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Report:

The purpose of our experiment was to measure *in situ* the time dependent structural changes on electrode surfaces using energy dispersive x-ray absorption spectroscopy in the reflection mode. The observed electrochemical processes should be the very first stages of copper and Pb-oxide deposition on gold surface as well as the first stages of oxide formation under electrochemical conditions. The main problem by the use of the dispersive optics for XAS is the proper calibration of the recorded spectrum i.e the determination of the I_0 -reference spectrum. This calibration has to be done before or after the actual measurement. In that way, any changes in the beam characteristics between these two measurements can strongly influence the resulting spectrum. In order to reduce the time between these two measurment copper deposition was choosen as the first experiment. The electrochemical metal deposition can be easily started by shifting the potential from a value well above the Nernst equilibrium potential to a value below of it. In the former case the electrode surface is free of any adsorbed metal ions and a reference I_0 spectrum can be recorded only some few seconds before the start of the deposition [1]. The electrode size for the *in situ* XAS measurements is limited by the absorption of x-rays in water. In the vicinity of the copper K-edge the useful electrode length is only about 2 mm. This small electrode size and the glancing angle of about 0.2 degrees reduce the useful beam height to only about 10 μm. Unfortunately, this small beam size resulted in a strong fluctuation of

the beam intensity with time. Two consecutive spectra recorded without any changes in the geometry of the system resulted, after their division, in a spectrum with very strong noise and the overall fluctuation of several per-cent, a value well above the desired sensitivity for the detection of the first stages of metal deposition [1]. Due to this instability of the reflected beam we were not able to obtain any meaningful I_0 spectra. The situation did not change after the deposition of a larger amount of copper onto the electrode surface indicating that the problems were due to the small sample size and not the small amount of the electrodeposits during the first stages of the deposition.

[1] P. Borthen, H.-H. Strehblow, *HASYLAB-Annual Report 1997*, p.781