Unraveling Hydrogen-Induced Phase Transitions in Palladium Nanoparticles via In Situ BCDI

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Abstract

The advent of 4th-generation x-ray light sources offers transformative opportunities for conducting *in situ*/*operando* studies on the structural evolution of nanoparticles in reactive liquid or gas environments. In this study, we demonstrate how Bragg Coherent x-ray Diffraction Imaging (BCDI) [1–3] enables direct probing of the strain fields, lattice deformations, and defect dynamics within palladium (Pd) nanocrystals under gas-phase hydrogenation reactions.

Palladium's unique hydrogen absorption properties underpin its widespread applications in water electrolysis, catalysis, hydrogen storage, and hydrogen sensing technologies [4]. However, key questions remain about the fundamental mechanisms governing hydrogen uptake, hydride nucleation, and the nanoscale dynamics of the hydrogen-poor (α) to the hydrogen-rich (β) phase transition in Pd crystals.

We systematically investigate the interplay between hydrogen partial pressure and lattice response at room temperature, using *in situ* BCDI, resolving strain and lattice parameter distributions with nanometer precision. The measurements have been performed at the ID01 beamline of the ESRF synchrotron. By tracking the kinetics of individual Pd nanoparticles during the α-to-β phase transition, we investigated how the particle responds structurally to varying hydrogen partial pressures. Our results reveal how lattice strain, defects, and phase nucleation evolve locally across the particle during the α–β transition, providing insights into hydrogen absorption dynamics at the nanoscale as shown in Figure 1. Our analysis addresses open questions regarding hydride nucleation mechanisms whether they initiate preferentially at defect or strained sites or uniformly across the nanoparticle surface and elucidates whether the α/β phase transformation occurs through sharp transitions or two-phase coexistence.

This work demonstrates the capability of BCDI to visualize structural dynamics in reactive environments, advancing our understanding of phase transformations in Pd systems and laying the foundation for optimizing Pd-based materials for energy conversion and storage applications. These insights also contribute to broader efforts in nanomaterials science to correlate nanoscale structure and functionality under operating conditions.

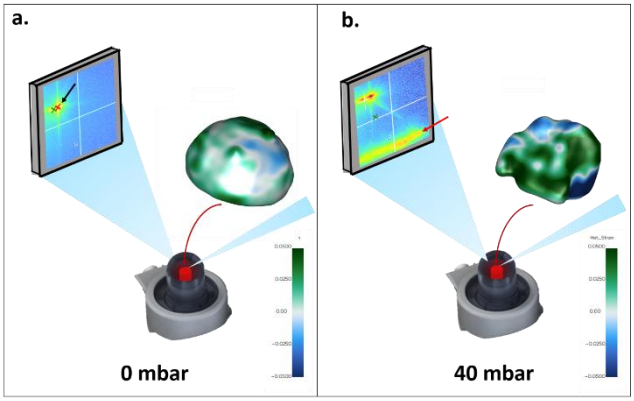

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Figure 1: Scheme of the furnace and BCDI technique, along with reconstructed particles under different applied hydrogen partial pressures. (a) Particle at 0 mbar, where only the α phase (indicated by the black arrow on the left detector) is present. (b) Same particle at 40 mbar, showing the coexistence of both alpha and β phase (red arrow on the right detector) simultaneously.

References:

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