Microsc. Microanal. Vol. 13 Suppl. 3 (2007) 258.

Structural studies of nanoparticles with an objective-lens C_s-corrected Titan microscope

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Recent tremendous progress in HRTEM made it possible to achieve sub-Å resolution in a 'mid-voltage' microscope through both hardware C_s corrector and software correction, i.e., focus-series reconstructions, to extend the resolution defined by Scherzer defocus to an expected information limit less than 1 Å. [1-3]. Such development in microscopy offers improvement for direct interpretation of image contrast and microanalysis with improved spatial resolution in materials science applications. With the tunable C_s, a novel imaging mode is exploited by a combination of a negative Cs and a small overfocus, giving the 'bright atoms' contrast [3], which is of great importance and particularly beneficial when imaging weakly scattering light elements. It thus opens up a way to look at the material at atomic level in more detail. Further significant benefits provided by aberration-corrections are the characterization of nanometer–sized particles without delocalized contrast and with largely suppressed phase contrast of the supporting substrate, which make precise size measurement and structure analysis as well as particle evolution observation possible.

Using a Cs–corrected microscope (Titan FEI 80-300 with field emission gun, HAADF detector and Tridiem system) we here demonstrate some particle examples with particle sizes in the range of ~2-10 nm. The first example concerns the core-shell CdSe/CdS (CdSe/ZnS) nanocrystals synthesized by a colloidal method in an organic solvent with a shell thickness of one to five monolayers. The experimental studies were combined with image calculation, and reveal that direct visualization of the shell is theoretically possible but experimentally difficult to achieve. This is most likely due to atom diffusion between the extreme thin shell and the core or epitaxially growth as the similarities in structure and lattice constants. The Cd-Se dumbbell and twin boundary with a distinct atomic arrangement are clearly visible under a small negative C_s and defocus value, as under these conditions, the phase contrasts of substrate is suppressed and the contrasts of Cd and Se is increased [5]. However, the HAADF image on an individual particle shows a slight intensity difference between the edge and the center of the particles.

As a catalyst system, the interface between Pt nanoparticles about 2 nm in diameter and carbon nanotube (CNT) is well characterized by using a small Cs value, which enables visualization of a distinct interface contact and the interaction process under the electron beam between the particles and CNT substrate (Fig. 2). As a result, a thin carbon layer forms outside the Pt particles. Fig. 3 displays the terminated atom arrangements and facet planes of an isolated FePt nanoparticle (*fcc*) about 5 nm on MgO (001) without fussy delocalization. An appropriate choice of Cs values provides the improvement of image quality and interpretability for such particles, and facilitates the determination of the relationship in betweens. Multiple twin structures in such particles are well resolved as well [5].

References
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[5]. We thank Dr.Dangsheng Su in Fritz-Haber institute Berlin for providing the sample of Pt-CNT catalyst, and the support from Germany Research Foundation DFG with SFB 569

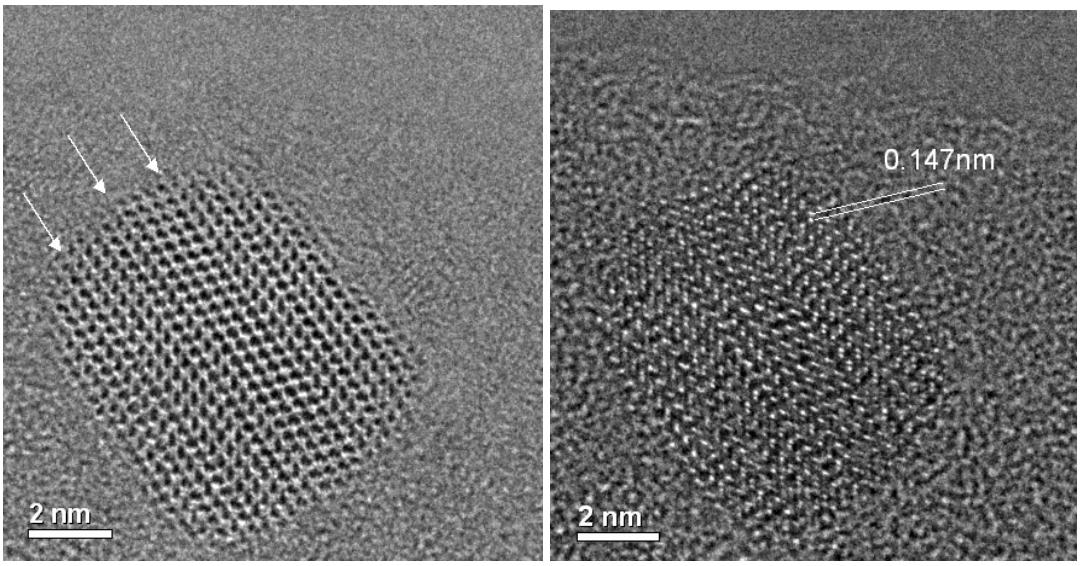

Figure 1. CdSe/CdS core-shell particle imaged with a small negative Cs and a small underfocus (a, left), and overfocus (b, right) with clearly resolved Cd-Se dumbbells with a spacing of 0.147 nm. (c) One STEM image from an individual particle.

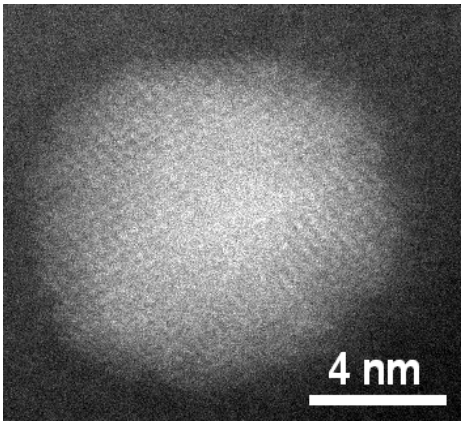

Figure 1c

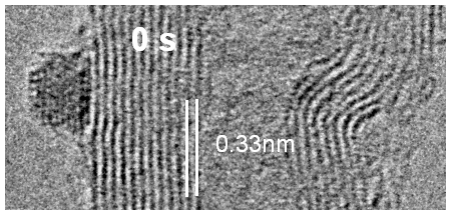

Figure 2

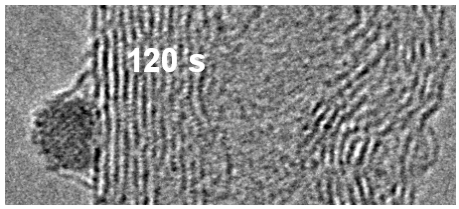

Figure 3

Figure 2. Pt nanocrystals on carbon nanotube imaged with a small Cs value. The interaction between Pt and CNT occurs under the electron beam. Note that the particle is eventually wrapped by a graphene shell.

Figure 3. FePt nanoparticles on MgO (001) imaged with a small spherical aberration.