ISSRNS 2025

XVI International School and Symposium
on Synchrotron Radiation in Natural Science
Poland, 25-30 May 2025

Visualizing the Competition between Etching and Sulfurization via *in situ* X-ray ptychography and X-ray Absorption Spectroscopy

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Chemical transformations, such as redox processes or anion and cation exchange reactions in nanoparticles, are crucial for tailoring their functional properties in catalysis and energy applications. In this study, we employ a multimodal X-ray approach to investigate the transformation of Cu$_2$O nanocubes into CuS in the liquid phase. In situ X-ray ptychography and nano diffraction reveal the morphological and structural transformations of the particles during sulfurization, showing the formation of hollow CuS structures. Complementary in situ high- energy resolution fluorescence detected X-ray absorption spectroscopy (HERFD-XAS) demonstrates that, at elevated temperatures, sulfurization proceeds through a direct conversion of Cu$_2$O to CuS. At room temperature, however, a competing etching reaction dominates, leading to the dissolution of Cu$_2$O and the formation of a solvent-coordinated copper complex.

Our results provide insight into both reaction pathways, emphasizing the influence of temperature and reaction conditions on the competing processes. This work highlights the power of multimodal and complementary X-ray techniques for in situ experiments in solution and offers new perspectives on the design and synthesis of metal chalcogenide nanoparticles from their precursors.

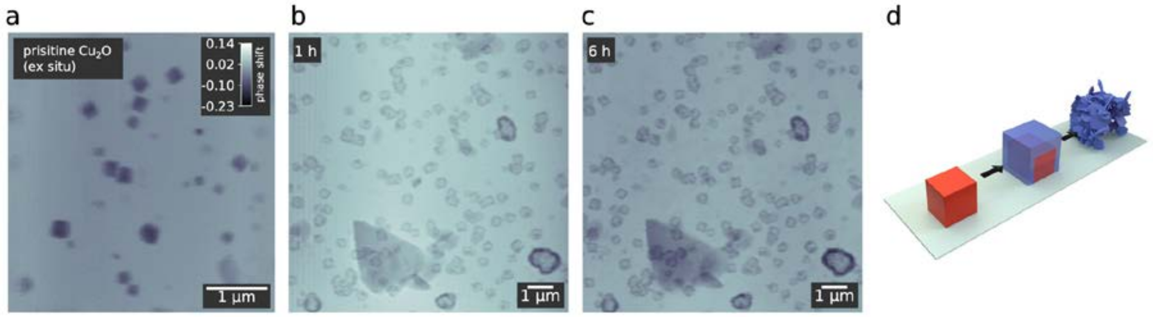

Figure 1. Ptychographic reconstructions of **a** pristine Cu$_2$O nanocubes on a Kapton substrate (ex situ), **b** CuS particles in the reaction solution after 1 h, and **c** after 6 h of sulfurization reaction. **d** Schematic illustration of the morphological changes during heat-induced sulfurization.

Acknowledgements: This research was financed by the Bundesministerium für Bildung und Forschung (BMBF) within the Röntgen-Ångström-Cluster via the project 05K2020-2019-06104 XStereoVision (grant no. 05K20GUA), the European Research Council (LINCHPIN project, grant no. 818941) and the Deutsche Forschungsgemeinschaft (DFG) through the Cluster of Excellence “Advanced Imaging of Matter” (EXC 2056, project ID 390715994).