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Report:

1. Background and Experimental

Ultrathin magnetic layers are important devices from a fundamental as well as from a technological viewpoint. Among specific properties, perpendicular magnetization anisotropy (PMA) is one phenomenon of strong interest for high density data storage. Many different systems exhibit interface driven PMA, for instance PMA at Co/Au(111) layers mainly arises from a modification of the magnetic moment of cobalt atoms due to their hybridization with gold atoms. To be observable PMA requires, in this peculiar case, cobalt layers that are thinner than 5-6 cobalt atomic planes and the method of sample preparation is key in determining the inner structure of the deposited metal layers and the morphology of the interfaces, e.g. roughness, intermixing etc. In this respect electrochemistry stands as a powerful method for preparing epitaxial films and nanostructures over large surfaces [2]. Initial studies have shown that Co/Au(111) layers



Fig. 1: Principle of the EC-cell. The Au(111) crystal is held by Vacuum pumping. Its height may be adjusted. The polymer window Is in position (1) after mounting the sample, which allows controlled Electrochemistry on the surface (edges are however not protected). X-ray measurements are performed with the window in position (2), obtained by lowering the pressure in the cell. Additional inlets / outlets And electrodes are not represented.

can be grown from a modified Watts bath [3] and the method was extended to produce thin Au/Co bilayer structures. In our experiment we followed this procedure to monitor the structure of the electrodeposited Co films using surface x-ray diffraction.

The Au(111) surface was prepared by flame annealing in a butane flame before being transferred to the x-ray electrochemical cell for cyclic voltammetry (CV) and surface x-ray diffraction (SXRD) measurements. The modified Watts bath electrolyte contained 0.5 mM CoSO₄. Standard hexagonal units are used for the Au reciprocal lattice whereby H and K lie in the surface place and subtend 60° and the L axis is along the surface normal direction. A schematic of the new cell design used in this experiment is shown in Figure 1. The key to the operation of this cell, as an improvement to the standard thin layer cell was that it was possible to change the thickness of the solution above the Au(111) single crystal surface.

2. Results and Discussion:

2.1 Electrochemistry:

Figure 2 shows a CV of the Au(111) sample in the x-ray electrochemical cell with the polymer window in position (1) (see Fig. 1). It resembles that obtained in a regular electrochemical cell. The reduction of protons (-1.1 V), deposition of Co bilayer and multilayer (U < -1.24 V) can all be seen. Prior to x-ray measurements several films were grown and stripped off to determine the deposition time suitable to obtain the desired film thickness average over the whole crystal, including its side edges. There remains therefore some difficulty to precisely estimate the exact layer thickness deposited on the top surface of the crystal. After deposition, the polymer window was set in position (2) (Fig. 2) for X-ray characterizations.



Figure 2: CV of Au(111) in 0.5 mM CoSO₄.

2.2 SXRD data for *bare* cobalt layers:

The X-ray data confirm the 2D growth modes observed by STM and revealed some *lateral* strain relaxation in the course of deposition **Figure 3** shows some typical results obtained during the experiment. The Co film was deposited to a nominal thickness of 2 ML (with the polymer window in position 1). It took however several attempts before a uniform controlled deposit was observed. The data shown is the specular crystal truncation rod (CTR) and the rod of scattering due to the Co film [measured at (0 1.1.25 L)]. The scattering from the Co film is separated from the scattering from the Au substrate as the Co latice appears to relaxed close to its bulk value in the surface plane although the lattice is aligned with the unerlying Au lattice. Clear oscillations in the specular CTR are observed which is consistent with a uniform deposit. The solid lines in Figure 3 are fits to the data which confirm a near to full coverage of the Co film and give information about the out-of-plane lattice parameter and the Au-Co spacing at the interface. This information is currently being analyzed to try and understand the nature of the Au-Co interface and the physical driving force for the observed epitaxial relationship between the Au and Co lattices.



Figure 3: Specular CTR (left panel) and scattering along (0 1.125 L) (right panel) from a film with a nominal thickness of 2 Co monolayers. The solid line is a fit to the data.

2.3 XRD data upon CO adsorption and potential sweep:

After deposition of 2ML of cobalt, a CO-saturated solution was circulated through the EC-cell to adsorb CO on the surface. The polymer window was mainted in position (1) to key the fill stabilized. The process was monitored by the decay of the EC-current (from the edges). Several experiments were performed. No structural change was detectable (H and L scans) in terms of the in-plane relaxation of the Co film. This might be due to the fact CO adsorption in not uniform. We also attempted to evidence potential induced structural changes on the CO covered surface because our MOKE experiments evidence changes in magnetization anisotropy. Specular L scans were affected by the electrode potential at low incidence angle only (although no changes were detectable at high incidence angle). We infer that this is because the probed region of the electrode (close to its centre) undergoes nearly no (or only partial) potential change due due to a large solution resistance in the thin film of solution above the crystal surface.

In order to properly quantify the effect of CO adsorption and potential on the magnetism of the Co films it will be necessary to perform the experiment with better control over the electric field distribution at the sample surface. This can be achieved by using a droplet electrochemical cell.

3. Conclusions

The above data are encouraging because we have now demonstrated the capability of depositing uniform Co films, *in-situ*, in an x-ray electrochemical cell. Confirmation of this effect, however, requires further experimentation, in particular because it was difficult to control the electric potential in a uniform way across the sample surface. This problem would be overcome with the use of a droplet electrochemical cell.