ESRF	**Experiment title:** In situ Bragg Coherent Diffraction Imaging of Pt nanocrystals of various morphologies in electrocatalytic conditions	**Experiment number**: HC-5163
Beamline: ID01	**Date of experiment**: from: 03/05/2023 to: 09/05/2023	**Date of report**: 11/09/2023
Shifts: 15	**Local contact(s)**: Steven Leake	*Received at ESRF:*

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Report:

This experiment was split between three different sample types. First, we examined palladium nanoparticles coated in cerium in an electrochemical cell. Second, we examined palladium nanoparticles with no coating. Third and finally, we examined platinum nanoparticles in an electrochemical cell. This report will summarize analysis and results on the former two.

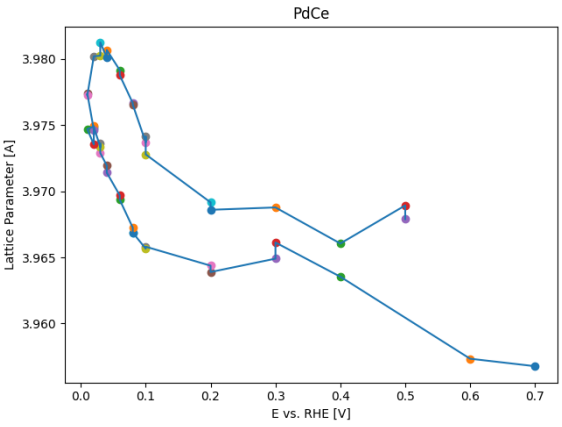

Figure 1: Hydrogen absorption is seen by the increase of the lattice parameter. A large hysteresis is observed.

Palladium nanoparticles were prepared by dewetting and were then coated with cerium. We found, during the beam-time, that the palladium had seemingly alloyed with the cerium coating. A lattice parameter consistent with a Pd-Ce alloy, known from literature, was observed. We are currently doing TEM experiments to analyze the chemical structure. The samples were in an electrochemical cell. We observed during hydrogen loading that the lattice parameter would increase, consistent with pure Pd samples, but we did not observe any phase transition associated with hydrogen uptake, unlike pure Pd. We also observed a hysteresis of the lattice parameter as the hydrogen desorbed, seen in figure 1. Bragg Coherent Diffraction Imaging (BCDI) shows that the heterogeneous

strain inverses as more hydrogen is absorbed. When the cell potential is increased and the hydrogen desorbs, we see a similar hysteresis of the strain. We observed that the strain is determined by the amount of hydrogen absorbed rather than the cell potential, due to hysteresis. With the BCDI reconstructions we area able to separate the surface and bulk strain. We observed that the strain on the surface starts in compression at high cell potentials (low hydrogen partial pressure), while the bulk is lightly expanded. In Figure b we see histograms of the strain separating the surface (blue) and bulk (yellow). At lower potential we see that the strain has inversed and the surface is expanded, while the bulk is compressed.

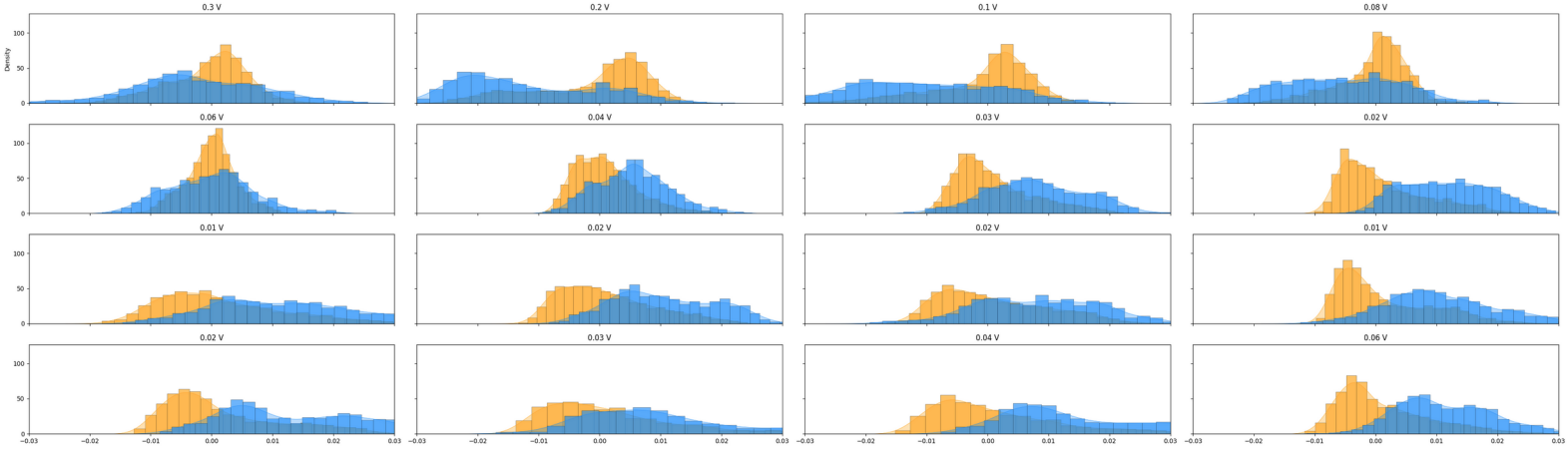

Figure 2: Histrograms of the strain separating the surface (blue) and bulk (yellow). This should be read from left to right as a book, in this order it shows decreasing potential from 0.7 to 0.01[V] for the first three lines. The last line increases to 0.04 [V].

The next experiment looked at time resolved hydrogen absorption in pure palladium. We examined the increase of the lattice parameter of a single palladium nanoparticle by taking quick rocking-curve scans around the 111 Bragg peak. The potential was decreased from 0.7 V and at each potential 5 scans were taken every 60 seconds to track the movement of the Bragg peak. The particle was followed up until the phase transition into the high concentration phase. We observed a an absorption time constant as the lattice parameter approached logarithmically an equilibrium value at each potential. This time constant was seen to be independent of the potential jump. As the potential was decreased the amplitude of the increase steadily grew. This is seen in Figure 3.

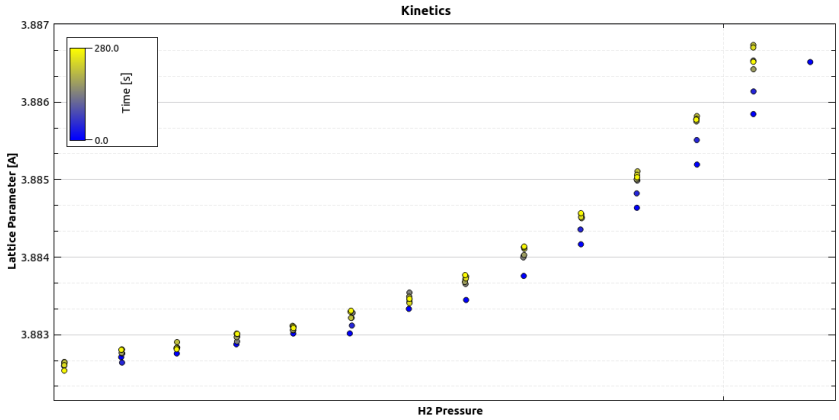

Figure 3: Dynamics of the hydrogen absorption. The time constant is seen to be independent of potential (or equivalently hydrogen pressure). The amplitude of the increase grows with the pressure.