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Standard Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating¹

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INTRODUCTION

Because of the high melting point of tungsten, (3410°C) this metal and its alloys are used in ultra-high-temperature applications. However, tungsten oxidizes readily and the oxides offer little or no protection to the metal because they melt or sublime below 2200°C. Tungsten has a body-centered-cubic structure as does chromium. Its coefficient of thermal expansion is $4.3 \mu\text{m}/\text{m} \cdot ^\circ\text{C}$; the coefficient of chromium is $6.1 \mu\text{m}/\text{m} \cdot ^\circ\text{C}$, therefore, chromium-coated tungsten offers a reasonable match based on crystal structure and coefficient of thermal expansion. The effect of hydrogen must be considered in the design of coating systems for tungsten. Molecular hydrogen when trapped under a coating can result in failure of the part. Although failure can occur if the adhesion of the coating to the tungsten is inadequate, failure within the basis metal as rupture at laminations of stringers has been observed with off-grade tungsten.

1. Scope

1.1 This practice makes recommendations of several reported practices for electroplating on tungsten and its alloys along with comments as a guide in the selection of a processing method for a given application. Because data on methods and results of evaluation with electroplated coatings on tungsten are limited, a recommendation of one method over another cannot be made.

1.2 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Processes

2.1 The several processes reported for electroplating on tungsten can be classified as either (1) providing as-deposited adhesion of the coating or (2) depending on diffusion alloy bonding to provide such adhesion. Appraisal of the relative merits of these processes depends on the application for the coating.

2.2 *Precleaning:*

2.2.1 The need for descaling, solvent cleaning, and alkaline-soak cleaning of tungsten is dictated by the degree of cleanliness of the surface to be coated. Remove gross scale and surface imperfections by mechanical means. Remove grease

and oil by organic solvents. Remove saponifiable and dispersible surface dirt by soak cleaning in a hot alkaline cleaner.

2.2.2 Anodic treatment of tungsten surfaces in a 5 to 25 mass % sodium hydroxide solution at $71 \pm 6^\circ\text{C}$ and 16 to 25 A/dm² can be used to remove undesired surface metal. Anodic etching in a 10 mass % hydrofluoric acid electrolyte at $24 \pm 3^\circ\text{C}$ and 5.4 A/dm² can be used to remove light scale and undesired surface metal before activation and electroplating of tungsten.

2.3 *Activation and Electroplating*—Two alternative methods are presented below. The selection of one process over the other should be based on preliminary experimentation. Both procedures produce as-deposited adhesion of electroplated metals on tungsten.

2.3.1 *Methods 1²*—Treat the precleaned tungsten in a hydrofluoric acid electrolyte (30 ± 10 mass % HF) at $24 \pm 3^\circ\text{C}$, using 2 to 5 V, 60-Hz ac, and 2 to 5 A/dm² for 1 to 2 min. Either divide the work between two work rods or make one electrode of platinum. After rinsing, the work is electroplated in acid electrolytes.

2.3.2 *Method 2*—Electroplate the precleaned tungsten with a chromium strike prior to subsequent electroplating with other metals^{3,4} using the following processing steps:

2.3.2.1 *Anodic Etch*—Etch anodically at 10 to 30 A/dm² in the following solution for 2 to 5 min.

KOH	30 mass %
Temperature	48 to 60°C

¹ This practice is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.02 on Substrate Preparation.

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² Cannizaro, J. J., U.S. Patent No. 2,433,651, June, 1948.

³ Korbela, A., U.S. Patent No. 2,697,130, Dec. 14, 1954.

⁴ Marzano, C., "Plating on Tungsten," *Plating*, Vol 51, 1964, pp. 207–211.

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2.3.2.2 *Rinse.*

2.3.2.3 *Acid Neutralize*—Immerse for 1 min in 10 mass % sulfuric acid at $27 \pm 6^\circ\text{C}$.

2.3.2.4 *Rinse.*

2.3.2.5 *Chromium Strike*—Strike at 15 to 25 A/dm² in the following chromium solution for 1 to 3 min. Enter the solution with the parts cathodic.

CrO ₃	250 g/L
H ₂ SO ₄	2.5 g/L
Temperature	60 to 72°C

2.3.2.6 *Rinse.*

2.3.2.7 *Acid Activation*—Immerse in 20 mass % hydrochloric acid at 21 to 33°C for 2 to 5 s.

2.3.2.8 *Nickel Strike*—Strike at 5 to 10 A/dm² in the following nickel solution for 2 to 5 min.

NiSO ₄ · 6H ₂ O	240 g/L
H ₂ SO ₄	40 g/L
Temperature	21 to 33°C

2.3.2.9 *Rinse.*

2.3.2.10 *Electroplate*—Electroplate with desired metal.

2.4 *Alloying and Heating Effects:*

2.4.1 A number of other processes, involving heat treating ($927 \pm 56^\circ\text{C}$) to diffusion-alloy bond an electrodeposited coating to the basis tungsten have been proposed but offer no apparent advantage.

2.4.2 Chromium-strike coated tungsten^{3,4} subsequently electroplated with nickel and heated for ½ h at 980°C shows no deterioration of the electroplated coatings. Chromium-tungsten diffusion does not form the weak brittle intermetallic alloy zones such as are evident when nickel, iron, or cobalt coatings on tungsten are similarly heat treated.

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