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| ESRF | **Experiment title:**<br>Structural determination of hte $C_{16}H_{33}SH$/Au(111)-c(4x2) surface by Grazing Incidence X-Ray Diffraction | **Experiment number**:<br>SI-483 |
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**Names and affiliations of applicants** (* indicates experimentalists):

**Carmen Ocal***

**Insituto de Cinecia de Materiales de Madrid (CSIC) Cantoblanco 28049-Madrid-Spain**

**Xavier Torrelles***

**Institut de Ciencia de Materials de barcelona (CSIC) Campus de la UAB 08193 Bellaterra (Barcelona) Spain**

**Esther Barrena***

ICMM-CSIC-Cantoblanco 28049-Madrid-Spain


## Report:

The main aim of the SI-483 experiment was to measure the structure of alkanethiol (C16) islands result of the self-assembly of the molecules on Au(111). As it is known, monolayers of thiols onAu(111) present a (√3x√3)R30º lattice modulated by a c(4x2) superstructure [1]. We have demonstrated this is also true for thiol islands [2].

A gold single crystal with (111) orientation (nominal roughness of polishing <0,03µm and orientation accuracy<0,25deg) from Mateck was used for the GIXRD experiments at the ESRF. $C_{16}H_{33}SH$ (C16) molecules form Aldrich (92%) were used as received.

The gold substrate was prepared in UHV by successive Ar+ sputtering plus annealing cycles at 800ºC. Substrate cleanness was first checked in UHV by LEED and AES. Obtaining atomic resolution on the gold terraces by AFM in air was the test for considering the flatness and definitive cleanness of the substrate.

After substrate preparation, the gold single crystal was driven trough air into the C16 solution in ethanol at room temperature. Appropriate immersion times and concentrations led to the formation of thiol islands located at the (111) terraces.

The morphology and characteristics of the sample prepared for the SI-384 experiment was measured by AFM. In that particular case the fresh island size was around ten nanometers in average and the islands covered homogeneously the gold surface (figure 1). Two days after (one day before going to the ESRF) the surface consisted of "aged" islands between 300 and 500nm in size (figure 2). The (√3x√3)R30º (inset in figure 2) and the c(4x2) superstructure existed on the sample.

X-rays can damage the thiol films probably due to the presence of ozone in the sample environment during the experiment [3]. For simplicity, we performed our GIXRD experiments by using a low vacuum chamber [4]. Even in such bad vacuum conditions the four days old sample gave an excellent prove of the viability of the experiment. Both the (√3x√3)R30º and c(4x2) reflections were measured (figure 3)

A simple calculation using the (1/4, 0, 0) reflection characteristic of the c(4x2) superstructure gives an averaged domain size of 420 Å, in excellent accordance with the islands size as measured by AFM in the real space. This means the islands under study are *single domain*. We note the advantages of this fact from the crystallographic point of view. The (√3x√3)R30º reflections were also measured.

With time, damage was observed by the decrease in intensity of the specific reflections. Changing sample position the intensity was recovered indicating that the damage (if due to the presence of ozone) is directly related to the

beam position on the sample. Although the sample was *locally damaged* during the measurements, at the end of the day the sample was destroyed.
As we could not count with an AFM to check the real morphology, size and number of islands in a sample prepared there, we prepared a **complete monolayer** instead.
Only the ($\sqrt{3}x\sqrt{3}$)R30º reflections could be measured due to the low intensity obtained and, surprisingly the width of the (1/3,1/3,0) reflection had increased by a factor of four. Basically this result indicates that the quality of the continuous film is considerably worse than that of the aged islands. The domain size being quantitatively smaller.

**References**

[1] P. Fenter, A. Eberhardt and P. Eisenberger, Science 266, 1216 (1996)
[2] E.Barrena, C. Ocal and M. Salmeron J. Chem. Phys. 111, 9797 (1999)
[3] P. Fenter in "Self-Assembled monolayers of thiols Ed. By A. Ulman Thin Films vol.24. Academic Press (1998)
[4] a small semispherical Be window mounted in a mini-conflack flange and evacuated by means of a membrane pump.