Quantitative High-Resolution TEM/STEM and Diffraction

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Calibration of HRTEM for lattice parameter measurements in nanocrystals

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Lattice parameter changes in nano-crystalline systems are, in most cases, indicative of altered physical, compositional and catalyst properties. High Resolution Transmission Electron Microscopy (HRTEM) is a very effective technique for the structural and morphological study of nano-crystals. Reliable interlattice spacings measurements, however, are usually problematic due to imprecise magnification of the microscope. In this work, we propose an in situ calibration technique for HRTEM: the nano-crystals of the sample are placed on the face side of a thin amorphous carbon foil, while reference (Au) nanocrystals are deposited on the back side either by evaporation or drop drying from sol. This arrangement enables, that the lattice fringes of sample and reference crystals are HRTEM-recorded simultaneously. This provides extremely precise calibration and, therefore, measurement of the interlattice spacings of the sample, as well, localization of any lattice distortions. Since sample and reference are separated by a substrate no artefacts may occur due to the physical or chemical interactions of sample and reference. The systematic calibration error caused by a typical displacement (~25nm) between sample and reference was found to be negligible (~0.001%) by simple optical model calculations. The advantages of this technique was shown by measuring decreasing lattice spacings in Au nanoparticles as a function of decreasing particles size (finite size effect). Lattice parameter changes in Au/Pd core/shell catalyst nanoparticles or in bimetallic alloy nanoparticles could be also detected. It is to mention that the measured data has to be averaged. The recorded interlattice spacings of- even- the Au reference particles shows a remarkable dispersion due to imaging conditions of particles with random misalignements to exact zone axis [1]. The buildup of a typical sample together with the reference layer is depicted by the cross sectional scheem shown in Figure 1. Figure 2. shows a high resolution TEM micrograph for to measure the finite size effect in small particles. Both the sample (2~4nm in size) and the reference (10~20nm) particles show lattice fringes simultaneously that allows precise comparison of lattice parameters. In this case we suggest that the particles of the sample (small) undergo, while that of the reference does not undergo a lattice distortion due to finite size effect, compared to bulk value.

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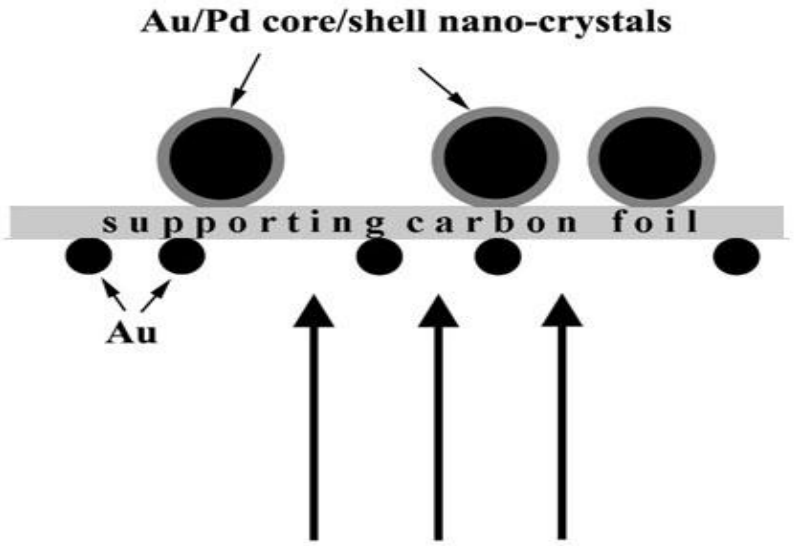

Figure 1. Set-up of a Au/Pd specimen for an in situ HRTEM calibration and measurement of fine lattice distortions. The particles of the sample are on top, while that of the reference are deposited on the bottom of a thin carbon foil.

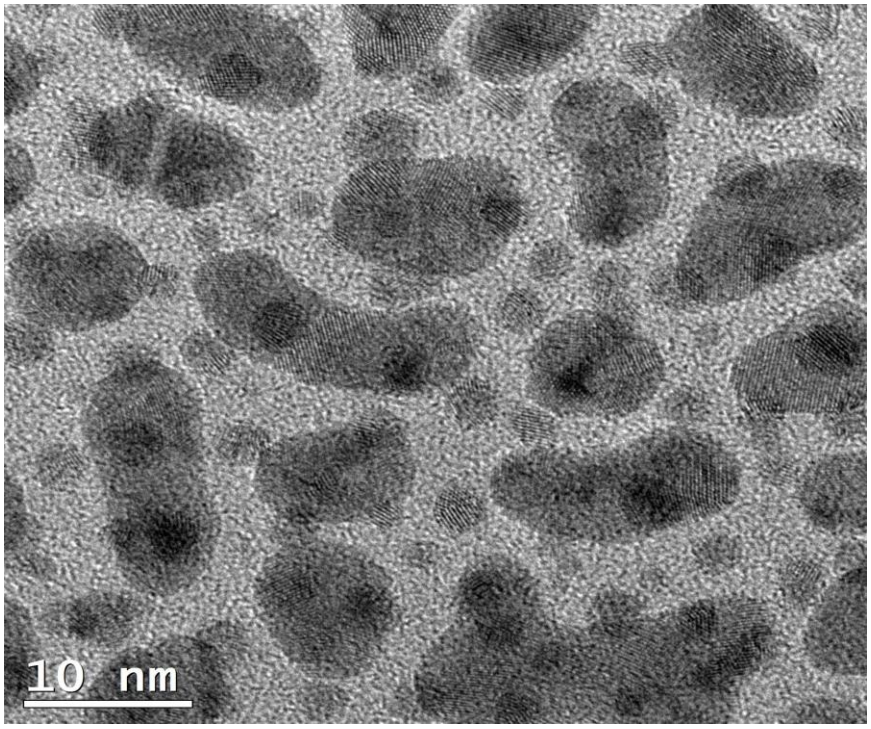

Figure 2. HRTEM micrograph that simultaneously reveal lattice fringes of both sample and reference Au nano-crystals for in situ calibration and measurements. Sample (particles 2~4 nm in size) and reference (particles 10~20nm in size) are deposited on the opposite faces of the carbon substrate.