In-situ Transmission Electron Microscopy with a case-study: cation exchange reactions between nanoparticles

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Abstract

A critical requirement for time-resolved transmission electron microscopy (TEM) is the ability to stimulate and excite a material so that the response can be imaged and recorded in real time: it's the *in-situ* TEM. To realize this aim, additional capabilities for stimulating the material require either incorporating a mechanism within the limited volume available in the sample holder or modifying the microscope itself.

In-situ specimen holders for heating, cooling, deforming, and indenting a material, as well as for applying magnetic or electric fields or exposing the sample to a gaseous or liquid environment, have been then increasingly developed and made commercially available in the last years, allowing to perform in-situ TEM studies that so far have never been feasible.

After a general introduction about some of the most recent and fascinating developments of *in-situ* TEM, as a case study I report the direct use of nanocrystals as source of cations in local cation exchange reactions, activated by *in-situ* thermal annealing. Cu_2Se nanocrystals, deposited on an amorphous substrate, were heated following an *in-situ* TEM approach, and underwent partial loss of Cu atoms. These atoms were then engaged in local cation exchange reactions with Cu "sinks" represented by CdSe rod-shaped nanocrystals. This treatment slowly transformed the initial CdSe nanocrystals into $Cu_{2-x}Se$ nanocrystals, with complete Cd sublimation, and partially of Se atoms. Since both Cu "donor" and "acceptor" particles were rarely in direct contact with each other, the gradual transfer of Cu species from the Cu_2Se to the CdSe nanocrystals was mediated by the substrate.

This type of cation exchange reactions, activated and followed using the TEM *in-situ* approach, is much different from the "classical" reactions taking place in the liquid phase: the very dissimilar nature of the environment where the reactions take place makes possible direct monitoring of the process, allowing single particle tracking with high spatial resolution. Also, thermally activated exchange reactions involving NCs deposited on a substrate take tens of minutes instead few milliseconds to complete. The *in-situ* TEM approach to the study of the solid-state exchange reactions could then open new perspectives in identifying the intermediate states of such transformations. In principle, the approach reported here should be further applicable to many other combinations of materials and could pave the way to methods for the modification of chemical composition, crystal structure and physicochemical properties of materials at a local scale.