

AEROSPACE MATERIAL SPECIFICATION

Passivation of Corrosion Resistant Steels

1. SCOPE:

1.1 Purpose:

This specification covers the requirements for a process to assure removal of free iron or other less noble contaminants from the surfaces of corrosion resistant steel parts.

1.2 Application:

The processes defined in this specification have been used typically to remove metallic contaminants from the surfaces of corrosion resistant steels using chemically oxidizing methods to prevent injury to the basis metals, but usage is not limited to such applications.

1.3 Classification:

1.3.1 Methods: Passivation methods covered by this specification are as follows:

Method 1 - Passivation in Nitric Acid

Method 2 - Passivation in Citric Acid

Method 1 shall be used unless Method 2 is authorized by purchaser.

1.3.2 Types: The following types are presented for use when Method 1 is specified by purchaser:

- | | |
|--------|---|
| Type 1 | Low Temperature Nitric Acid with Sodium Dichromate |
| Type 2 | Medium Temperature Nitric Acid with Sodium Dichromate |
| Type 3 | High Temperature Nitric Acid with Sodium Dichromate |
| Type 4 | 40% Nitric Acid for Free Machining Steels |
| Type 5 | Anodic, for High Carbon Martensitic Steels |
| Type 6 | Low Temperature Nitric Acid |
| Type 7 | Medium Temperature Nitric Acid |
| Type 8 | Medium Temperature, High Nitric Acid Concentration |

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1.3.2 (Continued):

Where no type is specified, the processor may use any of the listed types that meet the requirements given herein, unless citric acid passivation in accordance with Method 2 is acceptable to purchaser.

1.3.3 Classes: Passivation verification classes are as follows:

1.3.3.1 Class 1: The following types of parts shall be verified by testing for passivity on a statistical sampling frequency basis.

1.3.3.1.1 Fasteners, including nuts, bolts, washers, rivets and related hardware where a test frequency is not defined in the procurement documents.

1.3.3.1.2 Standard parts defined by drawings labelled AN, MS, NAS or similar where frequency of test is not otherwise defined.

1.3.3.1.3 When specified by purchaser.

1.3.3.2 Class 2: Frequency of corrosion testing shall be one part per lot.

1.3.3.3 Class 3: Frequency of testing shall be on a periodic basis.

1.3.3.4 For parts other than fasteners and standard parts, class 2 shall apply unless another class is specified by purchaser.

1.4 Safety - Hazardous Materials:

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

2. APPLICABLE DOCUMENTS:

The issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been cancelled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or www.astm.org.

ASTM B 117 Operating Salt Spray (Fog) Testing Apparatus

2.2 ASQ Publications:

Available from American Society for Quality, 600 North Plankinton Avenue, Milwaukee, WI 53203 or www.asq.org.

ASQC Z1.4 Sampling Procedures and Tables for Inspection by Attributes

3. REQUIREMENTS:

3.1 Procedure:

3.1.1 Prior to passivating, parts shall be free of grease, foreign materials and scale (See 8.9).

3.1.2 Passivating shall be accomplished after completion of all manufacturing operations, such as but not limited to forming, turning, milling, heat treatment or shot peening, that could affect the passivity of the surface of the material. Where other surface altering operations are performed, such as electroplating or nitriding, purchaser shall specify when the passivation operation is accomplished within the manufacturing sequence.

3.1.3 Method 1 - Passivation in Nitric Acid:

3.1.3.1 Passivation shall be accomplished by immersion in a bath of an aqueous solution of 20 to 55% by volume of nitric acid (HNO_3) (40° Baume or specific gravity 1.4).

3.1.3.1.1 Where the acid concentration is less than 35% by volume, and for ferritic and martensitic steels, it is recommended that additional oxidizers be added to the bath in the form of 2 to 6% by weight of sodium dichromate dihydrate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$).

3.1.3.1.2 For additional oxidation potential, it is permissible to use up to 6% by weight of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) instead of sodium dichromate in the passivating solution.

3.1.3.1.3 For the purpose of removing lead alloys from surfaces, molybdic acid (HMoO_3) may be added to the bath at a concentration of up to 0.35 weight percent.

3.1.3.1.4 Iron concentration in the passivating bath shall not exceed two weight percent.

3.1.3.1.5 For certain high carbon corrosion resistant steels, such as AISI 440C, it may be desirable to passivate with the parts anodic for 2 to 3 minutes at 2 to 3 volts to prevent etching.

3.1.3.2 Operating Conditions:

3.1.3.2.1 When a specific type of passivation as 1.3.2 is not specified, bath temperature shall be in the range of 70 to 155 °F (21 to 68 °C) with an immersion time of not less than 30 minutes for baths operating at temperatures below 100 °F (37 °C), not less than 20 minutes for baths operating at temperatures below 125 °F (52 °C), or not less than 10 minutes for baths operating at temperatures above 125 °F (52 °C).

