	Experiment title: Surface x-ray diffraction structural characterization of CdS electrochemical growth on Ag(111)	**Experiment number**: SI-795
Beamline: ID32	**Date of experiment**: from: 16-May-2002 to: 22-May-2002	**Date of report**:
Shifts: 18	**Local contact(s)**: Dr. S. Warren	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

F. Borgatti*, INFM-OGG

R.Felici*, INFM-OGG

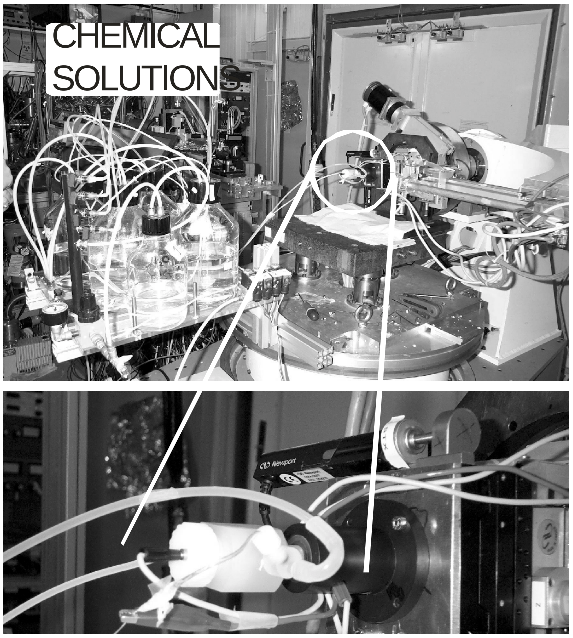
L.Foresti*, Dip. Chimica, Universita' di Firenze

M. Innocenti, *, Dip. Chimica, Universita' di Firenze

F. Loglio*, Dip. Chimica, Universita' di Firenze

Report:

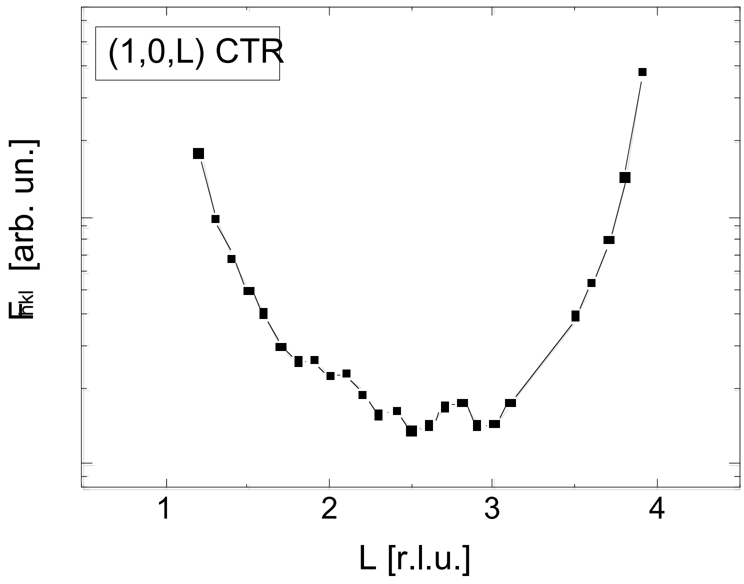
The scope of the experiment was the structural characterisation of CdS film growth on Ag(111) controlled by electro-chemical atomic layer epitaxy (ECALE) method. To this aim we employed Surface X-ray Diffraction technique to gather information about the structure of the adsorbed species and the substrate relaxation at different stages of the film growth. We performed both *in-situ* and *ex-situ* measurements on CdS films produced during the beamtime. The substrates were Ag(111) single crystals aligned better than 0.5°. Our results can be summarized in:



1. In this experiment we used for the first time an innovative setup for the electrochemical cell and

the complex procedures needed to grow alternate S/Cd layers, and by the exigency of performing in-situ SXRD measurements under controlled potential conditions. The electrochemical cell has a different design (shown in the lower panel of the figure), from the other ones commonly used in the ID32 beamine. This 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 cell walls. However because of the high x-ray energy the beam attenuation is negligeable 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 CTRs. A special circuit for the alternate fluxing of the electrolytes solutions in the cell has been mounted on the diffractometer (upper panel of the figure). The solutions are pumped into the cell by a nitrogen-compressed system. The whole setup can be remotely controlled from outside the experimental hutch by a dedicated computer.

2. We measured in-situ the (1,0,L), (1,1,L), and (2,0,L) Crystal Truncation Rods (CTRs) for the clean Ag(111) and different thicknesses of CdS. Here we show the (1,0,L) for the clean Ag(111. For coverages of a few S-Cd bilayers we do not observe any significative change from this curve. This can be explained assuming a lower coverage than expected. Meaning that instead of depositing one layer of S or Cd at the time, we deposite a fraction of monolayer. A quantitative analysis of the data will allow us to determine exactly the coverage.



3. For thick films, nominal coverage 100 S-Cd bilayers, we confirm that the SCd has a wurtzite strucure with the hexagonal basis parallel to the Ag(111) surface. The basis is rotated by 30° with respect to the surface substrate lattice, as shown in the picture reporting an azimuthal scan around the surface normal. The Ag peaks are crystal trunaction rods belonging to the substrate while the CdS peaks are the (10-12) Bragg refletion of the wurtwite structure. The CdS peak intensity is quite low because the