Spatially resolved studies of copper electroplating by scanning transmission X-ray microscopy

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Copper plating is used in various situations from decoration, to computer chips [1], and even in waste water treatments [2]. Furthermore, copper electrolysis is investigated to understand corrosion mechanisms [3]. Traditionally, the deposition mechanism was believed to be a direct 2 electron reduction from copper (II) in solution to metallic copper on the electrode [4]. However, copper (I) species have been observed and the traditional mechanism is thus questioned. To aid in further investigation, spatially resolved spectroscopic signals are desired, ideally with measurements under active electrochemical operation. We are using soft X-ray scanning transmission X-ray microscopy (STXM) for this purpose. STXM provides chemical speciation at 30 nm spatial resolution by measuring localized X-ray absorption signal [5,6]. Active *in situ* electrochemical control in STXM has been demonstrated [7].

We have used Cu 2p and O 1s edge STXM studies to investigate copper plating from 0.01M $CuSO_4$ in 0.1M H_2SO_4 using gold as both working electrode and counter electrode in a 2-electrode system. In addition to metallic copper, a significant amount of Cu(I) was produced at the edge of the working electrode. Results from ex situ and in situ studies will be presented. In-situ studies give insights into the kinetics of copper deposition and the influence of factors such as electrode surface chemistry, electrolytic medium, and the effect of additives on the mechanism and quality of copper plating. [8]

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