ESRF	**Experiment title: Restructuring of the Au(110)-(1x2) surface under CO at elevated pressures studied by surface X-ray diffraction**	**Experiment number**: SI-815
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Report:

The aim of the experiment SI-815 was the observation by surface X-ray diffraction (SXRD) of the evolution of the Au(110)-(1x2) surface structure under elevated CO pressures (1-500 Torr) so that it could be compared with structural information arising from STM studies performed under the same conditions[1]. The main issue to be adressed by this comparison was the attribution of the structural modification observed by STM to the CO-induced restructuring of gold and/or to the CO adsorbate geometry which cannot be directly done from the STM images.

After having prepared the surface, we clearly found, under UHV conditions, the SXRD signature of the (1x2) of Au(110) through the marked intensity of the (0 ½) rod . Then we performed our first experiment in the range 100 – 500 Torr of CO. Upon CO exposure we observed a (very) rapid decrease of the (0 ½) rod intensity. The rapidity of the phenomenon didn't allow us to perform a clear study of this decrease and afterwards no structural information could be gained, indicating a destructuring of the surface.

We then planned to work at lower pressures in order to slow the phenomenon and be able to follow it in real time. However, our efforts to re-prepare and characterise by SXRD the Au(110)-(1x2) surface were unsuccessfull over most of the single-crystal surface (this point is discussed later). After repeated ion sputtering and annealings we, finally, succeeded to observe the Au(110)-(1x2) structure on a very small region at the periphery of the sample. We were then able to perform two experiments with similar results on this small region under CO pressure of about 0.1 torr. We could clearly observe the decrease over time of the (0 ½) rod intensity together with the increase of the intensities of the (0 1) and (1 0) rods (Fig. 1). We could also record the evolution of (0 1 *l*), (1 0 *l*) and (1 1 *l*) rods as a function of *l* (Fig. 2). This suggests a transition from a Au(110)-(1x2) reconstructed surface to a Au(110) unrecontructed surface. In a previous work, we made the hypothesis that this transition is an intermediate structure of the Au(110) surface prior to the final c(4x4) reconstruction observed by STM under high pressure of CO (500 Torr) [2]. However we cannot exclude a roughening of the surface through a progressive destructuring of the the (1x2) reconstruction.

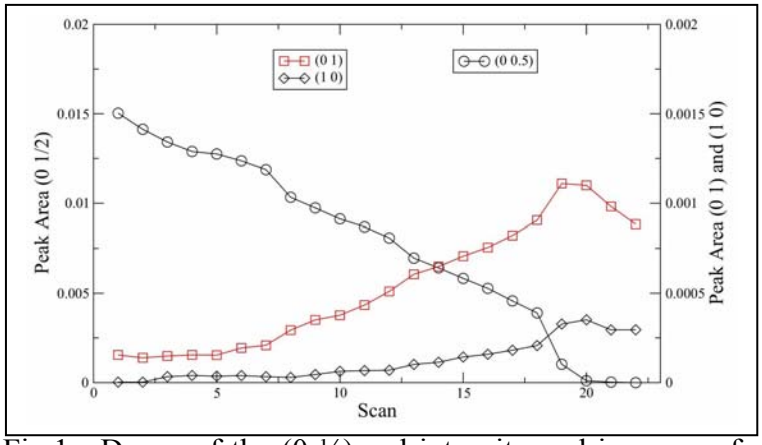

Fig.1 : Decay of the (0 ½) rod intensity and increase of the (1 0) and (0 1) rods' intensities over a period of 3 hours under 0.07 Torr of CO.

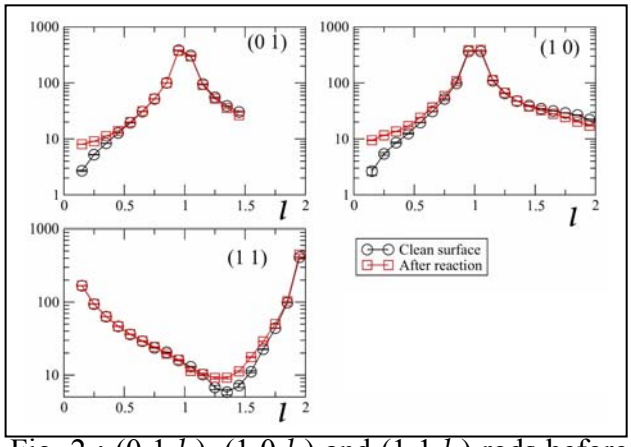

Fig. 2 : (0 1 *l*), (1 0 *l*) and (1 1 *l*) rods before (black) and after (red) CO adsorption at 0.07 Torr.

