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Experiment Brief

K3 IS Camera

Title

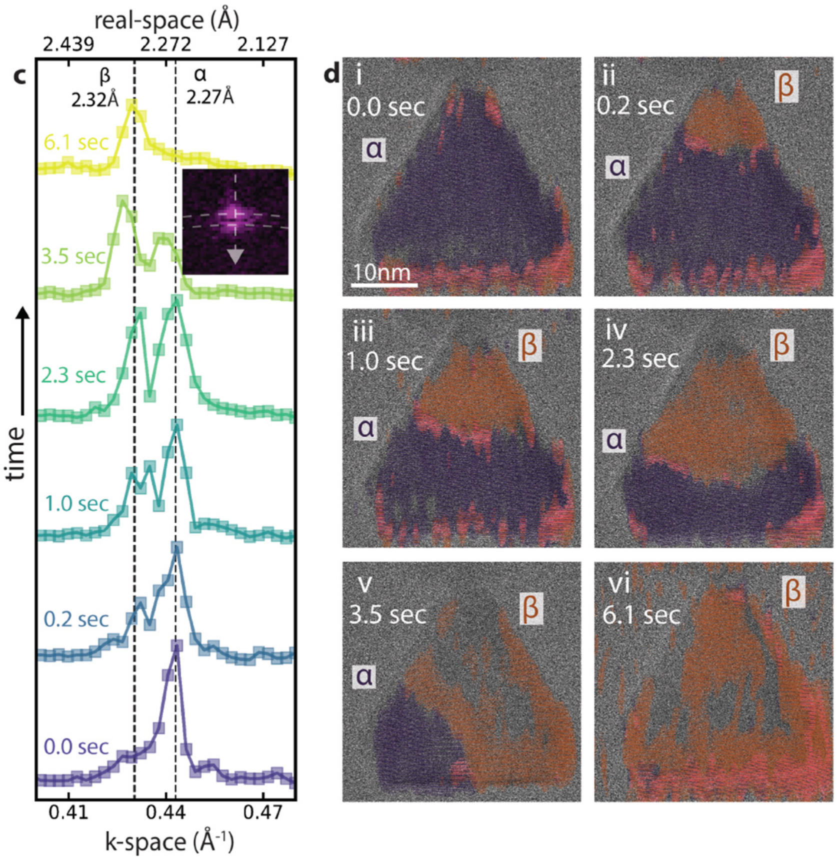
In-situ lattice-resolution imaging of hydrogen absorption into nanoparticles

Gatan Instrument Used

The K3® IS camera delivers simultaneous **low-dose imaging via real-time electron counting**, fast continuous data capture, and **a large field of view (FOV)**. The model 636 double tilt liquid nitrogen cooling holder enables imaging of radiation-sensitive frozen samples and **observation of low-temperature *in-situ* phase transitions at high resolution** with precise temperature measurement.

Background

Palladium can efficiently split H_2 and reversibly absorb large amounts of hydrogen, but pure Pd has some undesirable properties which can be mitigated by alloying with other metals like Ag. While ensemble or bulk studies of Pd hydrogenation have been performed direct observations of the movement of phase fronts in individual nanoparticles were achieved, enabling insights into the thermodynamics, nucleation, and dynamics of hydrogenation for precisely known particle geometries.



Materials and Methods

Here, 0.5% Ag in Pd triangular nanoprisms were observed using a K3 IS camera in a Titan ETEM with image correction operating at 300 kV. The sample was cooled to -30 °C with a model 636 holder, and the H_2 pressure changed in 10 Pa steps to initiate the hydrogenation or dehydrogenation of individual particles. The video was recorded at 26 fps with a dose rate of 530 $e^-/Å^2/s$. Data was analyzed frame-by-frame via Fourier filtering to identify the regions matching the α and β phases. A few frames are shown in Figure 1 (Figure 3 in [1].), but the entire video is available here. Drift correction was performed in post-processing, leveraging the camera's large FOV and speed.

Summary

An *in-situ* transmission electron microscope video captured with the K3 IS enabled direct atomic-scale visualization of the movement of phase fronts through an individual nanoparticle [1]. Frame-by-frame analysis revealed an abrupt reorientation of the phase front, as seen here in Figure 1d between iv and v. Molecular dynamics calculations showed that this reorientation minimizes strain and is dependent on the exact geometry of the particle.

Figure 1. c) Radial line plots of the FFT through the most prominent peak, as indicated in the inset. d) Selected images frames with Fourier filtered overlays showing the α (purple) and β (red) phases as the hydrogenation takes place. The full video can be found here. Adapted with permission from [1]. Copyright 2022 American Chemical Society.

Credit(s)

A special thanks to Stanford University, including Daniel K. Angell, Briley Bourgeois, Michal Vadai, and Jennifer A. Dionne.

[1] D.K. Angell, et al., Lattice-Resolution, Dynamic Imaging of Hydrogen Absorption into Bimetallic AgPd Nanoparticles, ACS Nano. 16 (2022) 1781–1790. https://doi.org/10.1021/acsnano.1c04602.

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