Element distribution at the interface of soft and hard materials.

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Hard/soft, organic/inorganic interfaces are important in many areas of our life. Respective research includes topics such as better implant surfaces (Fig.1) [1], bio-mimetics, trying to imitate or copy solutions from nature such as e.g. shell structures and similar. Nanotoxicity is another important topic connected to nano-sized objects, which could be useful or present a health hazard, be airborne, part of our food, ingredients of cosmetics and medication, used for drug delivery, as immuno-labels, for materials properties enhancement, catalysis and so on. For understanding most of these problems chemical analysis with spatial resolution on the micro to atomic scale as well as in situ, e.g. in liquid and at elevated temperature is necessary.

Energy-dispersive X-ray spectroscopy (EDS) in the electron microscope is one method to get information on all those levels. However, the interface between soft and hard materials poses challenges for this type of analysis. Samples resembling a close to natural state are often highly topographic, need analysis in liquid or air and can be beam sensitive. Preparation artefacts and absorption effects need to be considered quantitatively as well. This demands for high end instrumentation on both, the microscope and detector side.

We report on the use of existing technology and on advances in instrumentation for element mapping of bulk and electron transparent samples challenging as just described. The examples range from TEM- and T-SEM (TEM in SEM) suitable samples, including single atoms (Fig.2) [2], cells or nano- and core-shell particles, via samples in liquid to highly topographic bulk materials such as porous polymers with hard inclusions and crystals formed by bio-mineralisation in plants, insects and bacteria. Available low and high end detector arrangements and respective successful measurement conditions and analysis strategies used in TEM/STEM and SEM and T-SEM will be explained.

[1] T Yang et al., Colloids and Surfaces B: Biointerfaces 145 (2016) pp. 597-606.

[2] R M Stroud, et al., Appl. Phys. Lett. 108 (2016) p. 163101.

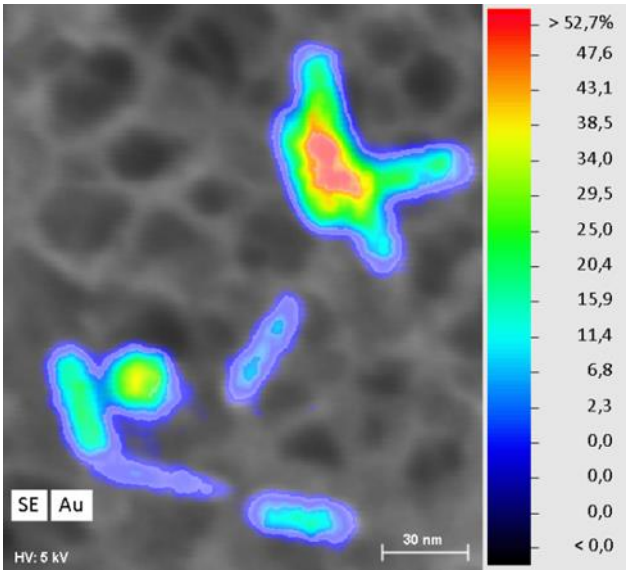

Figure.1: Gold nanoparticles on porous titanium oxide implant surfaces, used to lower the infection risk. They change the electric surface potential making it fatal for settling bacteria. High collection, high take-off angle low kV EDS in SEM helps to quickly judge the distribution and embedding of various nanoparticle types, sizes and shapes on the porous oxide surface of the potential implant. These data can be correlated with light microscopy

[1]. Acquisition using an annular detector with a solid angle of ~ 1sr, a FEG SEM at 5kV, Au net counts in pseudo colours normalized to the maximum net count sum. Scale bar: 30nm. Data courtesy: Guangxi University; X. Liu and Y. Zeng, Shanghai Institute of Ceramics, Chinese Academy of Science, China.

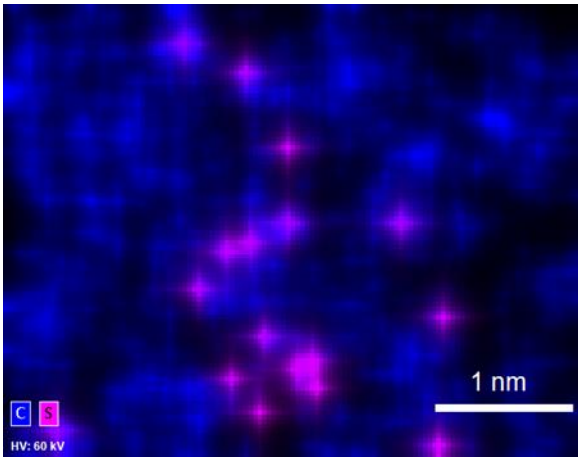

Figure 2: Single sulphur atom captured moving with the electron beam on carbonaceous material during a single scan. Single frame map acquired in 60 seconds using an oval 100mm2 EDS detector with 0.7sr solid angle in a dedicated Cs-corrected cold FEG UHV STEM at 60kV. Sample and data courtesy: R. Stroud, NRL, Washington, USA [2].