To understand the problem that we had during the experiment to regenerate the clean Au(1x2) missing row (destroyed by high pressure CO exposure under X-ray beam), we performed a careful characterisation of the surface after the ESRF experiment that yield the following results :

i – the iniatilly optically polished surface became opaque;

ii – AES and XPS showed the presence of carbon and nickel;

iii – a very important roughening of the surface was observed by AFM and SEM (Fig. 3);

iv – EDX in the SEM showed that the grains detected on the surface contained nickel (Fig. 3c).

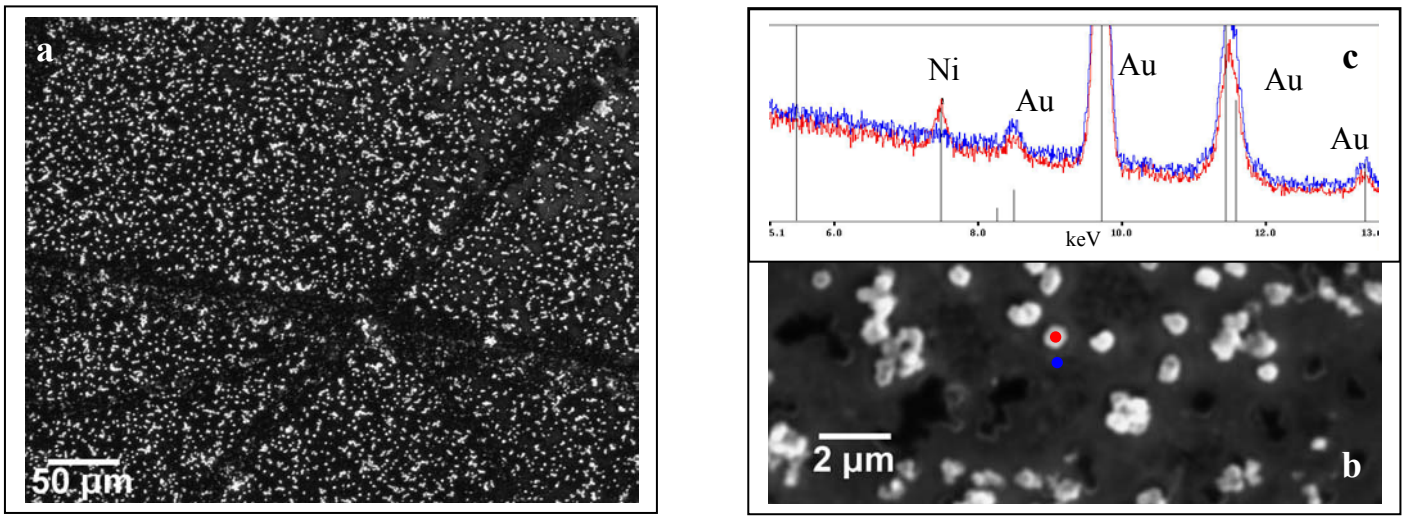

Fig 3 : **(a)** SEM image of the surface for a region close to the centre of the crystal. **(b)** close-up on the grains present on the surface. **(c)** EDX analysis on a grain (red & red dot on **(b)**) and on the surface near the grain (blue & blue dot on **(b)**).

These "post-mortem" analysis of our sample have clearly established that the surface was polluted during the experiment after the first high pressure CO exposure. The presence of Ni and carbon indicates that, most probably, the CO introduced in the chamber has formed Ni carbonyls that were decomposed under the X-ray beam. This could be due to Ni contamination present in the chamber consequently to the methanation experiment ($CO + 3H_2 \rightarrow CH_4 + H_2O$) over Ni surfaces performed in the same X-ray chamber just before our experiments, even if we cannot discard the possibility of Ni carbonyls present in the gas manifold. This leads us to propose that for future experiments on this subject, sufficient time should be allocated to properly clean the chamber and obtain a Ni-free chamber prior to sample introduction in order to avoid such problems. Furthermore, in order to minimize the interaction of the X-ray beam with adsorbed CO and the probable subsequent decomposition of the latter, all the displacements of the goniometer should be made with beam blanking.

[1] Y. Jugnet, F.J. Cadete Santos Aires, L. Piccolo, C. Deranlot, J.C. Bertolini, Surf. Sci. Lett. 521 (1/2) (2002) L639 – L644.
[2] F.J.Cadete Santos Aires et al. (unplubished).