



AEROSPACE MATERIAL SPECIFICATION

AMS-M-3171™**REV. A**

Issued 1998-04
Reaffirmed 2018-02
Stabilized 2022-09

Superseding AMS-M-3171

Magnesium Alloy, Processes for Pretreatment
and Prevention of Corrosion on

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1. SCOPE:

1.1 Scope:

This specification covers general requirements for the apparatus, material, and procedures to be used in the processing of magnesium base alloys for the purpose of increasing their corrosion resistance and by producing surfaces suitable for organic paint finish systems.

1.2 Classification:

1.2.1 Types - The magnesium corrosion preventive treatments shall be of the following types:

- Type I - Chrome Pickle Treatment
- Type III - Dichromate Treatment
- Type IV - Galvanic Anodizing Treatment
- Type VI - Chrome Acid Brush-on Treatment
- Type VII - Fluoride Anodizing Process plus Corrosion Preventive Treatment
- Type VIII - Chromate Treatment

2. APPLICABLE DOCUMENTS:

The following publications, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

2.1 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue,
Philadelphia, PA 19111-5094.

MIL-C-46079 Coating, Epoxy, Baking Type for Magnesium Casting
MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

3. REQUIREMENTS:

3.1 General requirements:

- 3.1.1 Materials: All materials used in the cleaning or treating of magnesium base alloys shall be of good quality and suitable for the purpose intended. All chemicals used in cleaning and treating shall be free from metallic impurities such as salts of heavy metals (lead, nickel, zinc, arsenic, antimony, etc.).
- 3.1.2 Workmanship: The treatments covered by this specification shall be controlled and operated to give a uniform surface conforming to the requirements specified herein. The surface shall be free from breaks, discontinuities, scratches, and other damage. The size and number of contact marks shall be held at a minimum.

3.2 Cleaning and pickling operations (see 3.12):

- 3.2.1 Solvent cleaning: Grease or oil shall be removed by means of vapor degreasing, ultrasonic cleaning, organic solvents, or by emulsion cleaning which employs a mineral oil distillate and an emulsifying agent. For this cleaning operation, chlorinated solvents, petroleum spirits, naphtha, lacquer thinners, and similar grease solvents which do not attack magnesium may be used. Methyl or wood alcohol (CH_3OH), is specifically prohibited as a solvent cleaner for magnesium alloys.
- 3.2.2 Mechanical cleaning: Mechanical cleaning shall consist of sand, shot, grit, or vapor blasting, sanding, hard bristle brushing, grinding and rough polishing. When sand, shot, or grit blasting methods are employed, there is danger of surface contamination which will greatly increase the initial corrosion rate of magnesium. In such case, subsequent pickling operations, as specified in 3.2.4.2, or fluoride anodizing as specified in 3.9.1 shall be employed.
 - 3.2.2.1 New sand castings: New sand castings shall be sand or grit blasted and then pickled in conformance with 3.2.4.2 or fluoride anodized as specified in 3.9.1.
 - 3.2.2.2 Discolored or corroded castings: Discoloration or corrosion products shall be removed from rough unmachined castings. This operation may be performed by blasting and subsequent pickling as specified in 3.2.4.2. Machine parts or sections, which may be held to close dimensional tolerances, may be cleaned by use of a chromic acid pickle solution conforming to 3.4.1.
 - 3.2.2.3 Welding flux: When using flux in the welding operations, the welded sections shall be thoroughly cleaned immediately after the operation to remove all traces of flux. Where possible, hot water and a hard bristle brush shall be used to insure thorough flux removal. Inaccessible welded sections shall be washed with a high velocity stream of water (preferably hot). They shall then be immersed for 1 hour in a 2 to 5 percent sodium dichromate solution maintained at 180 to 212 °F. The parts shall be thoroughly rinsed in cold running water.
- 3.2.3 Alkaline cleaning: Cleaning prior to application of treatments other than Type I when used for protection during shipment or storage, shall be done by cleaning the parts in an alkaline cleaner recommended for steel or in cleaning solution as specified in 3.3.2. Any alkaline cleaner used shall have a pH that is maintained above 8. Alkaline cleaning, prior to application of Type I when used for protection during shipment or storage only, may be omitted provided the parts are free from grease, oil, or other deleterious deposits at the time of application. Alkaline cleaners containing more than 2 percent caustic, such as sodium hydroxide will etch ZK60A, ZK60B, and some magnesium-lithium alloys with resultant decrease in dimensions. If such a dimensional change is not desirable, the alkaline cleaners used on these alloys should not contain more than 2 percent caustic.

