials by means of traceable x-ray spectrometry

Burkhard BECKHOFF - *Physikalisch-Technische Bundesanstalt (PTB), Germany*

Traceable characterization methods allow for the accurate correlation of the functionality of energy or nanomaterials with the underlaying chemical, structural or physical material properties. These correlations are required for the directed development of advanced materials at both the nano- and microscales to reach target functionalities such as conversion efficiencies or other specific device performances. The reliable characterization of these materials requires techniques that often need to be adapted to the nanoscaled dimensions of the samples with respect to both the spatial dimensions of the probe and the instrumental or experimental discrimination capability. The traceability of analytical methods revealing information on chemical material properties relies on reference materials or qualified calibration samples, the spatial elemental distributions of which must be very similar to the nanomaterial of interest. At the nano- and microscales, however, there exist only few well-known reference materials. An alternate route to establish the required traceability lays in the physical calibration of the analytical instrument's response behavior and efficiency in conjunction with a good knowledge of the various interaction probabilities. This SI-traceable approach for x-ray spectrometry has been established by Germany's metrology institute PTB. For the elemental analysis, speciation, and coordination of energy or nanomaterials, such a physical traceability can be achieved for x-ray spectrometry. This requires the radiometric calibration of energy- and wavelength-dispersive x-ray spectrometers as well as the reliable determination of atomic x-ray fundamental parameters using such instrumentation. In different operational configurations the information depths, discrimination capability and sensitivity of x-ray spectrometry can be considerably modified while preserving its traceability allowing for the characterization of surface contamination as well as interfacial thin layer and nano- or microparticle chemical compositions. Furthermore, time-resolved and hybrid approaches provide access to analytical information on energy storage materials such as batteries under operando conditions or allow to reveal dimensional information such as in elemental or species depth profiles of nanomaterials. Application examples for both nanoelectronics as well as energy storage and conversion materials regarding selective characterizations at the nanoscale and for the qualification of calibration samples that can employed in laboratory x-ray instruments will be provided.