

Real Time In Situ Observation of Metal Electro-deposition  
with High-Resolution Microradiology

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We used phase contrast radiography to study the electro-deposition of Zn, Ni and Cu in real time and with high lateral resolution. Using unmonochromatic synchrotron X-rays and an optics-less imaging setup, we were able to obtain real time radiographs with  $\mu\text{m}$  resolution. A miniaturized electro-chemical cell is designed to study the dynamical phenomenon in the Metal electro-deposition process at various electro-deposition conditions in situ. A detailed analysis of the microstructure evolution relates the growth morphology to the different growth parameters – such as the electric current density, the voltage bias, the pH value and the ion concentration. Much different growth morphology, range from film, porous, whisker and dendrite metal deposition. This link is both global and local. Local variations of the metal ion concentration in the electrolyte were also successfully imaged and the density profile is used to compare with the standard theory to explain the phenomenon of metal ion depletion near the electrode. The observation in real time the ion depletion removes a critical obstacle in studying the fundamental impact of this parameter on the growth quality. We also found that metal is deposited on the surface of hydrogen bubbles forming on the cathode. This phenomenon, as shown in Figure where the metal Zn whiskers grows radially from the surface of the hydrogen bubbles, explains common defects found in electro-deposited metal coatings. The unusual metal nucleation on gas bubbles can be explained by a high concentration of  $\text{Zn}(\text{OH})_2$  produced by cathode chemical reactions. The potential application of this technique to study growth with micropatterned electrodes and pulsed electric current is evaluated.

