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18	Christophe Gauthier	

Names and affiliations of applicants (* indicates experimentalists):

P. Borthen*, Inst. für Phys. Chemie und Elektrochemie, Univ. Düsseldorf, 40225 Düsseldorf, Germany

M. Brunnbauer*, Inst. für Anorg. und Angewandte Chemie, Univ. Hamburg, 20146 Hamburg, Germany

E. Garnier*, Catalyse en Chimie Organique, Univ. de Poitiers, 86022 Poitiers, France

H.-H. Strehblow, Inst. für Phys. Chemie und Elektrochemie, Univ. Düsseldorf, 40225 Düsseldorf, Germany

Report: (preliminary)

Our goal was to investigate in situ low dimensional Cu adsorbate structures electrochemically deposited onto single crystal platinum surfaces. For that purpose we planned to use a thin film electrochemical cell with a thin electrolyte solution layer (about 10 microns) trapped between the electrode surface and a covering polymer foil. The measurements should be performed at grazing incidence in order to enhance the surface sensitivity. Whereas the electrochemical cell worked well, a strong elastic scattering from the foil and the solution layer as well as a spurious instability of the vertical beam position during energy scans made meaningful in situ measurements impossible. Beside having a bad S/N ratio, the spectra in the vicinity of the Cu-K-edge were strongly distorted by spikes caused probably by some imperfections of the monochromator crystals. For the future work we plan to modify the cell geometry in order to make the set up less sensitive with respect to the beam position fluctuations and parasitic, elastic scattering from the polymer foil and the solution layer as well. In contrast, ex situ measurements with a copper monolayer deposited on the Pt(111) surface (Fig. 1) showed good performance of the beamline under optimal conditions and a sufficient sensitivity for adsorbate monolayer and submonolayer detection. The copper monolayer was electrochemically deposited at +0.3 V vs. SHE (standard hydrogen electrode) in 0.05 M H₂SO₄. After the deposition, the copper containing sulphuric acid solution was replaced by 0.1 M NaOH and subsequently the electrode was removed from the alkaline solution at -0.6 V. The spectrum recorded under ambient conditions clearly shows the presence of the copper monolayer on the platinum surface. The comparision with

the CuO reference compound spectrum revealed that the copper layer is fully oxidized i.e. its oxidation state is +2. The total integration time was about 30 min.

Fig. 1

