Spectroscopy in STEM/TEM

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Element analysis of nanostructures using EDS

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Modern silicon drift detector (SDD) based energy dispersive X-ray analysis (EDS) became a convenient powerful and more and more accepted tool for chemical analysis reaching the atomic level. Low dimensional and other nanoscale structures and compounds including just a few atomic percent of a particular element or even single atoms on graphene [1-2] can be analyzed using electron microscopy combined with energy dispersive X-ray spectroscopy (EDS). The use of multiple SDD-EDS detectors increases analysis speed [3] and improves analysis geometry avoiding shadowing effects in complicated structures. Thus, SDD-based EDS combined with conventional and aberration corrected scanning transmission electron microscopy (STEM) can help to solve challenging problems of nanoscale characterization in a reasonable amount of time, if proper sample preparation is provided and optimized illumination conditions ensure sample stability at sufficient X-ray yield.

Various examples of nanostructure analysis demonstrate what kind of results can be achieved with particular detector-microscope combinations. Investigated materials include, Pd/Pt core-shell catalysts (Figure 1), ALD coated carbon nanotubes for flexible interconnects in flip chip assemblies and nanorods, such as InAs/InP containing nm-layers rich in Phosphorus used for single-electron transistors development (Figure 2).

Furthermore, we show options for qualitative and quantitative analysis of element distributions which are used in the design of 2D- and other nanoscale (opto)electronic devices. The quantification of light element compounds such as BN, LaB6 and Si3N4 and the routine quantification of e.g. 2 at% of Pt alloyed NiSi thin films or nanostructures serve as examples to demonstrate, what level of characterization is currently accessible.

Data suggest, that the outcome of EDS experiments can be largely improved by Cs-correction, a high brightness electron source, suitable choice of accelerating voltage, EDS-adapted sample holders and not only a high solid angle for X-ray collection but also a high take-off angle of the EDS detector, which ensures a high peak to background ratio in the acquired spectra. Flexible and transparent analysis software is essential to provide complete data mining.

1. T. C. Lovejoy et al., Appl. Phys. Lett.100, 154101 (2012)
2. M. W. Chu et al., Phys. Rev. Lett. 104, 196101 (2010)].
3. S. von Harrach et al., Microsc Microanal 15 (Suppl. 2)(2009) 208 and Schlossmacher et al., Microscopy Today 18(4) (2010) 14-20.

