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In-situ TEM Study on Sub-10 nm Materials

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With the development of semiconductor technology, the 10 nm feature size of fabrication is approaching. It is thus quite essential to explore more precise nanofabrication and characterization method to evaluate the shape/structure stability and possible new properties of sub-10-nm material components, especially under external field such as strain, electric, or thermal fields. Here we review our recent progress in atomic resolution nanofabrication and dynamic characterization of individual nanostructures and nanodevices based on the idea of "setting up a nanolab inside a transmission electron microscope".

Inside the TEM, the electron beam can be used not only to image but also to induce nanofabrication on the atomic scale. Electron beam irradiation can be utilized to etch nanocrystals, as shown in Figure 1, CaO crystals are etched by an electron beam column by column, and the process is similar to chemical etching [1]. Electron beam irradiation can also be used to repair defects in nanocrystals. Nanopores in MoS_2 and Bi_2Te_3 can be repaired to high-quality crystals with a low number of defects [2]. Besides, electron beam structuring provides a top-down approach to create quasi-1D structure by cutting 2D sheets. Robust ultrafine molybdenum-sulfide ribbons with sub-1 nm width can be widely formed in a MoS_2 sheet [3]. Single-walled armchair BN tubes can be formed in AA' stacked bilayer h-BN due to the formation of covalent interlayer bonds at two parallel zigzag edges, and the diameter of the tube can be decreased in discrete steps to 0.45 nm, corresponding to a (3,3) tube [4].

Additional probes from a special-designed holder provide the possibility to further manipulate and measure the electric/mechanical properties of the nanostructures in the small specimen chamber of a TEM. As shown in Figure 2, if a CdS is inserted in the circuit with Cu electrode, Cd is eliminated due to Ohmic heating, whereas Cu^+ migrates into the crystal driven by the electrical field force under electric bias, resulting in the formation of CdS@Cu_2S core-shell structures [5]. Such an electrically driven cation exchange can be used to grow heterogeneous structures in situ. Some novel phenomena can also be observed if the mechanical force is applied to individual crystal. Sub-10 nm Ag particles can be deformed like a liquid droplet but remain highly crystalline in the interior at room temperature, which is a surface-diffusion-mediated pseudoelastic deformation [6].

Besides, the development of liquid cell TEM technique provides an opportunity to dynamically observe the nucleation and growth of nanocrystals in liquid environment. Figure 3 shows the oriented attachment of Au nanoparticles at {111} surface inside aqueous solution [7]. The particle pairs rotate randomly at a separation distance greater than twice the layer thickness of adsorbed ligands. When the particles get closer, the ligands overlap and guide the rotation until the particles share a common {111} orientation, then a sudden contact occurs accompanied by the expulsion of the ligands on this surface [8].

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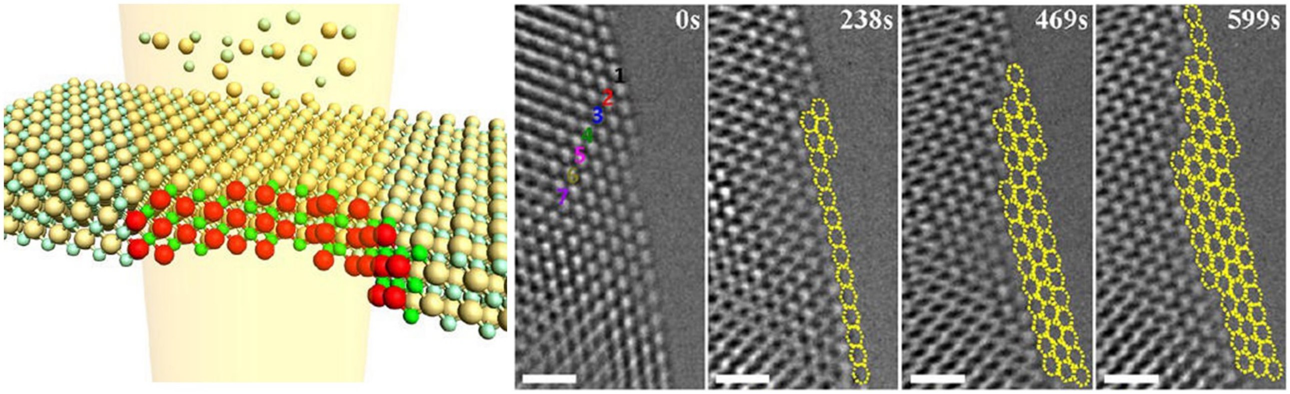

