



Electrochemical mechanism of tin membrane electrodeposition under ultrasonic waves

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ABSTRACT

Tin was electrodeposited from chloride solutions using a membrane cell under ultrasonic waves. Cyclic voltammetry (CV), linear sweep voltammetry (LSV), chronoamperometry (CHR), and chronopotentiometry were applied to investigate the electrochemical mechanism of tin electrodeposition under ultrasonic field. Chronoamperometry curves showed that the initial process of tin electrodeposition followed the diffusion controlled three-dimensional nucleation and grain growth mechanism. The analysis of the cyclic voltammetry and linear sweep voltammetry diagrams showed that the application of ultrasound can change the tin membrane electro-deposition reaction from diffusion to electrochemical control, and the optimum parameters for tin electrodeposition were H^+ concentration $3.5 \text{ mol}\cdot\text{L}^{-1}$, temperature 35°C and ultrasonic power 100 W . The coupling ultrasonic field played a role in refining the grain in this process. The growth of tin crystals showed no orientation preferential, and the tin deposition showed a tendency to form a regular network structure after ultrasonic coupling. While in the absence of ultrasonic coupling, the growth of tin crystals has a high preferential orientation, and the tin deposition showed a tendency to form tin whiskers. Ultrasonic coupling was more favorable for obtaining a more compact and smoother cathode tin layer.

1. Introduction

Tin is widely used in electronic industry, food packaging, perfume and paint, and so on, because of its excellent properties, such as high corrosion resistance and non-toxicity. Recent studies have discovered that the tin electrodeposition of porous membrane can improve the electrochemical performance of lithium ion batteries [1].

Tin electroplating research began in the mid-19th century. Two kinds of electrolytes have been used, namely, bivalent tin alkaline and tetravalent tin acidic electrolytes. Alkaline electrolyte is composed mainly of potassium or sodium stannate. Tin can obtain a uniform sediment by using alkaline electrolyte, but it requires high temperature and solution stability. Acidic electrolytes include sulfate sulfonate [2,3] and fluoroborate [4,5]. They are beneficial for avoiding the generation of hydroxyl, but organic additives, such as citrate [6–8], tartrate [9], gluconate [10,11], and chloride [12,13], must be added to suppress the formation of tin whisker. Not only can tin whisker increase the complexity of the electrolyte, but also results in bad morphology [14].

In recent years, ultrasonic has been extensively studied in electrochemical deposition, electroplating, metallurgy, material preparation, and other fields [15]. Chen HM [16] studied the impact of ultrasonic field on platings of tin–bismuth alloy. He found that ultrasound can

produce crystals of uniform density and improve the efficiency of electrodeposition. Chang HT [17] studied lanthanum and cerium separation through solvent extraction in the ultrasonic field and found that ultrasonic is beneficial for separating similar elements. Wang J [18] studied the effect of ultrasonic field on the preparation of superfine tin oxide nanomaterials and concluded that ultrasonic can improve material purity, refine grains, and favor cost reduction.

To investigate the electrochemical mechanism of tin membrane electro-deposition in chloride solutions under ultrasonic field [19–21], the cathodic polarization of tin electrodeposition under different ultrasonic power levels, temperatures, and compositions was investigated through linear sweep voltammetry (LSV) and cyclic voltammograms (CV) at different ultrasonic power levels. The effect of amine non-ionic surfactant on the nucleation and growth of tin in the ultrasonic field was studied using chronoamperometry (CHR).

2. Experimental

Electrochemical experiments were performed in a three-electrode membrane cell (Fig. 1). The anolyte was composed of $100 \text{ g}\cdot\text{L}^{-1} \text{ Sn}^{2+}$ (SnCl_2) and $3.5 \text{ mol}\cdot\text{L}^{-1} \text{ HCl}$ in all experiments, while the catholyte contained $80 \text{ g}\cdot\text{L}^{-1} \text{ Sn}^{2+}$ (SnCl_2) and $1.5\text{--}5.5 \text{ mol}\cdot\text{L}^{-1} \text{ HCl}$. All solutions

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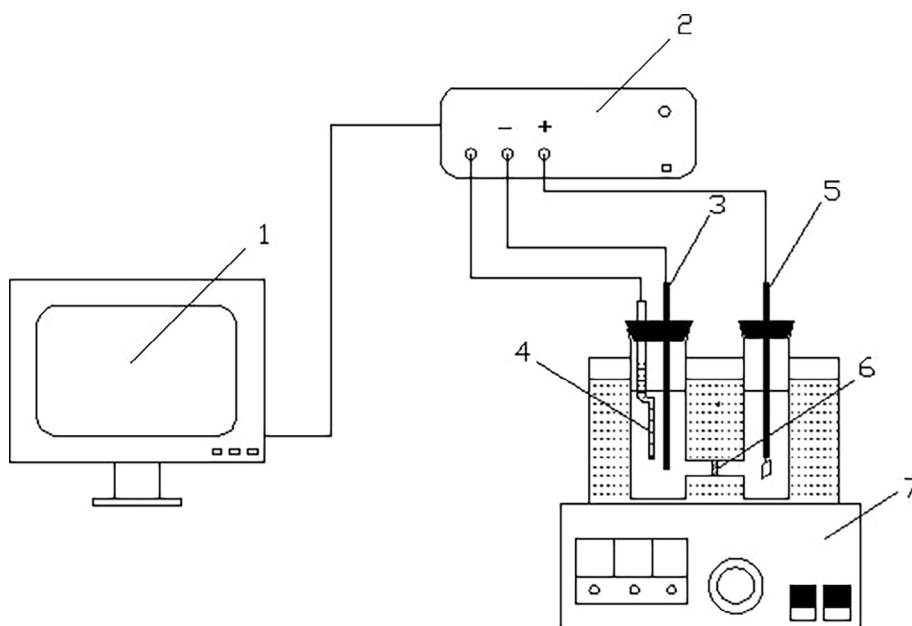