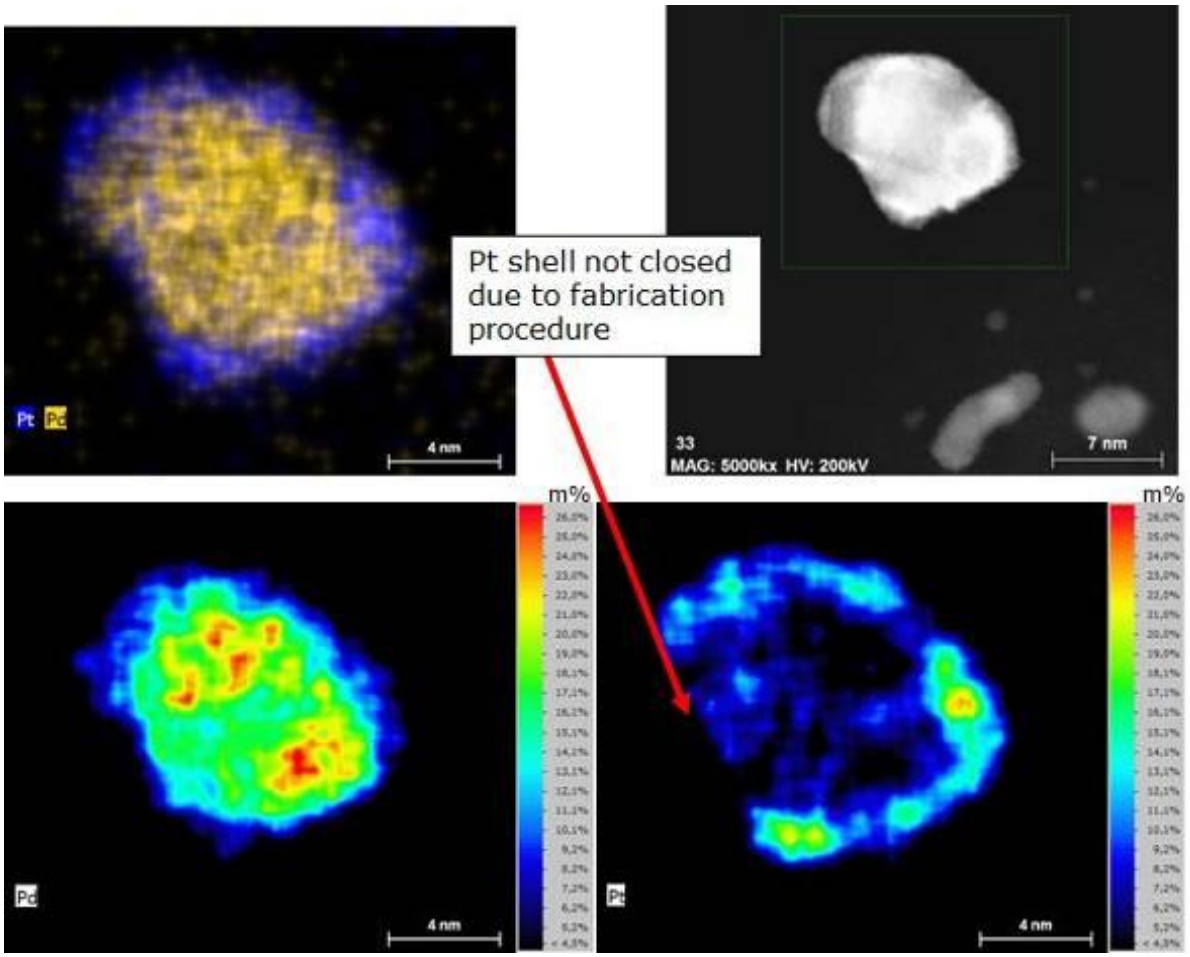

Figure 1. Pd-core / Pt-shell catalyst analysed at a solid angle of 0.12sr and a take-off angle of 22° using a Cs-corrected Schottky-FEG STEM. The open shell structure is due to the manufacturing process.
Data courtesy: D. Ozkaya, Johnson Matthey Technology Center, UK

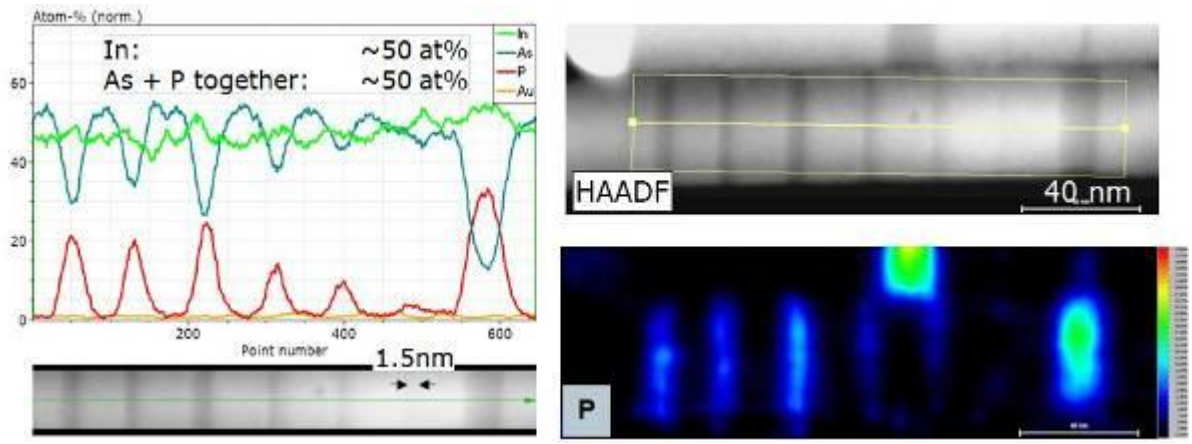

Figure 2. Phosphorus rich regions in InAs/InP nanorods, analysed at a solid angle of 0.12sr and a take-off angle of 22° using a conventional STEM.The line scan shows the quantitative data from the lower left false colour map.