

Electrodeposition of Mo/MoO_x from dimethyl sulfoxide solutions

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Abstract—Molybdenum (Mo) is a refractory metal used principally as an alloying agent in steels, cast irons, and super alloys to enhance hardness, strength, toughness, wear and corrosion resistance. It is also widely used in catalytic applications, lubricants and pigments. The single electrodeposition of Mo from aqueous solutions cannot be achieved but Mo can be co-deposited as an alloy with iron group metals (induced co-deposition). In this study, the electrodeposition of Mo/MoO_x from dimethyl sulfoxide solutions on a copper substrate has been investigated. Different experimental electrodeposition parameters have been assessed (i.e., supporting electrolyte concentration and addition of water to the electrolytic bath) to analyze their mechanistic influence of induced Mo/MoO_x deposition. Linear scan voltammetry has been used to follow up the electrodeposition of Mo/MoO_x films. Film morphology was characterized using scanning electron microscopy (SEM), compositional analysis was performed using X-ray photoelectron spectroscopy. Mo bearing films were also chemically characterized by ICP-OES analysis. An electrodeposition mechanism was developed and discussed.

Keywords: *electro-reduction, dimethyl sulfoxide, molybdate, deposit.*

I. INTRODUCTION

Molybdenum (Mo) is a refractory metallic element used principally as an alloying agent in steels, cast irons, and super alloys to enhance hardness, strength, toughness, and resistance to wear and corrosion. Moreover, molybdenum is also significantly used in numerous chemical applications, including catalysts, lubricants, and pigments.

The electrodeposition of molybdenum has been attempted in the past by many investigators with little or no success. Electrodeposition of metallic molybdenum from non-aqueous baths (i.e., solutions of molybdenum salts in organic solvents or in liquid ammonia) has also been investigated [1–2].

During the last few years, the use of non-aqueous solvents for a variety of applications has been widely investigated. Some examples are the use of solvents for chemical synthesis [3, 4], media for electrodeposition of metals [5–7], electrolytes for electrochemical devices such as batteries [8], and supercapacitors [9, 10]. Many researchers have succeeded in co-depositing Mo together with iron group metals [11–14]. Previous investigations show that it is not possible to electrochemically deposit molybdenum alone from aqueous systems, the reason being that molybdenum metal ions form complexes with OH⁻ [15, 16]. The objective of the present work is to study the feasibility of using dimethyl sulfoxide (DMSO) and DMSO/H₂O mixtures as electrolytic bath for the electrochemical deposition of molybdenum. Properties of the water–dimethyl sulfoxide system and the density of water–DMSO mixtures have been measured by numerous authors [17–20].

II. EXPERIMENTAL WORK

All reagents used were analytical grade. All salts were dried at suitable temperatures to remove humidity. The ingredients of the electrolytic bath were as follows: ammonium molybdate ((NH₄)₆Mo₇O₂₄), lithium chloride (LiCl), distilled water (DI-H₂O) and DMSO.

Electrochemical measurements were performed in a conventional three electrode Flat Cell Kit (model K0235) using a potentiostat/galvanostat, model VersaSTAT 3 from Princeton Applied Research equipment and VersaStudio software.

The layers were deposited on a copper (99.9%) foil substrate (working electrode), which was prepared by mechanical polishing of the exposed side using fine (P400/600) sand paper. The area of copper surface exposed was 1 cm². A platinum mesh (4 cm²) served as the anode (counter-electrode). The distance between the electrodes was approximately 9 cm. A standard calomel electrode was used as a reference. The cell configuration allows for the