

## Passivation of Corrosion Resistant Steels

### 1. SCOPE:

#### 1.1 Purpose:

This specification defines the engineering 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

Either method may be used unless a method is specified 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

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.

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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. 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 form 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 19248-2959.

ASTM B 117 Operating Salt Spray Testing Apparatus

3. REQUIREMENTS:

3.1 Procedure:

3.1.1 Prior to passivating, parts shall be free of grease, foreign materials and scale (See 8.8).

- 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 weight of nitric acid ( $\text{HNO}_3$ ) (40° Baume or specific gravity 1.4).
- 3.1.3.1.1 Where the acid concentration is less than 35% by weight, 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 ( $\text{HMoO}_3$ ) 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 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).
- 3.1.3.3 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 weight of HNO <sub>3</sub> 2 to 3% by weight Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ·2H <sub>2</sub> O
	Bath temperature	70 to 90 °F (21 to 32 °C)
	Immersion time	30 minutes minimum
2	Bath Composition	20 to 25% by weight of HNO <sub>3</sub> 2 to 3% by weight Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ·2H <sub>2</sub> O
	Bath temperature	120 to 130 °F (49 to 54 °C)
	Immersion time	20 minutes minimum
3	Bath Composition	20 to 25% by weight of HNO <sub>3</sub> 2 to 3% by weight Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ·2H <sub>2</sub> O
	Bath temperature	145 to 155 °F (63 to 68 °C)
	Immersion time	10 minutes minimum
4	Bath Composition	38 to 42% by weight of HNO <sub>3</sub> 2 to 3% by weight Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ·2H <sub>2</sub> O
	Bath temperature	70 to 120 °F (21 to 32 °C)
	Immersion time	30 minutes minimum
5	Bath Composition	20 to 25% by weight of HNO <sub>3</sub> 2 to 3% by weight Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ·2H <sub>2</sub> 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	20 to 50% by weight HNO <sub>3</sub>
	Bath temperature	70 to 90 °F (21 to 32 °C)
	Immersion time	30 minutes minimum
7	Bath Composition	20 to 25% by weight HNO <sub>3</sub>
	Bath temperature	120 to 140 °F (49 to 60 °C)
	Immersion time	20 minutes minimum
8	Bath Composition	45 to 55% by weight HNO <sub>3</sub>
	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.

#### 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 shall meet one of the following conditions:

3.2.1 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 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, for austenitic steels of the AISI 200 or AISI 300 series only, one of the following requirements shall be met in lieu of humidity or immersion testing:

3.2.3.1 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 ( $\text{H}_2\text{SO}_4$ , 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 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 processing vendor shall supply all test specimens for vendor'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 the requirements of this specification.

### 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 8.9) 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 vendor'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 corrosion 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 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 samples may be used.
- 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 vendor of processed parts 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