

July 2007, we realized the *in situ* Surface X-Ray Diffraction experiment number 02 02 697 on the French CRG beamline D2AM.

The new electrochemical cell we realized for this kind of experiment, allowing the *in situ* (electrochemical environment) SXRD measurements, was successfully tested.

Working at grazing incidence, we could measure the diffraction intensities for various rods on the electrochemical Pd deposition onto Pt(111). We followed the hydrogen insertion/desorption, recording our data before, during and after H insertion and this for different thickness of deposited palladium (5, 10 and 15 layers). We could then record the surface signals, through 13 μm of electrolyte, allowing the structure determination of the deposit and the modifications occurring due to hydrogen insertion.

We could not yet realise the complete quantitative analysis of our data (data have been recorded last July). Nevertheless, next figures show, besides the goodness of the recorded signals, that even from the qualitative point of view we already can observe interesting phenomena.

The signature of the Pd hydride formation during insertion is shown in figure 1. Clearly seen especially at higher L values, the surface Pd signal ($L \approx 9.1$) is replaced at lower potential value by the Pd hydride peak ($L \approx 8.6$). We remind that the expected variation for the lattice parameter is of about 3%: that's why we had to work at quite high photon energy, in order to reach high L values. Kiessig fringes, well seen at lower L values, disappear at hydrogen insertion.

After insertion (see figure 2), the fringes appear again. Nevertheless, the morphology of deposited palladium seems to be modified by hydrogen insertion, even after only one insertion cycle.

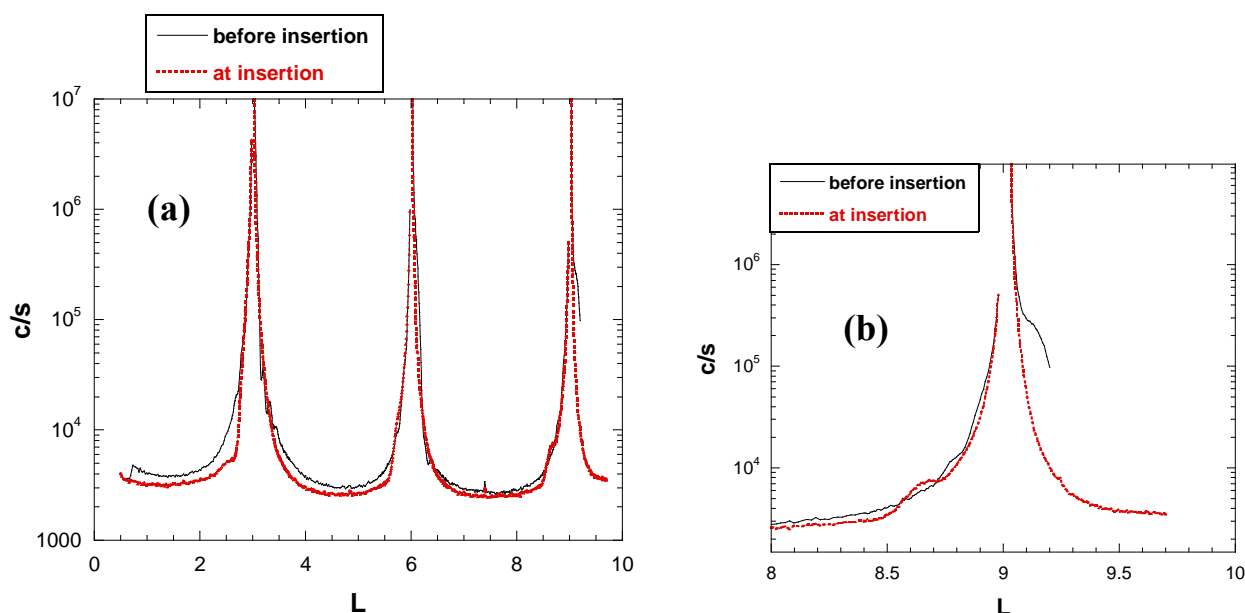


Figure 1: 15 monolayers of palladium electrodeposited on Pt(111). *In situ* recorded diffraction intensities, (11) rod, at -100 mV/ECS (before hydrogen insertion) and at -320 mV/ECS (at hydrogen insertion). (b) is an enlargement of (a)

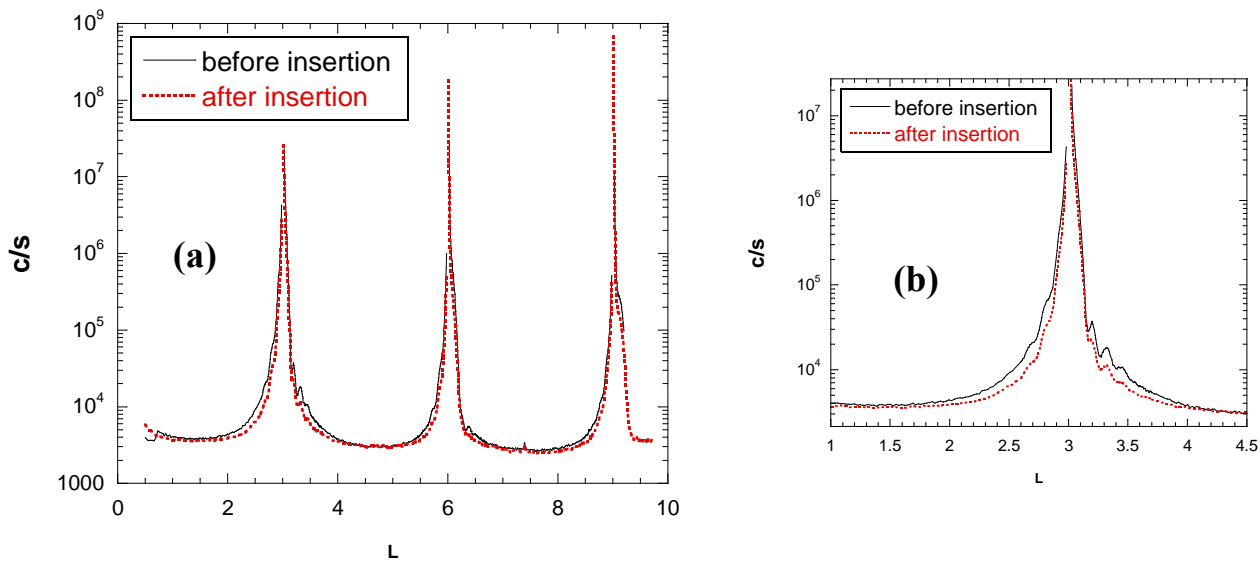


Figure 2: 15 monolayers of Palladium electrodeposited on Pt(111). *In situ* recorded diffraction intensities at -100 mV/ECS, (11) rod, before and after hydrogen insertion. (b) is an enlargement of (a)