Designation: A967/A967M - 17

Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts¹

This standard is issued under the fixed designation A967/A967M; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

- 1.1 This specification covers several different types of chemical passivation treatments for stainless steel parts. It includes recommendations and precautions for descaling, cleaning, and passivation of stainless steel parts. It includes several alternative tests, with acceptance criteria, for confirmation of effectiveness of such treatments for stainless steel parts.
- 1.2 Practices for the mechanical and chemical treatments of stainless steel surfaces are discussed more thoroughly in Practice A380/A380M.
- 1.3 Several alternative chemical treatments are defined for passivation of stainless steel parts. Appendix X1 and Appendix X2 give some nonmandatory information and provides some general guidelines regarding the selection of passivation treatments appropriate to particular grades of stainless steel. This specification makes no recommendations regarding the suitability of any grade, treatment, or acceptance criteria for any particular application or class of applications.
- 1.4 The tests in this specification are intended to confirm the effectiveness of passivation, particularly with regard to the removal of free iron and other exogenous matter. These tests include the following practices:
 - 1.4.1 Practice A—Water Immersion Test,
 - 1.4.2 *Practice B*—High Humidity Test,
 - 1.4.3 *Practice C*—Salt Spray Test,
 - 1.4.4 Practice D—Copper Sulfate Test,
- 1.4.5 Practice E—Potassium Ferricyanide-Nitric Acid Test, and
 - 1.4.6 Practice F—Damp Cloth Test, and
 - 1.4.7 *Practice G*—Boiling Water Immersion Test.

Note 1—Free iron denotes iron present on the surface of the parts, including but not limited to iron contamination, iron-tool marks, residualiron salts from pickling solutions, iron dust, atmospheric exposure, iron deposits in welds, embedded iron, and iron oxide.

- 1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.
- 1.6 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.
- 1.7 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

A380/A380M Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems

B117 Practice for Operating Salt Spray (Fog) Apparatus

B254 Practice for Preparation of and Electroplating on Stainless Steel

B600 Guide for Descaling and Cleaning Titanium and Titanium Alloy Surfaces

B912 Specification for Passivation of Stainless Steels Using Electropolishing

2.2 Federal Specification:³

QQ-P-35C Passivation Treatments for Corrosion-Resistant Steels⁴

¹ This specification is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloys and is the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

Current edition approved July 1, 2017. Published July 2017. Originally approved in 1996. Last previous edition approved in 2013 as A967/A967M – 13. DOI: 10.1520/A0967-17.

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.

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁴ In accordance with QQ-P-35C Notice 3, September 11, 1998, Specification QQ-P-35C is cancelled and Specification A967/A967M should be used in its place for DoD activities other than aerospace applications.

3. Terminology

- 3.1 Definition of Term Specific to This Standard—It is necessary to define which of the several commonly used definitions of the term *passivation* will be used in this specification. (See Discussion.)
- 3.1.1 Discussion—Stainless steels are autopassivating in the sense that the protective passive metal oxide film is formed spontaneously on exposure to air or moisture. The presence of exogenous surface contamination, including dirt, grease, free iron from contact with steel tooling, and so forth, may interfere with the formation of the passive metal oxide film. The cleaning of these contaminants from the stainless steel surface will facilitate the spontaneous passivation by allowing the oxygen uniform access to the surface. The passive metal oxide film may be augmented by chemical treatments that provide an oxidizing environment for the stainless steel surface.
- 3.1.1.1 In this specification, passivation, unless otherwise specified, is defined as the chemical treatment of a stainless steel with a mild oxidant, such as a nitric acid solution, for the purpose of the removal of free iron or other foreign matter, but which is generally not effective in removal of heat tint or oxide scale on stainless steel. In the case of stainless steels with additions of sulfur for the purpose of improved machinability, passivation may also include the removal of sulfides from the surface of the metal for the purpose of maximizing corrosion resistance.
- 3.1.1.2 The formation of the protective passive metal oxide film on a stainless steel, also called passivation in a more general context, will occur spontaneously in air or other oxygen-containing environment when the stainless steel surface is free of oxide scale and exogenous matter.
- 3.1.1.3 Chemical treatments, such as sodium dichromate solutions, may facilitate the more rapid formation of the passive film on a stainless steel surface already free of scale or foreign matter. Such treatments, also sometimes called passivation in common usage, are designated as post-cleaning treatments in this specification in order to distinguish them from chemical treatments capable of removing free iron from stainless steels.
- 3.1.1.4 The chemical treatments capable of removing heat tint or oxide scale from stainless steel and capable of dissolving the stainless steel itself, typically called pickling, are substantially more aggressive than treatments used for passivation, as defined in 3.1.1.1. The surface of stainless steel that has been pickled is free of scale, free iron, and exogenous foreign matter, and does not require a separate treatment for passivation as defined in 3.1.1.1. The passivation process defined in 3.1.1.2 will occur without further chemical treatment but may be augmented and improved by the post-cleaning treatments defined in 3.1.1.3.
- 3.1.1.5 Electrochemical treatments, including electropick-ling and electropolishing capable of removing heat tint or oxide scale from stainless steel and capable of dissolving the stainless steel itself, are substantially more aggressive than treatments used for passivation, as defined in 3.1.1.1. The surface of stainless steel resulting from these treatments is free of scale, free iron, and exogenous foreign matter, and does not require a separate treatment for passivation as defined in 3.1.1.1. The

