Designation: B480 – 88 (Reapproved 2017)

Standard Guide for
Preparation of Magnesium and Magnesium Alloys for Electroplating[1]

This standard is issued under the fixed designation B480; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide describes two processes used for plating on magnesium and magnesium alloys: direct electroless nickel plating and zinc immersion. Some users report that the direct electroless nickel procedure does not produce quite as high a level of adhesion as zinc immersion.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 5.1.1 and 5.2.9.2.

1.4 *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. Referenced Documents

2.1 *ASTM Standards:*[2]

B322 Guide for Cleaning Metals Prior to Electroplating

3. Significance and Use

3.1 Metals are electroplated on magnesium for various purposes: solderability, RF grounding, hermetic sealing, wear resistance, corrosion resistance, appearance, and electrical conductivity, for example. Because magnesium is covered with a naturally occurring oxide film, usual procedures for the preparation of metals for autocatalytic or electrolytic plating cannot be used.

4. Reagents

4.1 *Purity of Reagents*—All acids and chemicals used in this guide are of technical grade. Acid and base solutions are based on the following assay materials:

Ammonium hydroxide (NH_4OH)	30 mass %, density 0.895 g/L
Nitric acid (HNO_3)	67 mass %, density 1.16 g/L
Sulfuric acid (H_2SO_4)	93 mass %, density 1.40 g/L
Hydrofluoric acid (HF)	70 mass %, density 1.258 g/L
Phosphoric acid (H_3PO_4)	85 mass %, density 1.689 g/L

4.2 *Purity of Water*—All water used for solutions, whether new or recycled, should be monitored for cations, anions, and organic matter that are known to interfere with the plating process.

5. Processes

5.1 *Procedures:*

5.1.1 Wheel polish and buff parts for smooth, highly polished surfaces. Tumble and burnish small parts. Acid pickle the parts after use of wire brushing or steel wool. (**Warning—**Because of the high flammability of powdered magnesium, special precautions against fire are important. Polishing and buffing lathes should be kept scrupulously clean. Dust from grinding in and around lathes should be swept up and placed in closed containers for proper disposal. Exhaust systems should be cleaned frequently and the residues handled similarly. If abrasives are used in tumble finishing, similar precautionary techniques should be used for the solids from the abrasive slurry.)

5.1.2 *Chemical*—Remove oil and grease in an alkaline soak cleaner. Remove other soils and coatings in suitable acid pickling solutions.

NOTE 1—General information on the cleaning of metals is given in Practice B322.

5.2 *General Electroplating Procedure:*

5.2.1 Remove oil, grease, and other soils left from preplating procedures by soak cleaning in an alkaline cleaner suitable for magnesium.

5.2.2 Rinse in cold water.

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[1] This guide 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.

Current edition approved May 1, 2017. Published May 2017. Originally approved in 1968. Last previous edition approved in 2010 as B480 – 88(2010). DOI: 10.1520/B0480-88R17.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

NOTE 2—As generally used in rinsing terminology, cold water refers to water from an unheated water supply as opposed to heated water used for drying or other purposes. In some areas, particularly in water, ambient water temperatures may be too low for effective rinsing. In those instances, the rinse water may need to be heated. A minimum temperature of 16°C is recommended for effective rinsing.

5.2.3 Electroclean parts in an alkaline electrocleaner suitable for magnesium. Make the magnesium cathodic at 7.5 to 13 A/dm ^{2} and 85°C.

5.2.4 Rinse in cold water.

5.2.5 Pickle in one of the following solutions:

5.2.5.1 *Ferric Nitrate Pickle:*[3]

Chromic acid (CrO_3)	180 g/L
Ferric nitrate ($Fe(NO_3){\cdot}9H_2O$)	40 g/L
Potassium fluoride (KF)	3.5 g/L
Temperature	16 to 38°C
Time	15 s to 3 min

NOTE 3—This pickle removes metal from a surface at the rate of 3 µm/min at 38°C. Where no dimensional change can be tolerated, use of the chromic acid pickle in 5.2.5.2 is recommended.

5.2.5.2 *Chromic Acid Pickle:*[3]

Chromic acid (CrO_3)	180 g/L
Temperature	16 to 93°C
Time	2 to 10 min

NOTE 4—The use of chromic acid pickles may leave chromate films on the surface that will reduce the adhesion of the subsequently deposited coating.