- 3.2.3.1 Graphite lubricant removal: Graphite base lubricants shall be removed from hot formed magnesium sheet parts by soaking the parts for 10 to 20 minutes in an aqueous sodium hydroxide (NaOH) bath containing 13 oz/gallon of NaOH and maintained at 190 to 212 °F. This bath shall be maintained at a pH above 13. One-tenth ounce per gallon of soap or wetting agent may be added to this solution if removal of heavy films of mineral oil is required. The parts shall be thoroughly rinsed in cold water and immersed in a chromic-nitrate pickle as specified in 3.4.2 for approximately 3 minutes. If complete cleaning is not obtained the first time, this operation shall be repeated until the parts are clean. Because of the difficulty of removing graphite from chrome pickled sheet, such sheet shall not be used for forming unless the chrome pickle coating has been completely removed as specified in 3.2.3.2.
- 3.2.3.2 Previously applied chemical finishes: Previously applied chemical finishes shall be removed. Magnesium base alloys sometimes are furnished with Type I treatment which provides protection during shipment, storage, and machining. The coating resulting from Type I treatment remaining on the unmachined areas will impair the film produced by Type III and Type IV treatments and consequently, must be removed. This operation may be performed by use of alkaline cleaners such as the types specified in 3.2.3. If the finish is difficult to remove, the part should be immersed in the chromic acid pickle specified in 3.4.1. Alternate immersion in the alkaline cleaner and chromic acid pickle may be necessary to remove very old finishes.
- 3.2.3.2.1 The Type VI treatment may be applied over previously applied thin film by Type I or Type VI treatments without removing these finishes (see 3.8.1).
- 3.2.4 Acid pickling:
- 3.2.4.1 General pickling: General pickling to remove oxide layers, old chemical finishes, burned-on drawing and forming lubricants, and other water insoluble or non-emulsifiable substances shall be accomplished, preferably by using a chromic acid type pickle conforming to 3.4.1 or 3.4.2.
- 3.2.4.2 Pickling of magnesium sand and permanent mold castings: Magnesium sand and permanent mold castings which have been subjected to blasting operations shall be pickled in a sulfuric acid pickle conforming to 3.4.3 or in a sulfuric-nitric acid pickle conforming to 3.4.4. Pickling operations shall be continued until 0.002 inch of surface has been removed, whenever dimensional tolerances permit.
- 3.2.4.3 Pickling of magnesium sheet and plate: Magnesium sheet and plate up through 0.500 inch thick shall have the mill scale removed to insure a clean surface to which subsequent treatment will be applied. Where dimensional tolerances permit, 0.0005 inch to 0.001 of metal surface shall be removed. An acetic-nitrate pickle conforming to 3.4.7 or equivalent acid pickle shall be used for this operation.
- 3.2.4.4 Pickling of magnesium die castings: Die castings that are to be pickled for maximum corrosion resistance and a more uniform surface for treating shall be pickled in a chromic-nitric HF pickle conforming to 3.4.5 or a phosphoric acid pickle conforming to 3.4.6.

3.2.5 Retreatment of wrought and cast parts:

3.2.5.1 Grease and oil: Grease and oil shall be removed in conformance with 3.2.1.

3.2.5.2 Paint: Old paint coatings shall be removed, preferably by the use of an alkaline (caustic) liquid paint remover. Solvent type paint removers may be used provided the operation is followed by a wash with a wax-free solvent to remove any wax which may have been left on the surface. Light blasting may be used to remove paint from parts of substantial thickness, but only sanding shall be permitted on thin sheets. If sand-blasted, the part shall be pickled in conformance with 3.2.4.2.

3.2.5.2 Chemical finishes: Chemical finishes shall be removed as specified in 3.2.3.2 before retreatment.

3.2.5.3 Alkaline cleaning: Parts to be retreated shall be cleaned in an alkaline cleaning solution as specified in 3.2.3.

3.2.5.5 Corrosion or oxide films: Corrosion products or oxide films shall be removed, preferably by use of the chromic acid pickle or chromic-nitrate pickle as specified in 3.4.1 and 3.4.2, respectively.

