Bipolar Electrodeposition at the Sub-Micrometer Scale

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In the present study we explore the feasibility of applying Spatially Coupled Bipolar Electrochemistry to the sub-micrometer regime. This is a technique where electrically isolated objects can be interconnected by the application of electric fields and has previously been demonstrated on the millimeter and micrometer scales.

Three experimental designs were explored: annealed Au and Ag films on glass, annealed Ag films on silicon nitride membranes and Ag tubes immobilized within polycarbonate nuclear-track etched membranes. In the evaporated and annealed film part of this study, particles on the order of 20-50 nm were exposed to electric fields up to 2kV/cm for periods up to 180 seconds, in a mixture of toluene and acetonitrile. Plasmon resonance measurements and Scanning Electron Microscopy (SEM) were used to characterize the changes following field application. At all field intensities and times studied the gold particle sub-monolayers did not appear to be discernibly affected. The plasmon resonance absorption of the silver sub-monolayers displayed significant peak broadening after field application. Further experiments using Transmission Electron Microscopy (TEM) analysis of annealed silver films on silicon nitride membranes demonstrated particle agglomeration without evidence for particle interconnection or morphological change. This result suggests that, under these experimental conditions, physical movement of the Ag particles occurs instead electrochemical processes.

In order to prevent particle movement, polycarbonate membranes were used to anchor silver cylinders with diameters of 1 micron, 400 nm and 200 nm. Results from these experiments demonstrated that for Ag in 1:1 toluene/acetonitrile, Spatially Coupled Bipolar Electrochemistry (SCBE) reaches a practical limit for structures between 200-400 nm since the width of the deposit approaches the size of the metal particles. At 200 nm the result is electrochemical migration of the particles, where a commensurate amount of Ag is deposited on one side and dissolved on the other. This size limitation is specific only to SCBE, where structures are required to electro-dissolve, not to the application of bipolar electrochemistry to structures below 200 nm.