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Unraveling the mechanism of iron sulfide nanosheet formation: insights from in situ X-ray diffraction and photon-in photon-out spectroscopic studies

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The ability to control the morphology of colloidal nanoparticles is fundamental in materials science, as their shape directly determines their physical and chemical properties. Understanding the processes that guide the formation of nanoparticles into a specific shape is crucial for bridging the gap between synthesis and application. Here, we investigate the mechanism governing the formation of crumpled Fe3S4 nanosheets in solution. We developed a unique colloidal synthesis at 180 ℃, which we monitored using a combination of in situ synchrotron X-ray methods. In situ powder X-ray diffraction analysis reveals the formation of FeS as a crystalline intermediate of the reaction. Owing to its layered structure, this intermediate nucleates anisotropically into 2D nanosheets and subsequently transforms into Fe3S4, while partially preserving the nanosheet-like structure. Additionally, in situ high energy resolution fluorescence detected X-ray absorption near edge structure (HERFD-XANES) and valence-to-core X-ray emission spectroscopy (vtc-XES) enable us to monitor in real-time the evolution of the electronic structure during the reaction. By integrating experimental and theoretical data, we identify distinct components along the reaction pathway: precursor, molecular intermediate, crystalline intermediate, and final product. Initially, the Fe(acac)3 precursor reduces and coordinates with solvent molecules to form [Fe(acac)2(benzyl alcohol)2] complex, which converts into FeS and ultimately into Fe3S4. We capture the transition from oxygen- to sulfur-based coordination. Therefore, we demonstrate that the combination of in situ X-ray diffraction and spectroscopic methods provides key mechanistic insights into the formation of 2D nanostructures, offering valuable understanding of transition metal sulfides with relevance not only to materials science, but also to geochemistry and mineralogy.

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