3.3 Alkaline cleaning solutions:

3.3.1 Alkaline cleaning as required in 3.2.3 may be performed by using proprietary type cleaners, in which case the operation shall be as specified by the vendor. In no case will any such cleaner be used that has a pH lower than 8. An alkaline cleaner of the formulation given in Table I may be used quite satisfactorily. The parts are simply immersed in the solution, or, if direct current is available, the parts are made cathodes in the bath and a current density of 10 to 40 amp/sq. ft. at 6 volts, is applied. When cathodic cleaning is employed, the time required for adequate cleaning is reduced. Parts are given a thorough rinse in cold running water after alkaline cleaning. Parts shall be cleaned until no water breaks occurs in the rinse.

3.3.2 Anodic cleaning employing the work as the anode in the alkaline cleaning solution is generally not recommended because of the formation of undesirable oxide films as well as pitting of magnesium alloy surfaces from prolonged contact.

TABLE I
ALKALINE CLEANER SOLUTION AND OPERATION

Composition	Ounces per gallon (water added to make one gallon)	Operating temperature °F	Treatment time- minutes	Tank construc- tion
Sodium hydroxide (NaOH)	8	190 to 212°	3 to 10	Steel
Trisodium phosphate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$)	1-1/3			
Soluble soap or wetting agent	0.1			

3.4 Acid pickling solutions:

- 3.4.1 Chromic acid pickle: Chromic acid pickling may be used where parts with close tolerance limits are to be finished since this pickle causes no dimensional change. It can be used in removing old chemical finishes by alternate immersion in an alkaline cleaner specified in 3.3.2 and the chromic acid pickle. This pickle is satisfactory for removal of surface oxidation, corrosion products, and for general cleaning of parts. It is not satisfactory for the removal of sand or the effects of blasting and shall not be used for parts containing copper based inserts unless masked off. Excessive amounts of anions, such as chlorides, sulfates, and fluorides must not be allowed to build up in the solution as these tend to coat or etch the metal rather than clean the surface. Silver nitrate (AgNO_3) may be used to precipitate out any chloride buildup, thus extending the life of the bath, but it is better to discard the contaminated solution and replace with a new solution. The solution composition is indicated in Table II.
- 3.4.2 Chromic-nitrate pickle: The chromic-nitrate pickle is not generally used where surface oxidation or corrosion products are to be removed but can be substituted for the chromic acid pickle specified in 3.4.1. It is used, as specified in 3.2.3.1, primarily for removing burned-on graphite lubricants. It is not satisfactory for the removal of sand or the effects of blasting and shall not be used for parts containing copper base inserts unless masked off. Lack of chemical action and a pH of 1.7 or higher indicates depletion of the bath. The bath may be controlled by additions of chromic acid to bring the solution back to the original pH range of 0.5-0.7. Large tanks may be revived economically in this manner about 4 times after which tanks should be drained and refilled with new solution. Excessive revivification results in too great a consumption of chromic acid, excessive etching and staining of the magnesium. Actual time and temperature used should be regulated to produce satisfactory results. The solution composition is indicated in Table II.

TABLE II
Acid Pickling Solutions

Treatment	Composition 1/		Immersion Time	Operating Temperature °F	Tank Construction	Metal Removed, Mils 2/	Ref Para
	Material	oz.					
Chromic acid	Chromic acid (CrO ₃)	24	1 to 15 min.	190° - 202° 3/	Lead-lined steel, stainless steel, or 1100 aluminum.	None	3.4.1
Chromic-nitrate	Chromic acid (CrO ₃) Sodium nitrate (NaNO ₃)	24 4	2 to 20 min. 4/	60° - 90°	Ceramic, stainless steel, lead-lined or synthetic rubber lined tanks or tanks lined with vinyl based materials.	0.5	3.4.2
Sulfuric acid	Sulfuric acid (sp gr 1.84)	4	10 to 15 sec.	70° - 90°	Ceramic, rubber-lined, lead, or other suitable tanks	2.0	3.4.3
Nitric-sulfuric acid	Nitric acid (sp gr 1.42) Sulfuric acid (sp gr 1.84)	10-1/4 2-1/2	10 to 15 sec.	70° - 90°	Ceramic, rubber-lined, or other suitable tanks.	2.0	3.4.4
Chromic-nitric-HF	Chromic acid (CrO ₃) Nitric acid (sp gr 1.42) Hydrofluoric acid (60% HF)	37-1/2 3-1/4 1	1/2 to 2 min.	70° - 90°	Synthetic rubber-lined tank or tanks lined with vinyl base materials.	0.5 to 1	3.4.5
Phosphoric acid	Phosphoric acid (85% H ₃ PO ₄)	115.2	1/2 to 1 min.	70° - 80°	Ceramic or tank lined with lead, glass, or rubber.	0.5	3.4.6
Acetic-nitrate	Glacial acetic acid (CH ₃ COOH) Sodium nitrate (NaNO ₃)	25-1/2 6-2/3	1/2 to 1 min.	70° - 80°	3003 Aluminum, ceramic, or rubber lined tank.	0.5 to 1	3.4.7
Glycolic-nitrate	Glycolic acid (70% CH ₂ OHCOOH) Magnesium nitrate Nitric Acid	32 27 3.8	3 to 4	70° - 80°	Stainless steel, ceramic or other suitable tanks.	0.5 to 1	3.4.8
Spot weld chromic-sulfuric	Chromic acid (CrO ₃) Sulfuric acid (sp gp 1.84)	24 0.065	3 min.	70° - 90°	Stainless steel, 1100 aluminum, ceramic or synthetic rubber.	0.3	3.4.9

