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Statistical Determination of Atomic-Scale Characteristics of Gold Nanocrystals Based on Correlative Multiscale Transmission Electron Microscopy

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The functionality and performance of nanocrystals (NCs) are controlled primarily by their size and shape, but they are tremendously affected by atomic-scale characteristics of the NCs, such as microstructural defects or the crystallographic orientation of the facets. While size and shape of NCs are frequently determined on large NC populations, atomic-scale characteristics of NCs are in general determined by high-resolution transmission electron microscopy (HRTEM) on individual NCs, and thus with a limited statistical relevance. In the present work, a multiscale approach is presented, which enables the determination of atomic-scale characteristics of NCs in a statistical manner by correlating these atomic-scale characteristics observed in atomic resolution images with the morphological appearance of the NCs that can be easily investigated in low-magnification images utilized for statistical characterization.

Two different samples of Au NCs were investigated that were synthesized according to published procedures [1,2]. The first sample was a mixture of defective NCs consisting of multiple-twinned NCs and plate-shaped NCs containing stacking faults. The second sample contained Au nanorods (NRs) with mixed high- and low-index facets. The Au NCs and NRs were investigated by HRTEM in combination with local fast Fourier transformations (FFTs) and by high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM). HRTEM/FFTs was used to identify microstructure defects in the NCs and the crystallographic orientation of the facets of the NRs. HAADF-STEM images were recorded at low-magnification and subjected to a semi-automatic segmentation routine for statistical characterization of the projected size and shape [3], which was extended to consider the full HAADF-STEM intensity distribution for the data processing.

For the defective Au NCs, the correlative multiscale approach provided the amount of multiple-twinned and plate-shaped particles in the sample. These distinctive types of Au NCs were identified unambiguously from low-magnification HAADF-STEM images by using the correlation between the size of the NCs and the HAADF-STEM intensity variation. In plate-shaped NCs, the defect density was estimated qualitatively by considering the correlation between their defect density and morphology [4].

For the Au NRs, the facet configurations and the amount of high-index {*hk0*} and low-index {*100*} and {*110*} facets were determined in a statistical manner. While the crystallographic orientations of the facets at the tips of the NRs were determined from their projected shape, the facet configurations of the NR cross sections were derived from the HAADF-STEM intensity profile [5].

In conclusion, a correlative multiscale approach was introduced that enables a statistically relevant quantification of atomic-scale features of NCs, which were originally identified by HRTEM/FFT. This approach utilizes a correlation between various effects of the atomic-scale features on the morphological appearance of the NCs and the scattered intensities recorded by HAADF-STEM.

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