ESRF	**Experiment title:** Rationalization of CO2 and H2 Adsorptions on Ni Alloyed Nanocrystals Using In Situ Bragg Coherent Diffraction Imaging	**Experiment number**: HC4959
Beamline:	**Date of experiment**: **from:** 14/02/2023 **to:** 20/02/2023	**Date of report**: 11/06/2023
Shifts:	**Local contact(s)**: Ewen BELLEC	*Received at ESRF:*

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Report: The goal of this experiment is to study the CO_2 and H_2 adsorption/desorption at the surface of single alloyed Ni crystals using Bragg Coherent X-ray Diffraction Imaging (BCDI) under various in situ (temperature, T and pressure, p) conditions. The 3D intensity distribution in reciprocal space in the vicinity of a selected Bragg reflection contains information about the shape and strain states of the nanostructure, thus revealing insight into the nature of nanoscale deformation following chemical stimuli, such as adsorption of gases or changes in adsorbate structure. Here, we aim at imaging the strain evolution of pure Ni and alloyed NiFe particles as a function of CO_2/H_2 gas pressure (from 100 mbar to 800 mbar) and temperature (from 300 K to 773 K). Ni-Fe particles have been demonstrated as the best candidates for an "all-inclusive" material of choice for the carbon dioxide hydrogenation reaction.

A total of 4 nanoparticles (Ni) were probed for 8 different gas conditions (p_{tot} = 500 mbar : He, H_2 20%, CO_2 5%, 10%, 20%, 30%, 40%, 50%) at 3 different temperatures (350℃, 400℃ and 450℃). For each condition and particle, three rocking curves were measured to ensure data reproducibility. This results in the measurement of more than 600 relevant datasets. A few examples are shown in Figure 1.

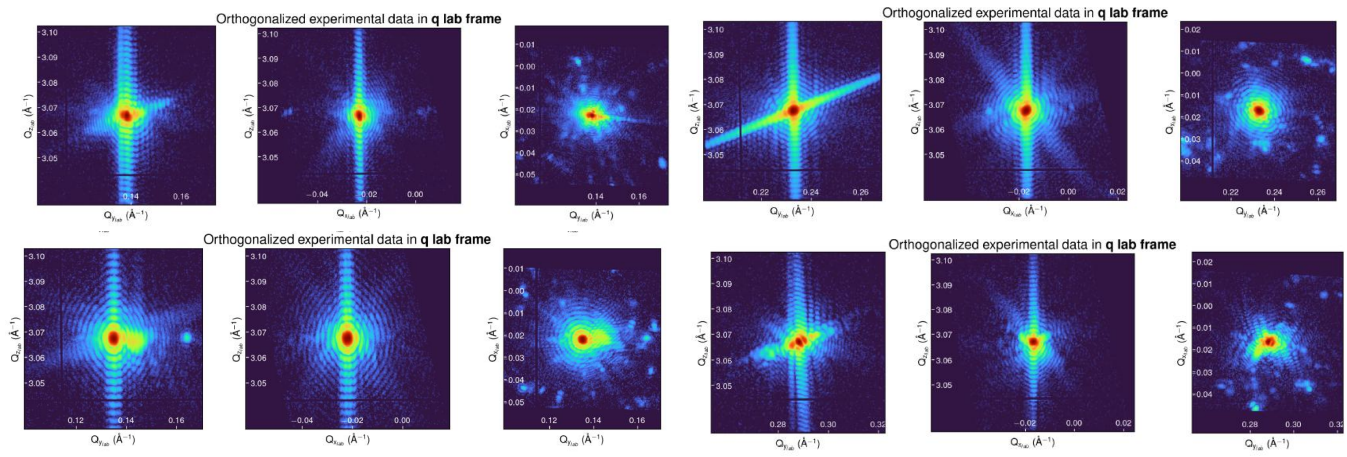
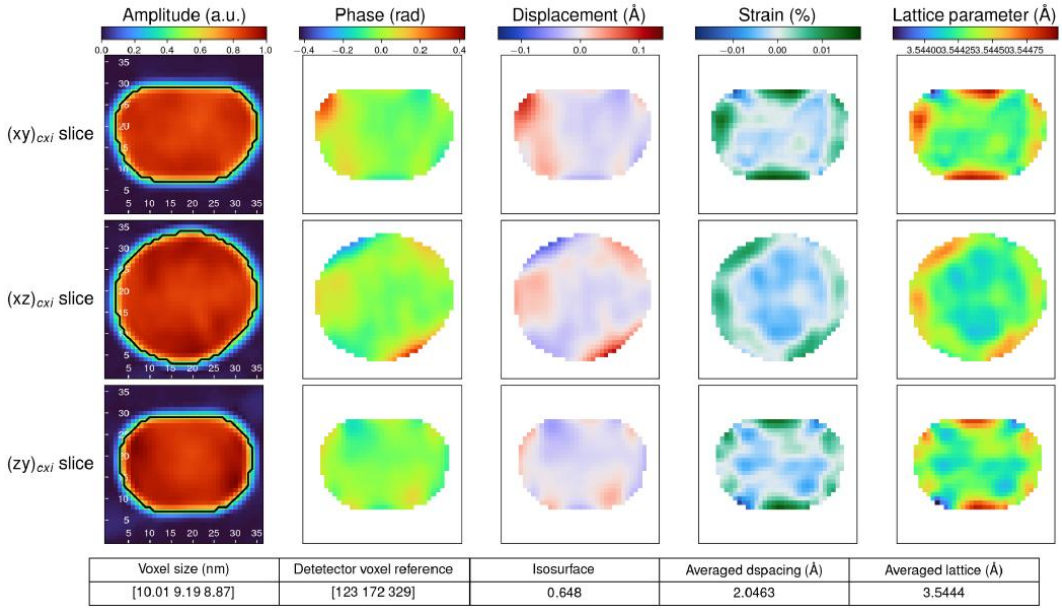


