Experiment N° SI-2427

Substrate effect of the hydrogen electro-insertion into Pd/Pt(100) nanofilms

In situ SXRD experiments in order to study the hydrogen electro-insertion into Pd nanofilms on Pt(100)° could be made on the French CRG D2AM beam line from June 27 to July 3, 2012. We recorded for two equivalent thicknesses (2ML and 16 ML) non specular [(10), (01), (11), (20) and (02)] as well as specular (00) reflexions before and after hydrogen insertion and after hydrogen desorption. Even if the data have not yet been analysed, preliminary and qualitative considerations are still possible.

In plane (HK) scans show that even for $Pd_{2ML}/Pt(100)$ the insertion/desorption cycle induces irreversible structural modifications (figure 1). Such result already shows the influence of the substrate in the behaviour of the Pd film upon cycling. Indeed, for the same equivalent thickness but with Pt(111) as a substrate we could previously reveal that structural modifications induced by hydrogen insertion are reversible ^[1].



Figure 1: $Pd2_{ML}/Pt(100)$ in 0.1M H_2SO_4 : k scan across the (20) CTR *in situ* recorded before and after hydrogen insertion and after hydrogen desorption.

The same conclusion seems to be shown by the data obtained for a thicker film, $Pd_{16ML}/Pt(100)$. In this case, the Kiessig fringes present in the non-specular and specular reflexions before hydrogen insertion, and disappearing after insertion, do not come back after desorption (figure 2). Again we observe here a different behaviour of the Pd/Pt(100) films upon insertion compared to Pd/Pt(111). In this last case, our previous results have shown that the system is characterised by a complete reversibility in the interplanar distances, as revealed by a perfect superposition of the specular signals before insertion and after desorption ^[2].



Figure 2: $Pd_{16ML}/Pt(100)$ in 0.1M H_2SO_4 : in situ specular reflexion recorded before and after hydrogen insertion and after hydrogen desorption.

Quantitative analysis is on the way and results will be published as soon as possible.

- 1) C. Lebouin, Y. Soldo-Olivier, E. Sibert, M. De Santis, F. Maillard, R. Faure, *Langmuir* **25(8)** (2009) 4251
- 2) Y. Soldo-Olivier, M. C. Lafouresse, M. De Santis, C. Lebouin, M. de Boissieu, E. Sibert, *J. Phys. Chem. C* **115(24)** (2011) 12041