Analysis of Carbon- and other Nano-Structures using Si Drift Detectors in Energy Dispersive X-ray Spectroscopy

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We want to explain the silicon drift detector (SDD) technology briefly and show examples of nanoanalysis using SDD for SEM and TEM. Peltier cooled Silicon drift detectors, originally developed for space research where the use of liquid nitrogen was impossible, have been introduced for energy dispersive X-ray spectroscopy (EDS) in scanning electron microscopy (SEM) more than a decade ago [1]. Now they are about to revolutionize nanoanalysis of element composition using EDS in SEM and transmission electron microscopy (TEM) [2].

SEM at low voltages is suitable to analyse surfaces of bulk structures and to investigate electron transparent samples with nm-resolution. Image resolution of up to atomic level is now routinely available using aberration corrected TEM. Brukers liquid nitrogen free EDS for element mapping is suitable for both SEM and aberration corrected TEM. Using one 30mm^2 SDD at 0.12sr solid angle for x-ray collection, atom column EDS in case of an aberration corrected instrument [3] and nm resolution EDS for a conventional TEM [2] have been shown. In TEM atom column EDS is seen as a directly interpretable important complement to the well established electron energy loss spectroscopy [4] used for chemical analysis of nano-structures on atomic level. Multiple detectors achieving a much higher solid angle for x-ray collection can be used for very fast elemental mapping and to detect small amounts of matter in the nanostructure [5].

Our first example of SDD-EDS is a combined SEM- and TEM-study of an array of multiwall carbon nanotubes (MWCNT). Fig. 1 shows the MWCNT array in the SEM and a signal mixed of transmitted and back scattered electrons, which reveals the whereabouts of the catalyst nanoparticles used for the CNT growth. Element mapping by EDS in a transmission electron microscope distinguishes between Ni and Co catalyst particles remaining inside the tubes and shows the successful Cu coating of the multiwall carbon nanotubes [6,7]. The Z-contrast in high angle annular dark field (HAADF) imaging clearly distinguishes between the heavier matter of the coating and catalyst particles and the lighter carbon of the tubes.

Figure 2 shows data on magnetic nanostructures, achieved using a combination of electron energy loss spectroscpüy and EDS in TEM. The study of the element composition on the nm-scale helped to explain the magnetic behaviour of these hedgehog like nanospheres [8].

The third example deals with the compositional analysis of quantum well samples used for the development of layser diodes (Figure 3). Utilising TEM EDS the element composition can be analysid within minutes [9].

In summary, SDD-technology is a valuable addition to the analytical tools provided for analysis of nanostructures on atomic level. The low interference, low noise and good light element performance as well as the robustness and capability to deal with high exposure rates as well as low x-ray input makes this technology particularly interesting for the use in high end aberration corrected microscopes and in combination with high brightness sources. Successful data analysis on atom column level is possible.

References

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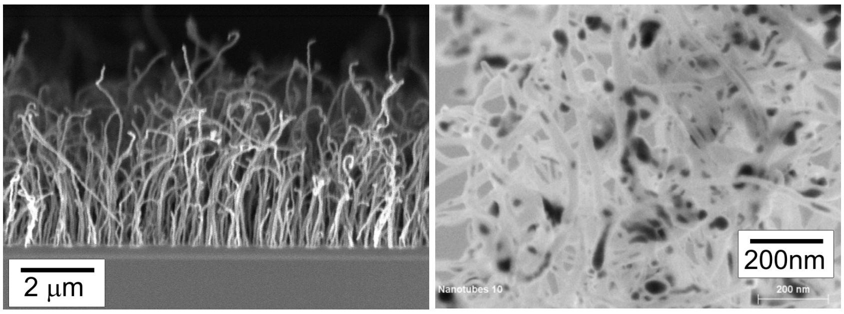
Figures



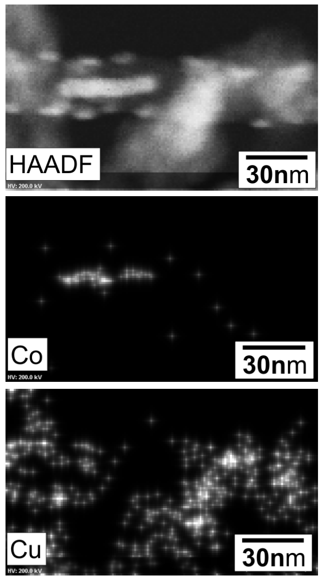


Figure 1. Left: Array of MWCNTs on a Si/Ta- substrate in SEM. The mixed transmission and BSE signal of disordered MWCNTs reveals the where about of the catalyst nanoparticles.

Right: EDS of multiwall carbon nanotubes on a lacy carbon gold grid: HAADF, Co-map and Cu-map. The Co-catalyst particle, and the oxidized Cu-coating, all just few nm in size, were clearly revealed during this 1 min EDS-map.

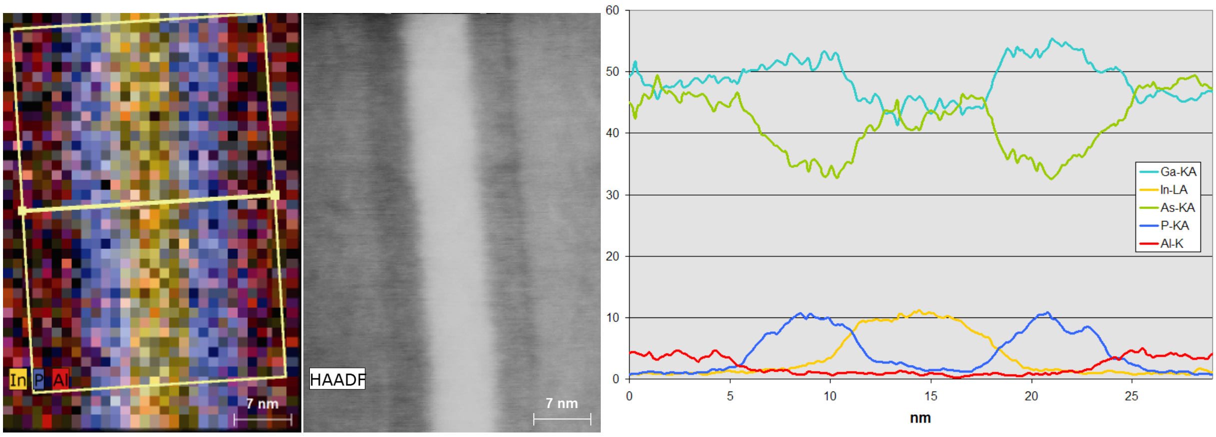


Figure 2. The element profile of a sample from research on laser diodes: The Indium distribution resembles the bright features in the HAADF signal showing heavier elements. The element profile was generated using 8by8 binning and adding up all spectra perpendicular to the layers in the indicated area. Even the nm-sized kink on the right side of the light element P-profile is reproduced in EDS as visible in the HAADF signal.

Figure 3. Upper part: HAADF and element map using EELS spectrum imaging in a NION Cs-corrected dedicated STEM. Lower Part: Liquid nitrogen free EDS map of the same sample area in an uncorrected conventional Jeol STEM, the second frame shows Ru, Pt and Cr. Pt appears with Co. Ru is part of the seed layer.