Fig. 1. Schematic diagram of electrochemical testing. 1 – Computer; 2 – Electrochemical workstation; 3 – 316L stainless steel electrode; 4 – Saturated calomel electrode; 5 – Platinum plate electrode; 6 – Anion exchange membrane; 7 – Ultrasonic generator.

were prepared using distilled water with analytically pure reagents. The experiments were conducted at 298–328 K (25–55 °C) with ultrasonic power at 0–100 W and 40-kHz ultrasonic frequency (Ultrasonic cleaning bath provided by Kunshan Meimei Ultrasonic Instruments Company, China) in an oxygen-free electrolyte, which was purged with nitrogen to displace oxygen. All electrochemical measurements were performed using a computer-controlled potentiostat/galvanostat (CHI660C Electrochemical Work Station provided by Shanghai CH Instruments Company, China). The working electrode used was a 316L stainless steel substrate embedded in PTFE (polytetrafluoroethylene) with 3-mm diameter and immersed in catholyte to offer an active flat disc shaped surface of 7.065 mm² geometric area. Before each measurement, the 316L stainless steel electrode was polished using 1200-grit and 3500-grit sandpapers, and then rinsed with ethanol absolute and distilled water. A platinum plate with a surface area of 1.5 cm² was used as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode to which all potentials in this study were based on.

Potentiodynamic polarization curves were scanned at a constant scan rate of 20 mVs⁻¹ and measured with and without surfactant in a membrane cell and in a non-membrane cell at various concentrations of H⁺ from 1.5 to 5.5 mol L⁻¹, at different temperatures from 298 K to 328 K, and at different ultrasonic power levels from 0 to 100 W in 40-kHz ultrasonic frequency. Cyclic voltammograms were registered at various ultrasonic power levels from 0 to 100 W in 40-kHz ultrasonic frequency. The nucleation and growth of tin was measured using chronoamperometry and chronopotentiometry at various constant potentials.

3. Results and discussion

3.1. Linear sweep voltammetry

To investigate the impact of ultrasonic wave on the electrochemical behavior of diaphragm tin electrodeposition, cathodic polarization curve is determined using linear sweep voltammetry at 25–55 °C, H⁺ concentration of 1.5–4.5 mol L⁻¹, ultrasonic frequency of 40, and ultrasonic power 0–100 W.

Polarization curves under a scanning rate 20 mVs⁻¹, H⁺ concentration of 3.5 mol L⁻¹, temperature of 35 °C, Sn²⁺ concentration of 80 g L⁻¹, ultrasonic frequency of 40 kHz, and ultrasonic powers of 0, 20, 40, 60, 80, and 100 W, cathodic are shown in Fig. 2. The analysis of

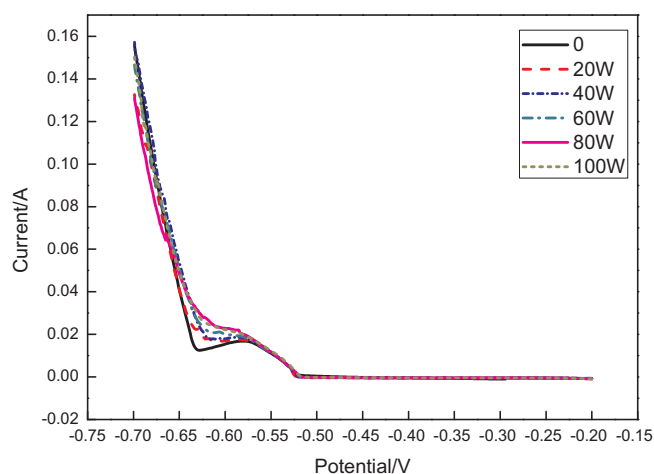


Fig. 2. The cathodic polarization curves under different ultrasonic power.

the law of cathodic polarization curves under different ultrasonic power levels reveals that ultrasonic has a significant impact on tin diaphragm electrodeposition. When not imposing ultrasonic, the current declines significantly when the reduction potential is at -0.58 V. With increasing ultrasonic power, the current decline at -0.58 V continues gradually and finally stabilizes into straight line. This is because the cavitation effect and microstreams of ultrasound can promote the diffusion of Sn(II) in the solution, the acceleration of diffusion, which increases the supply of ions near the cathode, causes changing of the control procedure from diffusion to the mixed diffusion and electrochemistry control [22,23]. Meanwhile, because the diffusion of Sn(II) increases, the capacity of it snatching electron strengthens while the electron snatching capacity of H⁺ in the solution weakens accordingly. This indicates that ultrasonic coupling promotes the electrodeposition of tin.

The cathodic polarization curve at a scanning rate of 20 mVs⁻¹, H⁺ concentration of 3.5 mol L⁻¹, Sn²⁺ concentration on the cathode of 80 g L⁻¹, ultrasonic frequency of 40 kHz, ultrasonic power of 100 W, and temperatures of 25 °C, 35 °C, 45 °C, 55 °C, is shown in Fig. 3. This figure shows that with the rise of temperature, the initial reduction potential of Sn(II) ion (-0.52 V) has no obvious change and the reduction current curve rises slightly. At the initial stage of reaction (tin

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