



Designation: B629 – 77 (Reapproved 2019)

## Standard Practice for Preparation of Molybdenum and Molybdenum Alloys for Electroplating<sup>1</sup>

This standard is issued under the fixed designation B629; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers a procedure for preparing molybdenum and its alloys for electroplating. The procedure utilizes techniques, equipment, and chemicals that are common to electroplating. Specialized procedures such as hydrogen heat treatment and fused salt pickling are not included. These and other procedures can be found in the references listed at the end of this practice.

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

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Significance and Use

2.1 Molybdenum is used in engineering applications where strength and rigidity of component parts are needed, especially at high temperatures. Its good strength at high temperatures, low vapor pressure, and good impact and creep resistance make it attractive for load-bearing items such as turbine blades, mechanical seals, rocket motor parts, heating elements, and electronic devices. Molybdenum, however, is easily oxidized, and the oxides are not protective. The volatility of the oxides above 800 °C can lead, in the case of unprotected parts, to catastrophic oxidation and to losses in dimensions. Electroplated coatings are applied to molybdenum to prevent oxidation, to prevent seizing and galling, and to assist joining by soldering, brazing, and diffusion bonding.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.02 on Pre Treatment.

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### 3. Procedure

#### 3.1 *Precleaning:*

3.1.1 The principles of electroplating on molybdenum are similar to those of other metals. The surfaces must be free of dirt, grease, and other foreign matter. Solvent, emulsion, and soak cleaners are suitable for precleaning to remove gross surface contaminants. If a molybdenum specimen is subjected to severe forming operations and retains a carbonaceous surface layer, descale the part by immersion in a 110 g/L solution of sodium hydroxide at or near boiling, or by mechanical finishing. Glass bead blasting can be used to remove uneven coatings of scale without disturbing the finish of the metal.

3.1.2 Cathodic alkaline cleaning is preferred for the final precleaning so as to avoid anodic oxidation. Proprietary formulations used for cathodic alkaline cleaning of other metals are suitable. Cathodic treatment at about 6 V for 30 to 60 s is usually adequate, using a cleaner concentration of 60 to 75g/L and a solution temperature of 60 to 75 °C.

3.2 *Rinse*—Here, and throughout the procedure, the water rinses should be warm (approximately 50 °C).

3.3 *Electropolishing*—Either of the following electropolishes may be used. The choice of electropolish and time depends on the amount of material that can be removed and the degree of surface improvement desired. The cathodes may be lead or carbon. The sulfuric acid bath produces a blue film that is partly removed in the rinse and completely removed in the alkaline dip.

#### 3.3.1 *Sulfuric Acid:*

H <sub>2</sub> SO <sub>4</sub> (sp gr 1.83)	80 mass % (755 mL of acid/L)
Temperature	20 to 30 °C
Current density	1100 to 2200 A/m <sup>2</sup> anodic
Time	30 s

#### 3.3.2 *Sulfuric - Phosphoric Acid:*

H <sub>2</sub> SO <sub>4</sub> (sp gr 1.83)	50 % vol (500 mL/L)
H <sub>3</sub> PO <sub>4</sub> (85 %)	50 % vol (500 mL/L)
Temperature	50 to 55 °C
Current density	2500 A/m <sup>2</sup> anodic
Time	180 s

#### 3.4 *Rinse.*

3.5 *Alkaline Dip*—Briefly immerse the specimen in a 45 g/L solution of sodium hydroxide at room temperature.

3.6 Rinse.

3.7 *Acid Dip*—Briefly immerse the specimen in a 10-mass % solution of sulfuric acid (58 mL of 1.83 sp gr acid/L) at room temperature.

3.8 Rinse.

3.9 *Chromium Striking*—Electroplate the specimen in a conventional chromium electroplating solution for 1 to 5 min at 1500 to 2500 A/m<sup>2</sup> at a temperature of 50 °C minimum. In some cases, it may be necessary to first make the part anodic in the chromium solution for 1 min at 2200 A/m<sup>2</sup> in order to attain good adhesion.

3.10 Rinse.

3.11 *Nickel Striking*—Electroplate the specimen in the following solution at 800 A/m<sup>2</sup> for 120 s.

NiCl <sub>2</sub> · 6H <sub>2</sub> O	240 g/L
HCl (sp gr 1.18)	10 vol % (100 mL/L)
Temperature	20 to 30 °C
Anode	nickel or carbon

3.12 Rinse.

3.13 Electroplate the specimen with the desired metal.

## REFERENCES

- (1) Beach, J. G., et al. U. S. Patent 2,928,169 (March 15, 1960).
- (2) Brenner, A., *Plating*, Vol 43, 1956, p. 1143.
- (3) Domnikov, L., *Metal Finishing*, Vol 61, April 1963, p. 64.
- (4) Finne, R. M., and Bracht, W. R., *Journal of Electrochemical Society*, Vol 113, 1966, p. 551.
- (5) Freeman, R. R., and Briggs, J. Z., “Electroplating on Molybdenum Metal,” Climax Molybdenum Co., Ann Arbor, MI, September 1958.
- (6) Korbelak, A., *Plating*, Vol 40, 1953, p. 1126, U. S. Patent 2,697,316.
- (7) Safranek, W. H., and Schaer, G. R., *Proceedings, American Electroplaters’ Society*, Vol 43, 1956, p. 105.
- (8) Schaer, G. R., U. S. Patent 2,859,158 (November 4, 1958).
- (9) Schaer, G. R., et al, U. S. Patent 2,886,499 (May 12, 1959).
- (10) Seegmiller, et al, *Proceedings, American Electroplaters’ Society*, Vol 49, 1962, p. 67.
- (11) Valler, L. E., et al. *USAED Report No. BMI 813*, (April 1, 1953 ) (Obtainable from National Technical Information Service, Springfield, VA).

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