3.1.3.2.2 When a specific passivating type is specified, the times, temperatures, and solutions used for passivating shall be as specified in Table 1.

TABLE 1 - Passivation Types

| Type | Feature | Value |
|------|------------------|---|
| 1 | Bath Composition | 20 to 25% by volume of HNO_3 2 to 3% by weight $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ |
| | Bath temperature | 70 to 90 °F (21 to 32 °C) |
| | Immersion time | 30 minutes minimum |
| 2 | Bath Composition | 20 to 25% by volume of HNO_3 2 to 3% by weight $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ |
| | Bath temperature | 120 to 130 °F (49 to 54 °C) |
| | Immersion time | 20 minutes minimum |
| 3 | Bath Composition | 20 to 25% by volume of HNO_3 2 to 3% by weight $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ |
| | Bath temperature | 145 to 155 °F (63 to 68 °C) |
| | Immersion time | 10 minutes minimum |
| 4 | Bath Composition | 38 to 42% by volume of HNO_3 2 to 3% by weight $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ |
| | Bath temperature | 70 to 120 °F (21 to 32 °C) |
| | Immersion time | 30 minutes minimum |
| 5 | Bath Composition | 20 to 25% by volume of HNO_3 2 to 3% by weight $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ |
| | Bath temperature | 70 to 90 °F (21 to 32 °C) |
| | Immersion time | 2 minutes minimum |
| | Voltage | Part anodic at 3 to 5 volts |
| 6 | Bath Composition | 25 to 45% by volume HNO_3 |
| | Bath temperature | 70 to 90 °F (21 to 32 °C) |
| | Immersion time | 30 minutes minimum |
| 7 | Bath Composition | 20 to 25% by volume HNO_3 |
| | Bath temperature | 120 to 140 °F (49 to 60 °C) |
| | Immersion time | 20 minutes minimum |
| 8 | Bath Composition | 45 to 55% by volume HNO_3 |
| | Bath temperature | 120 to 130 °F (49 to 54 °C) |
| | Immersion time | 30 minutes |

3.1.4 Method 2 - Passivation in Citric Acid:

3.1.4.1 Bath Composition: Parts shall be immersed in an aqueous solution of 4 to 10 weight percent citric acid, with additional wetting agents and inhibitors as applicable.

3.1.4.2 Operating Conditions:

3.1.4.2.1 Temperature: Bath temperature shall be 70 to 160 °F (21 to 60 °C) with an immersion time of not less than 4 minutes for baths operating over 140 °F (60 °C), not less than 10 minutes for baths operating in the 120 to 140 °F (49 to 60 °C) range, or not less than 20 minutes for baths operating below 120 °F (49 °C).

3.1.5 Post Treatment: When a post treatment is specified, after rinsing, parts made from ferritic, martensitic and precipitation hardening steels shall be immersed in a solution containing 4 to 6% by weight of sodium dichromate dihydrate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$) at 140 to 160 °F (60 to 71 °C) for one hour, rinsed, and dried (See 8.2).

3.2 Corrosion Resistance:

When tested, parts made from alloys containing less than 0.85% carbon shall meet one of the following conditions, or, when specified, a test method in Annex A.

3.2.1 Humidity: Parts shall be free from visible red rust after exposure to 95% minimum relative humidity at 95 to 115 °F (35 to 46 °C) for not less than 23 hours.

3.2.2 Water Immersion: Parts shall be free from visible red rust after alternately immersing in deionized or distilled water for one hour and allowing to dry in room temperature air for one hour, until 24 hours (12 cycles) have elapsed.

3.2.3 When specified or when permitted by purchaser, (1) for austenitic steels of the AISI 200 or AISI 300 series and (2) for precipitation hardened steels and ferritic steels containing more than 16% chromium, one of the following requirements shall be met in lieu of humidity or immersion testing:

3.2.3.1 Copper Sulfate Solution: Parts shall be swabbed with a test solution containing 8 grams of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) and 2 to 3 mL of sulfuric acid (H_2SO_4 , sp. gr. 1.84) in 500 mL of distilled or deionized water, keeping the surface wet for not less than six minutes. Rinse and dry the surface without disturbing any deposits. A copper colored deposit indicates the presence of unacceptable free iron.

3.2.3.2 Salt Spray: Parts shall withstand exposure to 2 hours \pm 10 minutes in a salt spray environment operated in accordance with ASTM B 117. Parts shall not show evidence of red rust following completion of the test.

3.3 Surface Appearance:

After completion of processing, there shall be no evidence of etching, pitting, smutting, frosting, dimensional changes, or other chemical attack on the parts. However, loss of temper color when Method 2 is used is acceptable.

3.4 Written Procedure:

All processing and testing shall be done in accordance with a written procedure acceptable to purchaser (See 4.4.3).

