		Proposal number: 02-02-883
Beamline: D2AM	Date(s) of experiment:	Date of report:
	22-28 february 2023	4 April 2023
Shifts: 18	Local contact(s):	Date of submission:
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Objective & expected results (less than 10 lines):		

## Experimental Report template

We performed *in situ* Surface Resonant X-Ray Diffraction (SRXRD) across the  $L_{III}$  edge of Pt and Surface X-Ray Diffraction (SXRD) on Pt(111) in two electrolytic solutions and in the  $\pi$  and  $\sigma$  polarisation, in several points of the repricrocal space. We wanted to specifically investigate the valence state of the surface Pt atoms as a fonction of the electrolyte in specific potential regions, in order to check the sensitivity of the technique to the presence of adsorbed species.

Results and the conclusions of the study (main part):

In 0.1M H<sub>2</sub>SO<sub>4</sub>, we succeeded in measuring the resonant spectra at 90 mV<sub>RHE</sub>, potential position where hydrogen is expected to be adsorbed. The comparison with the data obtained in the previous experiment [1] at higher potential, where no adsorption is present, unambigously show that spectra are influenced by the hydrogen adsorption (see figure 1).

In 0.1M HClO<sub>4</sub> at 440 mV no adsorption is expected. Unexpectedly, the recorded data show that spectra do not overlap (figure 2). It will be particularly interesting to analyze these signals, in order to check the depence of the surface structure from the electrolyte and the role of adsorbed atoms. Data analysis is in progress.





Figure 1 Pt(111)° in 0.1M  $H_2SO_4$ ,  $\pi$  polarisation: 350 mv (no adsorption), 90mv (H adsorbed)

**Figure 2** Comparison of the spectra recorded in the two electrolytes in potential regions where no adsorption is expected

Justification and comments about the use of beam time (5 lines max.):

In situ SRXRD/SXRD experiments in electrochemical environment on ultra-thin metallic layers can be done only with the high brilliance of a synchrotron source.

D2AM is a perfectly suited beam line for *in situ* surface diffraction study in electrochemical environement.