St. Martin d'Hères, 2010-31-08

The last week of the month of June 2010 we realized the *in situ* Surface X-Ray Diffraction experiment number 02 02-765 on the French CRG beamline D2AM.

The aim of this project was to follow *in situ* in electrochemical environment the growth of palladium deposit onto Pt(111) from a fraction of atomic layer up to ten layers.

We used for the first time the "column electrochemical cell" which has been specially designed and built for *in situ* SXRD during electrochemical process, experiments where an important quantity of electrolyte in front of the electrode is needed.

Working at grazing incidence, we firstly and successfully recorded the surface signal of Au(111) (figure 1), showing that the "column" electrochemical cell allows *in situ* SXRD experiments

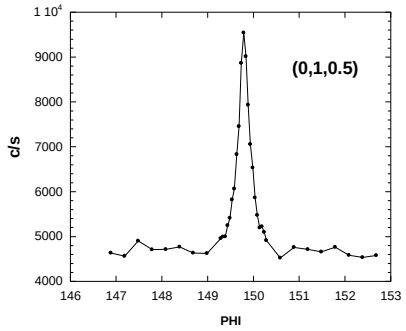

Figure 1: *In situ* measurements on Au(111) (H_2SO_4 0.1 M, 0.7 V *vs.* SCE): rocking scan measured for (0,1,0.5).

To follow the Pd growth, we measured the diffracted signal at the anti-Bragg position (0,1,0.5) as a function of the time (of the deposition rate); Even if the quantitative analysis has just began (the experiment has been made at the end of June 2010), the recorded signals already allow to have some qualitative results.

The diffraction signal recorded for (0,1,0.5) sweeping the applied potential form 0.7 vs. SCE down to 0.4 *vs.* SCE at 0.1 mV/sec (see figure 2) shows a first oscillation, corresponding to the first complete deposited Pd layer, followed by a second one. It seems that a layer-by-layer growth is present for the two first deposited layers. We could verify that the first oscillation corresponds to the UPD peak we observe in the electrochemical deposition.

We recorded the diffraction signal in the (0,1,0.5) position for different chloride concentrations and different deposition rates, showing a strong influence of these parameters on the Pd layers growth mecanismes.

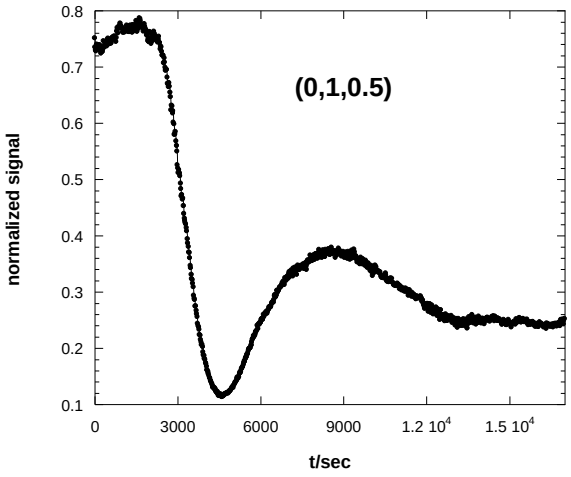

Figure 2: Diffraction signal recorded *in situ* (H_2SO_4 0.1 M + $PdCl_2$ 10-5 M,+ HCl 10^{-3} M)for (0,1,0.5) during a potential sweep from 0.7 V *vs.* SCE down to 0.4 *vs.* SCE, 0.1 mV/sec.