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INFLUENCE OF BATH COMPOSITION ON THE ELECTRODEPOSITION OF MOOX THIN FILM FROM DIMETHYL SULFOXIDE

Abstract

The electrodeposition of Mo/MoO_x from dimethyl sulfoxide has been investigated. The Mo/MoO_x thin films were deposited on copper substrates by the electrochemical method from dimethyl sulfoxide solution. Among the experimental electro-deposition parameters, only the concentration ratio of additional substances to molybdate ions was varied to analyze its influence on mechanism of induced Mo/MoO_x deposition. Voltammetry was one of the main techniques, which was used to examine the voltammetric response, revealing that Mo/MoO_x deposition depended on the nature of the species in solution. When optimal potential is applied, a colored Mo/MoO_x thin film is formed on the electrode. Chemical ICP-OES analysis was used to corroborate the almost presence in these films. Intermediate Mo/MoO_x was characterized using scanning electron microscopy (SEM), compositional analysis was determined with X-ray photoelectron spectroscopy.

1. Introduction

Molybdenum is called refractory metal, is widely used for industrial applications such as heat resistance material because of its high melting point and high strength. However, molybdenum is often used in simple shapes, such as plate or rod, because it is hard to be processed. Although the gas phase methods are used when a complicated shape is demanded, there are some problems in the rate or the uniformity of deposition. Many investigations have been devoted to the electrodeposition of with iron group metals [1-4]. However, the mechanism of molybdenum electrocrystallization is

still not elucidated. Indeed this metal has never been deposited in a pure state from aqueous solutions whereas their codeposition with iron group metals is possible [5].

Several hypotheses have been proposed. The most recent investigation mentioned the possible multistep reduction of some molybdc species leading to a molybdenum oxide which would be reduced by atomic hydrogen previously adsorbed on the inducing metal [6].

Results

Influence of citric and boric acid on the deposition process

Based in a review of the literature [39-41], boric and citric acid were chosen as addition species, two kinds of voltammetric responses were observed the citric and boric acid concentration range. For LiCl + molybdate solutions in the presence of citric acid 0.001 and 0.01M, boric acid 0.003 and 0.03M, deposition began slightly after -1V (Fig. 1). The negative scan revealed a double reduction peak.

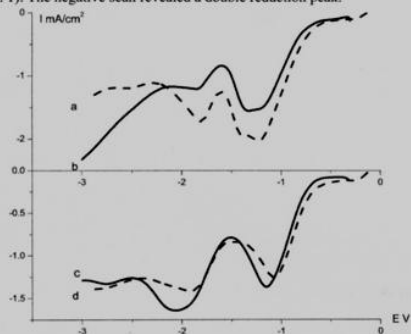


Fig.1 Linear scan voltammogram of 1 M LiCl + 0.01 M $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$ (x M citric + y M boric) acid dimethyl sulfoxide solutions, at 50 mV/s scan rate. (a) x = 0.001, y = 0; (b) x = 0.01 y = 0; (c) x = 0, y = 0.003; (d) x = 0, y = 0.03.

Table 1. IPC-OES analysis of solution obtained after dissolving films in 5 ml of 5% nitric acid. The bath employed for preparing the films contained 0.01M concentration of molybdate and variable acid concentrations.

E/V	0.75	-1	-1.2	-1.35	-1.75	-1.90	-2	-2.15	
C/ mol. L ⁻¹									
Citric	0.001	0	0.0019	0.0119	0.0254	0.0368	0.0429	0.0469	0.0519
	0.01	0	0.0005	0.0089	0.0162	0.0222	0.0314	0.0340	0.0402
Boric	0.003	0	0.0061	0.0113	0.0246	0.0420	0.0490	0.0593	0.0653
	0.03	0	0.0017	0.0093	0.0154	0.0218	0.0315	0.0372	0.0499

Moreover, the charge involved in the first and second reduction processes were increased by increasing the citric acid concentration (Fig. 1, curve a, b), but when the presence of boric acid revealed decreasing on the contrary (Fig. 1, curve c, d). Can be supposed, citric and boric acid was more helpful for decomposition of DMSO, also prevented deposition of molybdenum compounds. Because both of these acids produce more protons. Table 2 corroborated this supposition. The films were deposited to sue recurrent potential pluses different constant potentials and time (20 minutes). Furthermore, ICP-OES analysis revealed that the molybdenum compound decreased greatly when the presence of acids in the bath solution.

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