5.2.6 Rinse in cold water.

5.2.7 Activate in the following solution:[3]

Phosphoric acid, (H_3PO_4)	20 % by vol
Ammonium bifluoride (NH_4HF_2)	105 g/L
Temperature	16 to 38°C
Time	15 s to 2 min

5.2.8 Rinse in cold water.

5.2.9 Zinc coat in the following solution:

Zinc sulfate ($ZnSO_4{\cdot}H_2O$)	30 g/L
Tetrasodium pyrophosphate ($Na_4P_2O_7$)	120 g/L
Sodium fluoride (NaF) or	5 g/L or
Lithium fluoride (LiF)	2 g/L
Sodium carbonate (Na_2CO_3)	5 g/L

5.2.9.1 Because of the low solubility of the tetrasodium pyrophosphate, it is generally necessary to mix this solution in an elevated temperature from 70 to 85°C. It is also advantageous to alternately add portions of the zinc sulfate and pyrophosphate. When these ingredients are completely dissolved, add and dissolve the balance of the ingredients in the order given.

NOTE 5—Either sodium fluoride or lithium fluoride may be used as a constituent of this bath. Sodium fluoride is widely used, but requires careful control. Potassium fluoride is too hygroscopic and should not be used because the variation in water content makes it impossible to be sure of how much is being added. Lithium fluoride has been found highly desirable because it is soluble only to the proper concentration level and is self-regulating. Thus, the use of lithium fluoride eliminates the need for fluoride analysis. During bath makeup, 3 g/L lithium fluoride is added. This small quantity saturates the solution, and an excess suspended in the bath in a canvas or nylon anode bag automatically replaces any fluoride consumed during the operation.

5.2.9.2 Immerse the parts for 3 to 10 min in the solution operated at 79 to 85°C and agitate mildly. Do not use glass or fiberglass equipment of any kind. Careful control is essential for best results. Maintain the pH between 10.2 and 10.4 electrometric (glass electrode) measured at 25°C. (**Warning—**The glass electrode must be used with caution. Do not allow the electrode to remain in contact with the zincate solution for prolonged periods. Inspect and test the electrode regularly to ensure that no change has occurred from contact with fluoride ion. Colorimetric (paper) methods may be used. However, the colorimetric readings may vary ±0.5 pH units.)

5.2.10 Rinse thoroughly in cold water.

NOTE 6—For alloys M1660, M13120, M13312, or M13310, a double zinc immersion is required. After step 5.2.10, steps 5.2.7 – 5.2.10 are repeated followed by step 5.2.11. It is advisable to use separate solutions for steps 5.2.7 and 5.2.9 when the double zinc immersion technique is practiced.

5.2.11 Copper strike in either of the following:

Bath 1:

Copper cyanide (CuCN)	38 to 42 g/L
Potassium cyanide (KCN)	64.5 to 71.5 g/L
Potassium fluoride (KF)	28.5 to 31.5 g/L
Free potassium cyanide	7 to 8 g/L
pH	9.6 to 10.4
Temperature	54 to 60°C

Bath 2:

Copper cyanide (CuCN)	38 to 42 g/L
Sodium cyanide (NaCN)	50 to 55 g/L
Rochelle salt ($KNaC_4H_4O_6{\cdot}4H_2O$)	40 to 48 g/L
Free sodium cyanide	7 to 8 g/L
pH	9.6 to 10.4
Temperature	54 to 60°C

5.2.11.1 Plate the parts about 6 min. Cathode rod agitation is suggested. With either bath make electrical contact quickly with initial current 5 to 10 A/dm^2, then lower current to 1 to 2.5 A/dm^2.

5.2.12 Rinse thoroughly in cold water.

5.2.13 Dip in diluted acid (1 % by vol sulfuric acid + 99 % by vol water).

5.2.14 Rinse in cold water.

5.2.15 Apply subsequent electrodeposits or autocatalytic nickel in accordance with standard commercial electroplating practice.[3] Use plastisol-coated racks that are fitted with stainless steel or phosphor-bronze rack tips.

5.3 *Autocatalytic Nickel Plating Magnesium:*

5.3.1 *Surface Conditioning*—See 5.1.1 and 5.1.2, and 5.2.1 – 5.2.4.

5.3.2 *Pickling*—Pickle in (*1*) chromic acid[3] as directed in 5.2.5.2 or in (*2*) phosphoric acid[3] (90 % by vol phosphoric acid + 10 % by vol water) using a lead, glass, ceramic, or rubber-lined tank of polyethylene, polypropylene, or other suitably corrosion resistant material.

5.3.3 Rinse in cold water.

5.3.4 Chemical etch in one of the following:

Etch 1—For Alloys Containing Aluminum:[3]

Chromic acid (CrO_3)	120 g/L
Nitric acid (HNO_3)	(11 % by vol nitric acid + 89 % by vol water) to make 1L

Etch 2—For Other Magnesium Alloys:[3]

Chromic acid (CrO_3)	60 g/L
Nitric acid (HNO_3)	(9 % by vol nitric acid + 91 % by vol water) to make 1 L

Stainless steel tank or tank lined with glass, ceramic, polyvinyl chloride, polyethylene, or other suitably resistant plastic material.

