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Scale-Bridging 3D-Analysis of Colloidal Clusters Using 360° Electron Tomography and X-Ray Nano-CT

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Colloidal clusters are self-organized assemblies of colloidal particles, which often constitute highly ordered structures (Fig. 1). Such clusters have attracted a lot of research interest regarding their formation mechanism and photonic crystal properties. Recently, clusters with magic numbers leading to exceptional stability have been identified [1]. In general, colloidal clusters contain highly ordered crystalline regions, amorphous areas and defects. The structure of a cluster depends on the number and size of its primary particles and on the formation conditions, but also varies between individual clusters in an ensemble. In order to gain further insight, appropriate clusters have to be selected for a detailed three-dimensional (3D) analysis revealing the position (x,y,z) of each individual particle in the cluster. In this contribution, we report on corresponding tomography studies of polystyrene clusters with primary particle sizes ranging from 150 nm to 500 nm.

Depending on the size and number of primary particles Electron Tomography (ET) or Nano-CT is better suited for 3D-analysis. ET shows much better resolution down to a few nm but can only be applied to clusters with sizes up to a few µm. Nano-CT can be employed for larger clusters but shows limited resolution in the range of 50 nm. Moreover, Nano-CT requires phase contrast imaging since the low density of polymer material does not provide sufficient absorption for conventional absorption contrast imaging. In our study, we used a FEI Titan Themis 300 and a ZEISS Xradia 810 Ultra for ET and Nano-CT, respectively.

Two separate workflows have been established, comprising preparation, particle screening and selection, particle transfer and 3D analysis using ET and Nano-CT, respectively. In the first workflow (Fig. 1), the clusters are dispersed onto a lacey C grid. Next, SEM imaging is employed for screening the surface structure of many clusters and select a particular one for subsequent 3D-analysis. Using our recently developed *in situ* 'stamping transfer' method the cluster is then transferred inside the SEM onto a tungsten tip for 360° ET [2]. ET is carried out in dark-field STEM mode and enables reconstruction of the complete 3D particle arrangement in the whole cluster volume [1]. In the second workflow (Fig. 2), the clusters are transferred onto a carbon pad attached to a glass substrate, which is charged to attract the clusters to the pad. The clusters form a one-dimensional arrangement on the carbon pad enabling screening of a considerable number of clusters directly in the X-ray microscope (XRM). In addition, a preliminary tilt-series can be acquired to obtain a 3D-impression of several clusters. For 360° Nano-CT (Fig. 2e, f), a cluster of interest is selected and transferred onto a tomography tip using a hair in the light microscope. The tomographic datasets of both Nano-CT and ET can be merged (if the same cluster with intermediate size is studied) or used separately to further understand the size-dependent formation process of such colloidal clusters or to serve as input for correlative simulations [3].

References:

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[3] The authors thank the DFG for financial support within the frameworks of the research training group GRK1896 'In situ Microscopy with Electrons, X-rays and Scanning Probes' and the project SP648/8 'High-resolution X-ray microscopy for correlative tomography, high throughput screening and in situ mechanical testing of structural and functional materials'

