In-Situ TEM Investigation of Dynamic Processes and Chemical Reactions

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Understanding the formation mechanisms of nanostructures is crucial for gaining control over the synthesis process to design functional materials with defined shapes, crystal structures, and interfaces.[1] Tailoring these properties is essential for enhancing, e.g., the performance of catalysts. Common approaches for characterizing the synthesis products are based on ex-situ measurements, which can lead to misinterpretations of the synthesis mechanisms. Therefore, the development of in-situ characterization techniques is critical for progressing in this field.

Transmission electron microscopy (TEM) is a powerful tool to study nanostructures on an atomic level and perform detailed analyses of the specimens' local chemical composition. In this study, we use an environmental TEM with the unique ability to perform gas-phase reactions via the integrated metal-organic chemical vapor deposition system.[2] The setup allows for studying the specimen at elevated temperatures and under different chemical environments. Moreover, the supply of reactive metal-organic precursors initiates chemical reactions, studied with high-resolution TEM imaging, chemical analysis tools, and advanced techniques, such as exit wave function reconstruction.[3,4]

Multi-component nanoparticles generated in a spark ablation system are the focus of this work. Our results enlighten and discuss dynamic processes at the interface of a Ag-Cu_3P nanoparticle (Figure 1a) as well as chemical reactions leading to Cu-Cu_3P phase transitions (Figure 1b) and GaP nucleation at the Cu_3P surface (Figure 1c). Detailed analyses allow for drawing conclusions about the driving forces of the observed processes and potential reaction pathways.

The results highlight the potential of electron microscopy to create a deeper understanding of fundamental mechanisms involved in the formation of nanomaterials. Progress in this field will allow for designing and tailoring functional materials with enhanced performances.

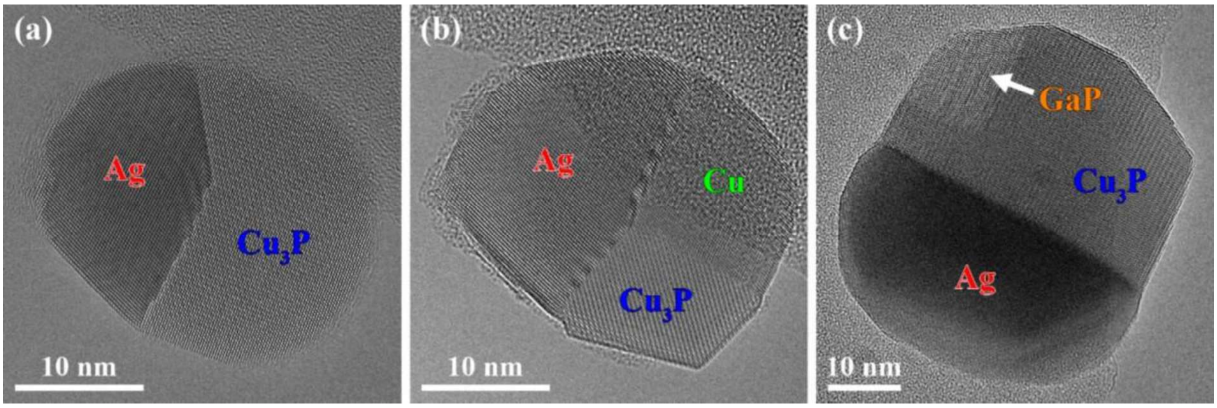

Figure 1: (a) Dynamic processes at the Ag-Cu_3P interface. (b) Phase transition from Cu to Cu_3P. (c) GaP nucleation at the Cu_3P surface with subsequent growth promoted by the counter-diffusion of cations.

[1] S. Bai, et al. *Adv. Sci.* **2017,** *4*, 1600216.

[2] M. Tornberg, et al. *Microsc. Microanal.* **2022,** *28*, 1484.

[3] M. S. Seifner, et al. *J. Am. Chem. Soc.* **2022,** *144*, 248.

[4] M. S. Seifner, et al. *ACS Nano* **2023,** *17*, 7674.