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

The processor shall supply all test specimens for processor's tests and shall be responsible for the performance of all required tests. When parts are to be tested, such parts shall be supplied by purchaser, and, if acceptable after testing, may be included with the lot of processed parts. Purchaser reserves the right to perform any confirmatory testing deemed necessary to ensure that processing conforms to specified requirements.

4.2 Classification of Tests:

4.2.1 Acceptance Tests:

4.2.1.1 Classes 1 and 2: Corrosion resistance (3.2) and surface appearance (3.4) are acceptance tests and shall be performed on each lot.

4.2.1.2 Class 3: Surface appearance (3.3) is an acceptance test and shall be performed on each lot.

4.2.2 Periodic Tests:

4.2.2.1 Composition of passivating and post treatment solutions (See 8.10) are periodic tests, and shall be performed at a frequency selected by the processor unless frequency of testing is specified by purchaser.

4.2.2.2 Class 3 Parts: Corrosion resistance is a periodic test and shall be performed at a frequency selected by the processor unless frequency of testing is specified by purchaser.

4.2.3 Preproduction Tests: All technical requirements of this specification are preproduction tests, and shall be performed prior to or on the initial shipment of each part number to a purchaser, or when a change in processing requires reapproval as in 4.4.2, or when purchaser deems confirmatory testing is required.

4.3 Sampling and Testing:

A lot shall be all parts of the same part number, processed in the same set of solutions within a 24 hour period, and presented for processor's inspection at the same time. Tested parts shall be selected randomly from all parts in each lot.

- 4.3.1 Class 1 Parts: Unless purchaser specifies a different sampling plan, the minimum number of samples selected for test shall be as shown in Table 2.

TABLE 2 - Number of Parts to be Tested

| Number of Parts in Lot | Visual | Corrosion Test |
|---------------------------|--------|-------------------|
| 1 | 1 | 1 |
| 2 to 6 | All | 2 |
| 7 to 15 | 7 | 2 |
| 16 to 40 | 10 | 3 |
| 41 to 51 | 15 | 3 |
| 51 to 110 | 15 | 5 |
| 111 to 150 | 25 | 8 |
| 151 to 500 | 35 | 8 |
| 501 to 701 | 50 | 13 |
| 701 to 1200 | 75 | 13 |
| Over 1200 | 125 | 13 |

- 4.3.2 Class 2 parts or samples shall be corrosion tested at a frequency of one part per lot, and visually examined at the frequency given in Table 2.
- 4.3.3 Where parts are not available for test, as in the case of large parts or parts that might be damaged by such testing, identically processed specimens fabricated from the same alloy as the parts represented may be used. See 8.11.

4.4 Approval:

- 4.4.1 The process and control procedures, or a preproduction processed part, or both, whichever is specified, shall be approved by the cognizant engineering organization before production parts are supplied.
- 4.4.2 The processor shall make no significant change to materials, processes or controls from those on which the approval was based, unless the change is approved by the cognizant engineering organization. A significant change is one which, in the judgment of the cognizant engineering organization, could affect the properties or performance of the parts.

4.4.3 Control factors shall include, but are not limited to the following:

Composition and composition limits of the processing solutions
Temperature and temperature limits of the processing solutions
Immersion time limits of the process for each processing solution
Method(s) for precleaning in preparation for passivating (See 3.1.1)
Method(s) used to test for corrosion resistance
Periodic test plan (See 8.10)

4.5 Reports:

The processor shall furnish with each shipment a report stating that parts have been processed and tested in accordance with the specified requirements and that they conform to acceptance tests requirements. Where post treatment is used, the report shall so indicate that it was completed. The report shall state the type or (if applicable) method of passivation used. This report shall also include the purchase order number, lot number, AMS 2700B, part number, and quantity of parts processed.

4.6 Resampling and Retesting:

4.6.1 If any part in the lot exhibits etching or frosting, that part shall be subject to rejection, and all parts in the lot shall be visually examined or subject to rejection.

4.6.2 If any part subjected to corrosion testing fails to meet corrosion test requirements, that part shall be subject to rejection. The balance of the lot may be reprocessed and retested at the frequency defined by Table 2 for the original number of parts in the lot, or the remaining parts in the lot shall be tested.

5. PREPARATION FOR DELIVERY:

Packages of passivated parts shall be prepared for shipment in accordance with commercial practice and in compliance with applicable rules and regulations pertaining to the handling, packaging, and transportation of the processed parts.

6. ACKNOWLEDGMENT:

A processor shall mention this specification number and its revision letter in all quotations and when acknowledging purchase orders.

7. REJECTIONS:

Parts that are not processed in accordance with the requirements of this specification or to modifications authorized by purchaser will be subject to rejection.

