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Electrodeposition of Indium from a Phosphonium-Based Ionic Liquid

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Electrochemical deposition of indium metal from aqueous electrolytes is challenging as it occurs simultaneously with hydrogen evolution. The evolution of hydrogen can compromise cathodic current efficiency and can cause porosity of the deposited metal, but it can be avoided by using ionic liquid electrolytes. The high thermal stability of ionic liquids allows for high-temperature electrodeposition on a liquid cathode, offering several advantages over a solid electrode, such as easier separation of electrolysis products and the possibility to design a continuous electrowinning process. In this paper, the electrochemical behavior of indium in the ionic liquid trihexyl(tetradecyl)phosphonium chloride (Cyphos IL 101) was studied at 120 °C and 180 °C. Indium metal was electrodeposited on platinum and molybdenum substrates and on a liquid indium cathode at 180 °C. Impurities present in commercial Cyphos IL 101 interfered with the measurement of the electrochemical window, so that it had to be carefully purified. The electrochemistry of indium in Cyphos IL 101 was studied by cyclic voltammetry, QMCB, rotating disk and rotating ring disk voltammetry. InCl_3 concentration between 10 and 400 mM in Cyphos IL 101 were used in this study. Therefore, the major indium(III) species in solution corresponds to the $[\text{InCl}_6]^{3-}$ complex. Using the Levich equation, a diffusion coefficient of $(7.5 \pm 0.1) \cdot 10^{-12} \text{ m}^2 \text{ s}^{-1}$ at 120 °C was calculated for $[\text{InCl}_6]^{3-}$. It was found that indium(III) is the most stable oxidation state in the ionic liquid. This ion is reduced via a two-step process, going from indium(III) to indium(I) and subsequently to indium(0). The disproportionation of indium(I) to indium(III) and indium(0) was clearly illustrated. During cyclic voltammograms, the anodic and cathodic peak potentials was found to depend on the scan rate. As the scan rate increases, the separation between the anodic and cathodic peaks becomes greater, indicating that the deposition of indium is irreversible or quasi-reversible. The indium electrodeposits were characterized by SEM-EDS. When depositing indium on molybdenum substrates, the deposition of spherical indium droplets was observed, even at temperatures substantially below the melting point of indium (see figure). The probable mechanism behind this deposition morphology will be explained.

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