Figure 1. Etching process of CaO under electron beam. Scale bar is 1 nm

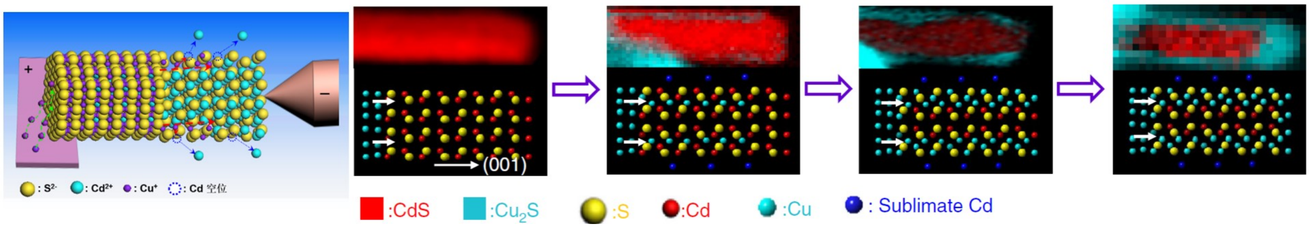

Figure 2. Electrically driven cation exchange for in-situ fabrication of individual nanostructure

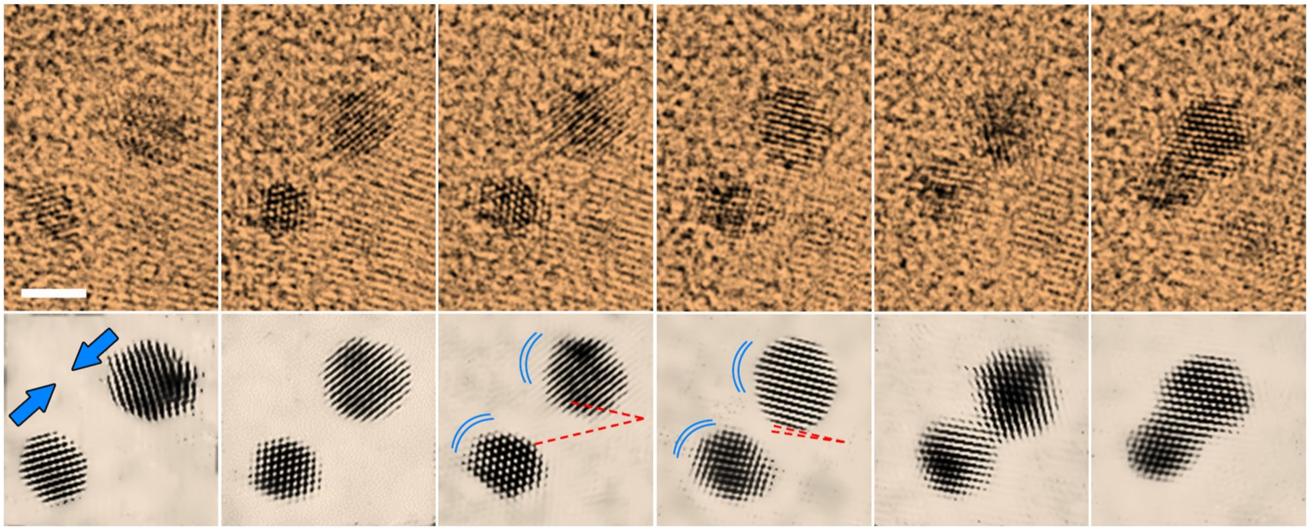

Figure 3. Oriented attachment of Au nanoparticles at {111} surface, evolving into a twin structure. Scale bar is 2 nm