ESRF	**Experiment title:** Strain fields and interdiffusion in core-shell nanorods using anomalous coherent diffraction	**Experiment number**: HS-4033
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Report:

The relatively high volume fraction of interfaces and surfaces in low-dimensional systems leads to favourable tuneable properties, but makes them also prone to degradation. In this experiment, core-shell nanorods and core-shell islands were investigated by anomalous coherent diffraction to study the relationship between stresses and interdiffusion. The 3D strain field inside such objects can be analyzed by inversion of the coherent diffraction patterns at Bragg peaks, whereas information on the elemental distribution can be deduced from differences in the diffraction patterns at different beam energies. The combination of the two methods allows for the non-destructive, simultaneous characterization of the strain fields and compositional gradients.

We investigated two types of Ag/Au core-shell structures, namely nanorods and islands, prepared by physical vapour deposition [1] (see Figure 1.a). Before the experiment, single Ag/Au nanorods with diameters of ~ 100 – 200 nm were selected from their growth substrate and glued on Cu TEM grids with Pt (see Figure 1.b). At first, such a grid with ~ 100 nm thick rods was mounted onto a heating stage (see Figure 1.c).

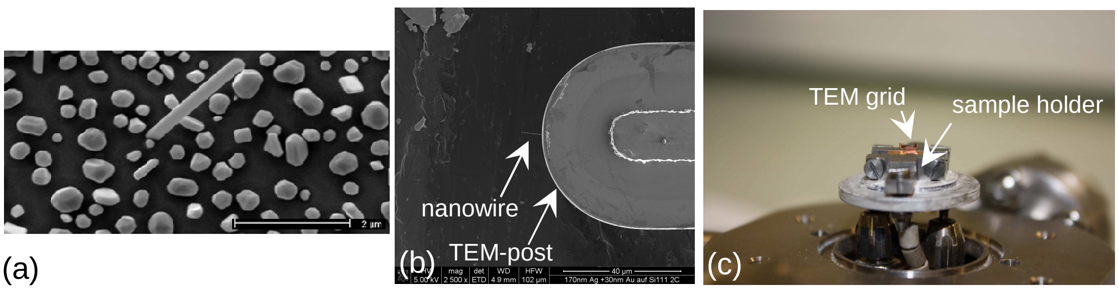

Figure 1. a) SEM image of nanorods and islands on their growth substrate, b) SEM image of a single nanowire on top of a TEM-post, c) Photograph of the sample holder which clamps the TEM grid. The sample holder is a part of the heating stage.

The beam energy was adjusted to the Au L_{III} edge (11.919 keV). The coherent x-ray beam was focused by a KB system at the sample position (~3μm (H) x3μm (V)). It was possible to identify the position of the TEM grid by the Cu fluorescence. Unfortunately, the fluorescence signal from a single rod could not be detected, probably due to the small signal on the very high background from the Cu fluorescence. Therefore, one sample with ~ 200 nm thick rods (prepared by a two-step growth process of the Au shell) was mounted, but still no fluorescence signal could be found from the rod. To find a 111 reflection (the (111) planes are

perpendicular to the <110> rod axis), the Maxipix detector was placed close to the sample to increase the angular range, and 3D mesh for the x-axis, z-axis and the rotation around the rod axis was performed. A 111 diffraction spot was found, and the detector was placed at a larger distance from the sample to increase the resolution of the fringes. The observed barcode pattern (see Figure 2.a) hints at many stacking faults [2], which hinder the data evaluation. For that reason, we again mounted the sample with the thinner rods and repeated the procedure to find the 111 reflection. But during precise alignment for subsequent rocking curve measurements, the coherent pattern disappeared due to beam dammage (see Figure 2.b).

