

Electrodeposition of Molybdenum Alloys from Aqueous Solutions¹

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ABSTRACT

Nickel-molybdenum, cobalt-molybdenum, and iron-molybdenum alloys have been electrodeposited from aqueous solutions containing sodium molybdate, the sulfate of the codeposited metal, sodium citrate, and ammonium hydroxide. Typical baths were made up with 0.3 M/l of the codepositing metal sulfate, 0.3 M/l of sodium citrate, varying amounts of sodium molybdate, and ammonium hydroxide to pH about 10.5. The maximum amount of molybdenum in the electrodeposited alloys depends on the alloying metal. When a typical bath was used, nickel alloys were found to contain up to 20% molybdenum, cobalt alloys contained up to 40% molybdenum, and iron alloys contained about 50% molybdenum. The cathodic current efficiency in the above cases ranged from 75-85% for the nickel-molybdenum bath, 50-60% for cobalt-molybdenum, and 10-20% for iron-molybdenum. The effects of pH, concentration, temperature, and cathodic current density on cathodic current efficiency and alloy composition were studied.

These electrodeposited molybdenum alloys were metallic and either bright or light gray in appearance, with a large number of cracks in the bright deposits. Adherence of the deposit to well-cleaned flat cathodes seemed to be good, but adherence to tubing or rods was poor; usually these deposits could be brushed off in flakes or powder form.

INTRODUCTION

The electrodeposition of molybdenum alloys containing iron, cobalt, or nickel has been reported previously from this laboratory (1) and a patent has been issued (2). Yntema and Kuyrki (3, 4) described the use of solutions containing either aliphatic acids or their salts, or various fluorides to electrodeposit molybdenum alloys. Brenner and Barkhead (5) electrodeposited cobalt-molybdenum alloys from strongly alkaline solutions containing potassium carbonate, cobalt chloride, and sodium molybdate. Nix (6) described the electrodeposition of chromium-molybdenum alloys from a regular chromium plating bath containing molybdenum(VI) oxide. A ternary alloy consisting of molybdenum, tungsten, and cobalt has been electrodeposited from aqueous citrate solutions by McElroy and Holt (7). The electrodeposition of nickel-molybdenum alloys from tartrate and citrate baths has been reported recently (8).

Neim and Holt (1) prepared their plating solutions using sodium molybdate, the sulfate of the codepositing metal, citric acid, and ammonium hydroxide. In this work sodium citrate was used in place of citric acid, being chosen after studying several different complexing agents. An attempt was also made to find another reagent for adjusting the pH

of the plating solutions because of the volatility of ammonia.

EXPERIMENTAL METHODS AND RESULTS

The methods used were similar to those described previously (1) except that in some cases $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ was used instead of anhydrous Na_2MoO_4 . The volume of plating solution used for each electrolysis was 250 ml.

In analyzing cobalt-molybdenum and nickel-molybdenum deposits, the codeposited metal was always determined and molybdenum was usually found by difference. Occasionally molybdenum was determined by the lead molybdate method (9). When both metals were determined the analysis usually totaled 98% or more. Several of the nickel-molybdenum deposits were heated in hydrogen to about 850°C and showed a loss in weight of only 0.5%. Very few of the deposits obtained from the Fe-Mo bath were completely metallic, being covered in the center with a dark black material believed to be a molybdenum oxide; because of this, both components of these alloys were determined, and the total percentage varied from 80-95%, depending on the molybdenum content of the deposit. When several of the iron-molybdenum alloys were heated in hydrogen at about 850°C, the resulting loss in weight was 8 to 9% and the total analysis was then close to 100% (8.2% loss in weight, 40.0% Fe, 51.6% Mo).

Cathodic current efficiencies were calculated using

¹ Manuscript received September 4, 1953. This paper was prepared for delivery before the Wrightville Beach Meeting, September 13 to 16, 1953.

Alloys Of Iron Molydenum

LL Leslie

A red circular graphic with a gradient, appearing as a stylized arrow or a partial circle, located to the right of the author's name.

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