NOTE:

1/ Avoidupois or fluid ounces per gallon. Water is added to make one gallon.

2/ Average removed per surface during immersion time in pickle solution.

3/ Bath can be operated at room temperature for a longer time if desired.

4/ Bath maintained at pH of 0.0 to 1.7.

- 3.4.3 Sulfuric acid pickle: Sulfuric acid pickling is used on magnesium sand castings to remove the effects of blasting operations. The pickle should be applied before any machining operation, since the amount of metal removed is likely to exceed permissible tolerances. The solution composition is indicated in Table II.
- 3.4.4 Nitric-sulfuric acid pickle: As an alternate for the sulfuric acid pickle (see 3.4.3) the nitric-sulfuric pickle may be used. The solution composition is indicated in Table II.
- 3.4.5 Chromic-nitric-HF pickle: A chromic-nitric-HF pickle may be used for pickling of castings, particularly die castings. This pickle bath removes about 0.0005 inch per minute per surface. The solution composition is indicated in Table II.
- 3.4.6 Phosphoric acid pickle: The phosphoric acid pickle may be used for pickling all types of castings, particularly die castings. It is especially effective in removing aluminum segregation from the surfaces of AZ91A and AZ91B alloys. It has been used for some wrought alloys, such as HK31A, prior to plating. This pickle bath removes about 0.0005 inch per minute per surface. The solution composition is indicated in Table II.
- 3.4.7 Acetic-nitrate pickle: The acetic-nitrate pickle is suitable for removal of mill scale and other surface contamination from magnesium sheet to ensure maximum protective finishing. This pickle can be used on other wrought forms and on solution heat treated magnesium castings. Castings in the as-cast condition (-F) or in the solution heat-treated and aged condition (-T6) should not be pickled by dipping in an acetic-nitrate pickle bath as a loose gray smut is formed. Magnesium castings in these conditions should be treated with a chromic-nitric-HF pickle (see 3.4.5). For most effective results the pickle should be allowed to remove 0.0005 inch to 0.001 inch metal per surface and therefore may not be suitable for parts requiring close tolerances. The solution composition is indicated in Table II.
- 3.4.8 Glycolic-nitrate pickle: In cases where spray pickling is used or fumes are a problem, the glycolic-nitrate pickle may be preferred to the acetic-nitrate pickle (3.4.7) to reduce acid loss from vaporization. The solution composition is indicated in Table II.
- 3.4.9 Spot weld chromic-sulphuric pickle: A chromic-sulphuric pickle may be used where parts are to be cleaned prior to spot welding. Parts are first treated by immersion in an alkaline cleaner and then given a thorough rinse in cold running water as specified in 3.3.2. Parts are then immersed in an alkali neutralizing pickle consisting of an aqueous solution of either 0.5 to 1 percent of sulphuric acid by volume or a 1 to 2 percent solution of sodium bisulphate (sodium acid sulphate) (weight by volume). Following the neutralizing dip, the parts are immersed in the chromic-sulphuric acid pickle. This treatment gives a low resistance surface for spot welding. The solution composition is indicated in Table II.

3.5 Type I treatment:

Cleaning of parts to receive the Type I or chrome pickle treatment shall be in accordance with other requirements of this specification.