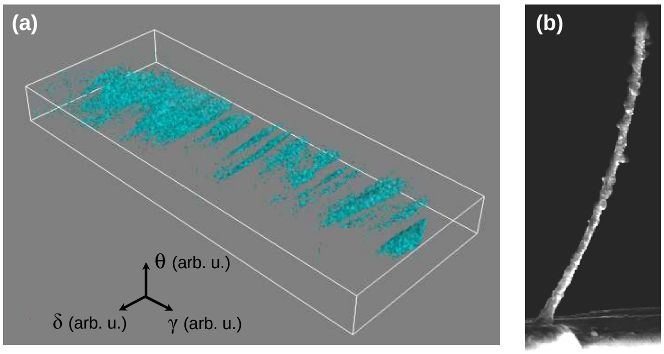

Figure 2. a) Barcode diffraction pattern from a faulted nanorod at the 111 reflection. θ is the rotation angle around the rod axis. δ and γ are the in-plane and out-of-plane rotation angles of the detector arm, b) SEM image of the nanorod with severe beam damage after the CXD experiment.

The following measurements were performed on the core-shell islands. A 220 out-of-plane reflection was selected and the sample was scanned until finding one island with the predefined orientation (all island have (111) planes parallel to the substrate). In air, the carbon layer underneath the island was not stable under X-ray irradiation, but under vacuum it was stable. Full 3D rocking curves were recorded at the Au L_{III} edge and below for two islands (see Figure 3). The position of the beam was very stable with energy due to the KB mirror.

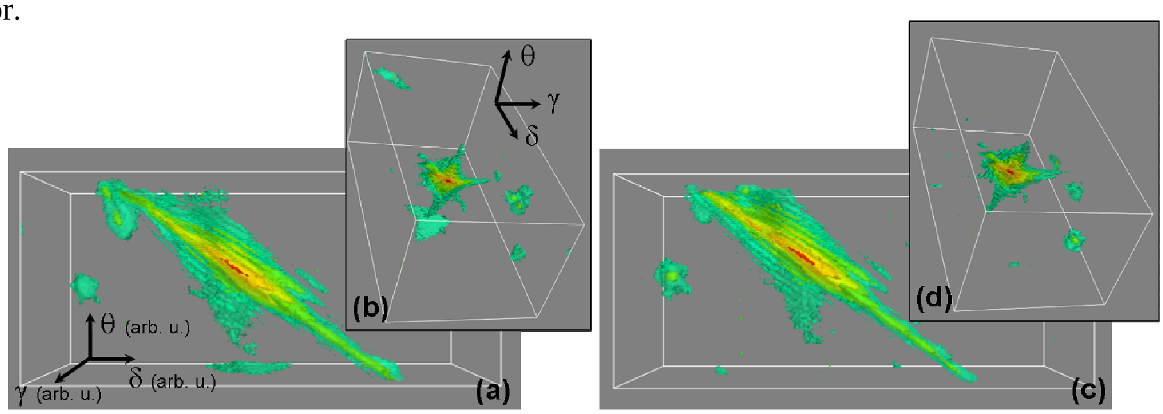

Figure 3. Isosurface representations of 3D rocking curves of a (220) Ag/Au reflection of a Ag/Au core-shell island a,b) at the Au L_{III} edge and b) below the Au L_{III} edge.

The data are first evaluated qualitatively by comparing the different patterns at different energies. First, the spacings of the fringes give valuable information on the dimensions. The patterns will be compared to simulations from model assumptions. Furthermore, it will be examined if the inversion of the patterns by phase retrieval algorithms [3-5] is possible, including the elemental information from the anomalous part. This work is under progress.

In summary, it was possible to record 3D diffraction patterns around a Bragg peak at different energies for core-shell islands. It can be concluded, that this study shows the promising combination of anomalous and coherent X-ray diffraction to examine strain and diffusion in such small-scale objects. Beam damage has to be considered, but could be avoided by measurements under vacuum. In-situ heating could not yet be performed during this beamtime. It would provide unique fundamental knowledge on the coupling between chemical intermixing and stresses for nano-structures with core-shell geometry.

References:

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