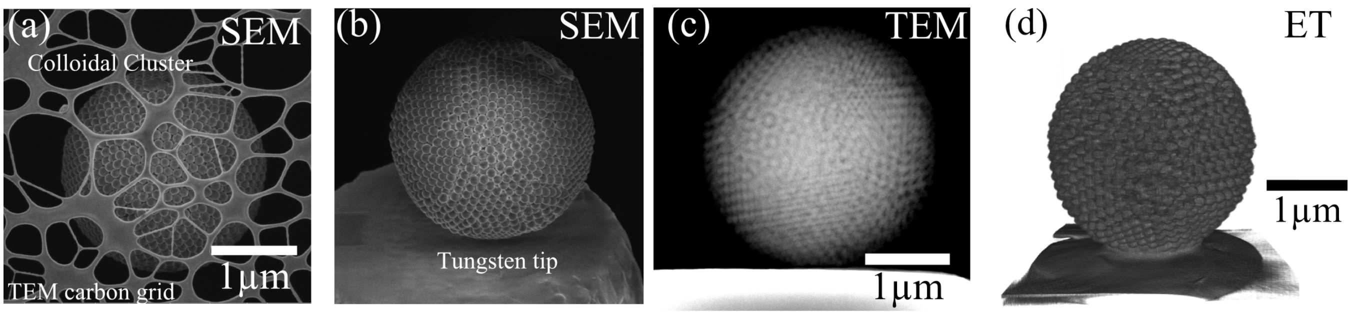

Figure 1. (a) SEM image of selected colloidal cluster on lacey carbon grid. (b) Same cluster after *in situ* stamping transfer [2] onto tungsten tip for 360° ET. (c) Individual STEM image taken from ET tilt series. (d) Volume rendering of tomogram derived by reconstruction of tilt series.

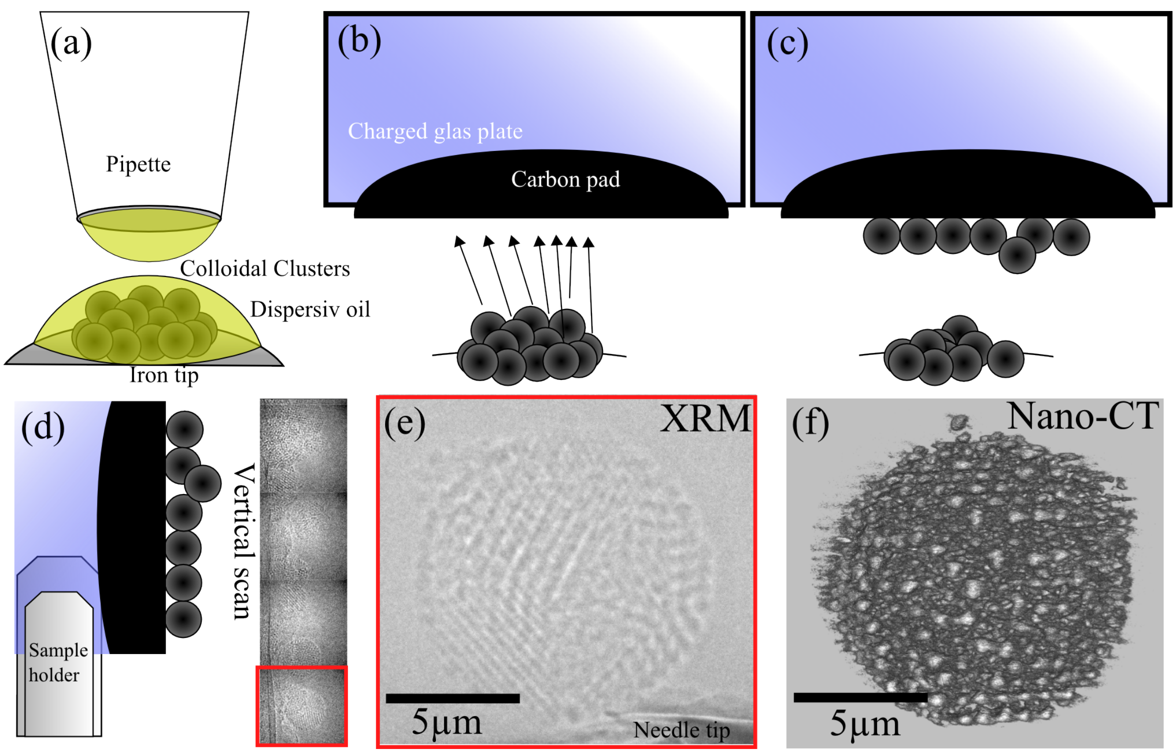

Figure 2. (a-c) Schematic illustration of transfer method enabling pre-screening of colloidal clusters in the XRM. Upon charging the glass plate colloidal clusters are attracted and stick to the attached carbon pad. (d) Pre-screening of clusters using Zernike phase contrast imaging in XRM. (e) XRM image of selected particle after transfer onto tip for Nano-CT measurement. (f) Volume rendering of tomogram derived from full Nano-CT tilt series.