- 3.5.1 Procedures for wrought parts: Wrought magnesium parts shall be treated in a chrome pickle bath whose solution composition and operation is stated in Table III. The parts shall be immersed and agitated in the bath for a period of time ranging from 1/2 minute in a solution containing the full quantity of nitric acid to 2 minutes in a solution in which the nitric acid is nearing depletion. Usually a one minute dip will be sufficient in a properly controlled bath. After the immersion period, the parts shall be held above the tank for 5 seconds. This allows the adhering solution to drain off and produce a better colored coating. The parts shall then be washed in cold running water followed by a dip in hot water, to facilitate drying, or by exposure to heated air. Other methods, such as spraying may be used to expose the parts to the chrome pickle solution, provided the other requirements of this specification are met.
- 3.5.2 Procedures for sand, permanent mold, and die castings: Magnesium sand, permanent mold, and die castings shall be treated in a chrome pickle bath as given in Table III. Die castings and aged sand castings shall be given a 15 to 30 second hot water dip followed immediately by immersion in the solution specified in Table III. A 10 second immersion is sufficient if the bath is operated at 120 to 140 °F. If the bath is held at a lower temperature a longer time is needed. Excessive treatment time results in a powdery coating and failure to pre-heat the castings in hot water may result in no coating. If the bath for castings is not available, the bath for wrought parts may be used for die castings and aged sand castings. Sand castings in the solution heat treated condition can be chrome pickled with the solution in Table III at room temperature. After dipping, parts shall be rinsed as described in 3.5.1.
- 3.5.3 Precautions: Chrome pickle baths (see Table III) may remove as much as 0.0006 inch of metal per surface during treatment and, therefore, cannot be used on machined surfaces unless tolerances will permit or proper allowances have been made. Magnesium parts containing steel inserts can be given the Type I treatment. The color, luster, and etch produced by the Type I treatment will vary with age of the solution, with the alloy composition, and with the condition of heat treatment of the alloy. The most desirable paint base is a matte gray to yellow-red, iridescent coating, which exhibits a network of pebbled etch finish as viewed under magnification. Bright, brassy coatings, which show a relatively smooth surface with only occasionally rounded pits when examined with magnification, shall be unsatisfactory where the treatment is used as a paint base, but shall be acceptable for protection during shipping and storage. This shade indicates an excess of nitric acid or nitrate salt buildup in the bath.

TABLE III
TYPE I CHROME PICKLE BATHS FOR TREATING MAGNESIUM ALLOYS

Composition <u>1/</u>						
Types for	Material	Oz.	Immersion time	Operating temperature °F	Tank construction	Rack and basket construction
Wrought parts	Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$) Nitric acid, concentrated (sp gr 1.42)	24	30 sec. to 2 min. followed by 5 sec. draining	70 to 110°	Stainless steel or tanks lined with glass, ceramics, synthetic rubber, or vinyl base materials.	Stainless steel or monel
		24				
Sand, permanent mold, and die castings	Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$) Nitric acid, concentrated (sp gr 1.42) Sodium, potassium, or ammonium acid fluoride (NaHF_2 , KHF_2 , NH_4HF_2) <u>2/</u>	24	30 sec. to 2 min. followed by 5 sec. draining	70 to 140°	Type 316 stainless steel preferred. Tanks lined with synthetic rubber or vinyl base materials.	Type 316 stainless steel
		16 to 24				
		2				
<u>1/</u> Avoirdupois or fluid ounces per gallon. Water is added to make 1 gallon. When available, water from steam condensate or treated by ion exchange should be used in preference to well or hard tap water. <u>2/</u> If sodium acid fluoride (NaHF_2) is used, add it before the nitric acid or dissolve in small quantity of water before adding since sodium acid fluoride does not go into solution with nitric acid present.						

3.5.4 Brush application: Articles too large to be immersed shall be carefully brushed with a generous amount of fresh pickle solutions specified in Table III. The solution shall be allowed to remain on the surface for at least 1 minute while brushing and shall then be washed off immediately with plenty of cold running water. The coating thus formed is less uniform in color than that produced by immersion but is equally good as a paint base. Powdery coatings are not good as paint bases and indicate poor rinsing or failure to keep the surface wet with solution during the one minute treatment time by continuous brushing over the same area. In treating riveted assemblies, care must be taken to avoid entrapping solution in the joints. Brush application is suitable for touchup of all types of treatments which have been damaged. Type I coatings are best for touchup of base areas before making an electrical bonding joint and where no primer can be used to seal the treatment.

3.5.4.1 Parts such as tanks which take a long time to fill and empty may be treated with chrome pickle solution for wrought parts diluted with an equal quantity of water. The time of treatment shall be such as to produce a complete coating on all areas. Similarly, if a more dilute solution is required to treat parts by automated time cycle, more water may be used in the pickle bath.