

Electrodeposition and Electrochemical Properties of Nanostructured Palladium Films

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Mesoporous metals with regular nanoarchitectures can be prepared using chemical or electrochemical reduction of a metal salt dissolved in the aqueous domains of a lyotropic liquid crystalline phase[1]. In this approach the metal is formed from the lyotropic liquid crystalline phase of a non-ionic surfactant (typically between 30 and 60 wt%) and an aqueous solution of the appropriate metal salt. The resulting H₁-e metals are “casts” of the particular lyotropic phase used so that it is possible to produce mesoporous metals with different pore topologies and with different pore and wall thicknesses depending on the choice of surfactant and template solution composition[1]. H₁-e metal films have very high surface area (in excess of 10⁶ cm²/cm³) and were found to be useful as catalysts in chemical sensors[2], batteries[3] etc. Following the original work on the electrochemical deposition of mesoporous platinum films[1], this approach has been extended in our studies to the deposition of rhodium[4, 5] and palladium[6, 7].

We have shown that we can prepare nanostructured H₁-e Pd films by electrochemical deposition from the liquid crystalline phases of either C₁₆EO₈ or Brij[®]56. These films contain regular hexagonal arrangement of cylindrical pores with pore size diameter of 2.5 nm and wall thickness of 2.5 nm. Electrochemical studies showed that H₁-e Pd films have high electroactive surface areas of the order of 91 m²/g (corresponding to 1×10⁷ cm²/cm³). Cyclic voltammetry with the simultaneous use of the EQCM provided us with the fact that H₁-e Pd when cycled in the oxide region dissolves subsequently with each voltammetric cycle so that mass loss dominates over mass gain. Cyclic voltammetric measurements of Pd deposited from the aqueous solution showed that the charge passed for formation and stripping of the surface oxide layer reached smaller values than for H₁-e Pd. This is based on the obtained high surface area. We also found the hydrogen region of H₁-e Pd in the cyclic voltammetry of 1 M sulfuric acid more clearly resolved than of plain Pd. Due to the very high surface to volume ratio of H₁-e Pd films we can readily distinguish the formation of hydrogen between adsorption and absorption processes. With the use of crystal violet, as an appropriate surface modification, we were able to knock out the H-adsorption on the Pd surface completely. With the remaining peaks in the hydrogen region associated to the α- β hydride phases we studied in detail the absorption process and focused on the characteristics of the phase transition. Because of the fast kinetic for the formation of α- and β hydride phases we noticed clearly resolved peaks in the absorption region and a sharp rise in the H/Pd ratio.

References

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