In-situ X-Ray Analysis under Controlled Potential Conditions: An Innovative Setup and its Application to the Investigation of Ultrathin Films Electrodeposited on Ag(111)

Francesca Loglio^a, Maria Luisa Foresti^a, Andrea Pozzi^a, Fabio D'Anca^b, Roberto Felici^b, Francesco Borgatti^b, Massimo Innocenti^a, Emanuele Salvietti^a, Anna Giusti^a, ^aDepartment of Chemistry, University of Florence, via della lastruccia,3 50019 Sesto F.no Firenze. ^bOGG-INFM c/o ESRF, BP220, F-38043, Grenoble, France. E-mail: logliof@unifi.it

An innovative setup to combine electrochemical and in-situ Surface X-ray Diffraction (SXRD) measurements is described. The electrochemical cell has a different design from the other ones commonly used in the beamlines. In particular, the cell arrangement allows the sample surface to stay completely immersed into the solution under controlled potential conditions even during the SXRD measurements. The X-ray beam goes through the liquid (about 1 cm) and the cell walls. However, because of the high X-ray energy, the beam attenuation is negligible and because of the detector arm slit positions, the diffuse scattering induced by the liquid is still low enough to detect the minima of the Crystal Truncation Rods (CTRs). The cell is connected to a special circuit for the alternate fluxing of the electrolyte solutions in the cell. The whole setup can be remotely controlled from outside the experimental hutch by a dedicated computer. The first measurements obtained on S films deposited at underpotential, and on CdS films of increasing thickness are reported.

Keywords: *in-situ* analysis, surface x-ray diffraction, electrochemical cell