8. NOTES:

- 8.1 A change bar (I) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of a specification. An (R) symbol to the left of the document title indicates a complete revision of the specification, including technical revision. Change bars and (R) are not used in original publications, nor in specifications that contain editorial changes only.
- 8.2 When post treatment is not specified, parts should be neutralized, preferably in a solution of 2 to 5% sodium hydroxide, rinsed and dried.
- 8.3 Terms used in AMS are clarified in ARP1917.
- 8.4 Dimensions and properties in inch-pound units and the Fahrenheit temperatures are primary; dimensions and properties in SI units and the Celsius temperatures are shown as approximate equivalents of the primary units and are presented only for information.
- 8.5 Purchase documents should specify not less than the following:
- AMS 2700B
 - Material being processed
 - Quantity of parts to be processed
 - Method (See 1.3.1), type (See 1.3.2), or class (See 1.3.3) if required
 - Test method in Annex A, if required
 - Post treatment when required (See 3.1.5).
- 8.6 These processes have been used primarily to enhance the corrosion resistance of corrosion resistant steel alloys, but the nitric acid process has also been successfully and historically applied to nickel-chromium high temperature alloys for removal of free iron resulting from machining or other processing. Different types of smeared metal on the corrosion resistant surfaces, or the presence of other surface treatments such as plating or braze filler metals may dictate the use of either nitric acid or citric acid as applicable to the specific case.
- 8.7 It is recommended that the concentration of the nitric acid be above 40% for free machining steels.
- 8.8 It is recommended that this process be used prior to heating corrosion resistant steel parts to temperatures exceeding 1200 °F (649 °C) to prevent diffusion of free iron from the surface into the surfaces of parts.
- 8.9 This document does not address methods for removal of scale or foreign materials from the surfaces of parts prior to passivation. Methods for accomplishing this may be found in such other documents as ISO 8074, ISO 8075 or ASTM A 380.
- 8.10 ARP4992, Periodic Test Plan for Process Solutions, is recommended to satisfy the requirements for control of processing solutions.

8.11 “Identically processed” as used in 4.3.3 refers to such operations as machining, grinding, heat treating, welding, and similar processes.

8.12 Carburized and nitrided surfaces should not be passivated.

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PREPARED UNDER THE JURISDICTION OF AMS COMMITTEE “B”

ANNEX A
Test Methods for Determination of Passivity

A.1 SCOPE:

The test methods in this annex are to be used only when specified by the purchaser.

A.1.1 Safety - Hazardous Materials:

While the materials, methods, applications, and processes described or referenced in this annex may involve the use of hazardous materials, this annex does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

A.2 APPLICABLE DOCUMENTS:

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A.2.1 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or www.astm.org.

ASTM B 117 Operating Salt Spray Testing Apparatus

A.2.2 ASQ Publications:

Available from American Society for Quality, 600 North Plankinton Avenue, Milwaukee, WI 53203 or www.asq.org.

ASQC Z1.4 Sampling Procedures and Tables for Inspection by Attributes

A.3 TEST REQUIREMENTS:

A.3.1 Method 100 - Water Immersion Test:

This method is used to detect anodic surface contamination, including free iron, on corrosion resistant steel.

A.3.1.1 Apparatus and Materials: A tank that will not rust and reagent grade water.

A.3.1.2 Procedure:

A.3.1.2.1 Parts shall be alternately immersed in reagent grade water for one hour, removed from the tank, and allowed to dry for one hour for a minimum of 24 hours.

A.3.1.2.2 After completion of the test, parts shall show no evidence of rust or corrosion.

A.3.2 Method 101 - High Humidity Test:

This method is used to detect anodic surface contamination on corrosion resistant steel, including free iron.

A.3.2.1.1 Apparatus and Materials: A humidity cabinet capable of maintaining the conditions specified herein.

A.3.2.2 Procedure:

A.3.2.2.1 Parts shall be placed in a humidity cabinet and exposed to $97 \pm 3\%$ relative humidity at $100^\circ\text{F} \pm 5$ ($38^\circ\text{C} \pm 3$) for 24 hours minimum.

A.3.2.2.2 After completion of the test, parts shall show no evidence of rust or corrosion.

A.3.3 Method 102 - Copper Sulfate Test:

This method is recommended to detect the presence of free iron on the surface of austenitic chromium-nickel steels of the AISI 200 and 300 series alloys, precipitation hardened types, and ferritic AISI 400 series alloys having a minimum of 16% chromium. It is not recommended for use on martensitic AISI 400 series alloys or ferritic AISI 400 series alloys with less than 16% chromium because the test will be positive for presence of iron. The test is sensitive and should be used and interpreted only by personnel familiar with its limitations. The test should not be used on parts to be used for food processing.

A.3.3.1 Apparatus:

10 ml graduated cylinder
500ml graduated cylinder
1000 ml beaker
Cotton swab
Balance or scale