Figure 1 : experimental data of four different particles (under He and 450C)

Thanks to the newly developed cdiutils package (work of Clément Atlan), the on-the-fly data treatment and preliminary analysis has become much more efficient (Figure 2). This allows to quickly obtain the amplitude, phase, displacement, strain and d-spacing fields all at once in a few minutes (for good datasets).



Voxel size (nm)	Detetector voxel reference	Isosurface	Averaged dspacing (Å)	Averaged lattice (Å)
[10.01 9.19 8.87]	[123 172 329]	0.648	2.0463	3.5444

Figure 2 : particle reconstruction showing the amplitude, phase, displacement, strain and d-spacing fields

On the technical side, we encountered some issues related to the sample environment. The PID regulation that ensures the thermal stability (+/- 0.1C) affects the measurement (as shown in Figure 3). The temporary solution was to switch off the thermal regulation during a relevant scan (as shown in Figure 4). This is not ideal as the regulation is necessary to maintain a stable temperature, fluctuations can occur if it is off.

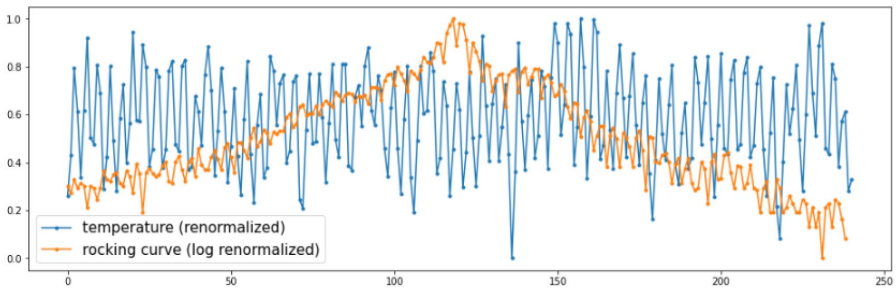


Figure 3 : oscillations in the rocking curve observed during the use of the PID regulation of the sample heater

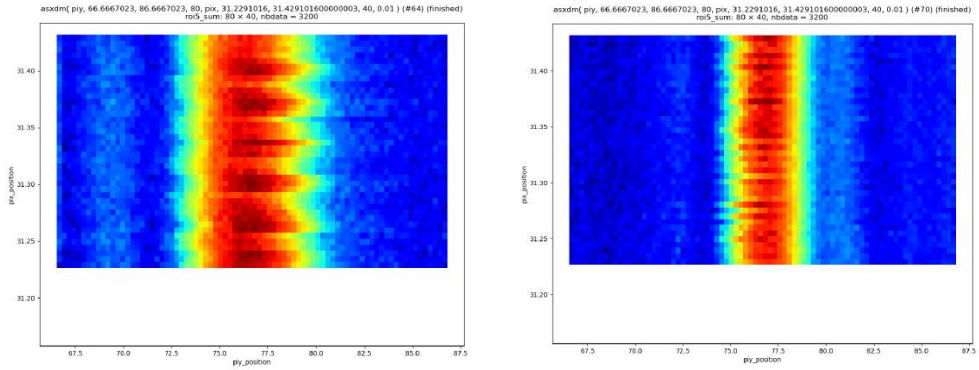


Figure 4 : transverse scan of the particle (left) with and (right) without the PID regulation

The full set of data is still under treatment as the full analysis of each measurement takes a long time and the amount of collected data is huge (600+ relevant scans).