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[3] *Magnesium Finishing*, The Dow Metal Products Co., Midland, MI.

Immerse parts 20 to 60 s in solution at room temperature. Rinse thoroughly and proceed immediately to hydrofluoric acid dip.

NOTE 7—Use the chromic acid pickle where dimensional change cannot be tolerated. Do not use on alloys containing thorium. For thorium-containing alloys and on other alloys where dimensional change is not critical, use phosphoric acid pickle. Immerse parts 30 s to 1 min in the phosphoric acid pickle at 21 to 32°C. Metal loss is about 13 µm/surface. If thorium alloys have critical dimensions, use chromic acid pickle and follow with an immersion in a strongly alkaline cleaner to remove possible chromate films.

5.3.5 Dip[3] in hydrofluoric acid (5.5 vol % hydrofluoric acid + 94.5 vol % water) using a tank lined with polyvinyl chloride, polyethylene, or polypropylene. Immerse parts 10 min in solution at room temperature. For treating M11610 or M11800 use a 20 vol % hydrofluoric acid + 80 vol % water solution. Transfer to nickel bath immediately after rinsing. Use of ammonium bifluoride (NH_4HF_2), at 60 to 90 g/L (8 to 12 oz/gal) can often be substituted for hydrofluoric acid. Use of ammonium bifluoride eliminates the hazard of handling concentrated hydrofluoric acid.

NOTE 8—The zinc immersion coating plus copper strike can be used instead of chemical etching (see Section 1). Follow steps 5.2.5 – 5.2.11.

5.3.6 Nickel plate[3] in an autocatalytic bath specific for magnesium. The following[4] is an example of a magnesium specific autocatalytic bath:

Basic nickel carbonate ($2NiCO_3 \cdot 3Ni(OH)_2 \cdot 4H_2O$)	10 g/L
Hydrofluoric acid (HF)	(0.6 vol % hydrofluoric acid + 79.4 vol % water)
Citric acid ($C_6H_8O_7$)	5.2 g/L
Ammonium bifluoride (NH_4HF_2)	10 g/L
Sodium hypophosphite ($NaH_2PO_2 \cdot H_2O$)	20 g/L
Ammonium hydroxide (NH_4OH)	(3.9 % by vol ammonium hydroxide + 96.1 % by vol water)
Adjust pH to 5.5 to 6.3	

Use a tank lined with polyvinyl chloride plastisol, baked-phenolic based enamels, polyethylene, polypropylene, or other suitably resistant material. Use mild mechanical agitation. Immerse parts in nickel solution operated at 77 to 82°C with a pH range of 5.5 to 6.3, measured at 25°C. The bath should be filtered either periodically or continuously. The use of a pump which is sealess or designed for operation in fluoride and with electroless nickel solution is recommended. To ensure adequate filtration, the solution should be filtered in excess of 20 tank volumes per hour through a 5-micron filter. The use of a plastic, magnetically coupled pump will eliminate problems with shaft seals.

NOTE 9—If heavy electroless nickel deposits are to be applied, the parts can be transferred to the appropriate bath after a thickness of 5 µm has been deposited from this bath.

5.3.7 Rinse in cold water. For maximum corrosion resistance when the above plate is the final deposit, immerse for 10 min in (1) chromic acid (CrO_3)[5] (2.5 g/L) or (2) sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$) (120 g/L). Operate both solutions at 88 to 100°C. Proprietary compounds are available which operate at room temperature.

5.3.8 Thoroughly rinse in cold water and dry. To improve adhesion, bake at 200°C for 1 h. The deposit should remain adherent and free of blisters.

6. Keywords

6.1 activation; cleaning; deoxidizing; magnesium; preparation; striking

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[4] This bath was patented (U.S. Patent 3,152,009, Oct. 6, 1964, DeLong, H.K.). It was assigned to the Dow Chemical Co., Midland, MI. Other autocatalytic plating solutions that may be used are described in U.S. Patent Nos. 2,983,634, May 9, 1961, Budininkas, P; 3,121,644, Feb. 18, 1964, Gutzheit, G. and Lee, W.G.; and 3,211,578, Oct. 12, 1965, Gutzheit, G. There are also many specialized proprietary baths available which may be applied directly or after a minimum of 5 µm coating has been applied from any of the above baths. All the patents listed have expired.

[5] Lee, W.G., U.S. Patent No. 3,088,846, May 7, 1963 (expired).