passivation process defined in 3.1.1.2 will occur without further chemical treatment, but may be augmented and improved by the post-cleaning treatments defined in 3.1.1.3. Statements regarding chemical treatments, unless otherwise specified, are taken to include electrochemical treatments.

4. Ordering Information

- 4.1 This specification was written for the purpose of providing an alternative to United States Federal Specification QQ-P-35C. Determination of the suitability of this specification for that purpose is the responsibility of the purchaser.
- 4.2 Unless specified by the purchaser, the chemical treatment applied to the stainless steel parts shall be selected by the supplier from among the listed passivation treatments. (See 5.1.)
- 4.3 Unless specified by the purchaser, the test practice(s) and test frequency applied to the stainless steel parts shall be selected by the supplier from among the listed test practices. (See Section 13.)
- 4.4 The purchaser may also specify additional requirements, including the following:
 - 4.4.1 Post-cleaning treatments. (See Section 10.)
- 4.4.2 Availability of reports and records. (See 5.2.2 and Section 17.)

5. Materials and Preparation for Passivation Treatments

- 5.1 The passivation treatments shall be of one or more of the following types. The effectiveness of a particular treatment for a particular grade of stainless steel in a particular application is demonstrated by meeting the specified testing requirements:
 - 5.1.1 Treatments in nitric acid solutions (see Section 6),
 - 5.1.2 Treatments in citric acid solutions (see Section 7).
- 675.1.3 Treatments in other chemical solutions, including electrochemical treatments (see Section 8),
 - 5.1.4 Rinsing and neutralization (see Section 9), and
 - 5.1.5 Post-cleaning treatments (see Section 10).
 - 5.2 Materials:
- 5.2.1 The chemicals used for passivation treatments shall produce passivated surfaces that meet the requirements of one or more of the tests of this specification. Attention shall be given to maintaining adequate volume appropriate to the size and amount of stainless steel to be treated, as well as concentration and temperature control appropriate to the selected treatment. (See Note 2.)

Note 2—Attention should also be given to maintaining good cleanliness of the passivation solution to ensure the production of acceptable surfaces that meet the requirements.

5.2.2 The supplier shall maintain a record with regard to concentration and temperature of the passivation solution sufficient to demonstrate that the specified passivation conditions were maintained for each lot of stainless steel parts processed. Such records shall be available for inspection when specified in the purchase order. The supplier is not required by this specification to reveal the precise composition of proprietary chemical mixtures but shall maintain a unique identification of the mixture that will ensure its accurate representation for subsequent use.

- 5.2.3 The supplier shall be responsible for the safe disposal of all material generated by this process.
 - 5.3 Preparation for Passivation Treatments:
- 5.3.1 Any pretreatment methods and procedures used prior to the passivation treatment, including mechanical and chemical methods, singly or in combination, for descaling and pickling, shall be in accordance with Practice A380/A380M. When electrochemical cleaning is required, it shall be performed in accordance with Practice B254.
- 5.3.2 The resulting pretreated surface shall be substantially free of oil, grease, rust, scale, and other foreign matter.
- 5.3.3 When the final pretreatment of a part includes pickling of the entire surface of the part, no further passivation treatment is required prior to testing of the surface unless specified by the purchaser.

6. Treatments in Nitric Acid Solutions

- 6.1 Passivation Treatment:
- 6.1.1 Stainless steel parts shall be treated in one of the following aqueous solutions and maintained within the specified temperature range for the specified time. Where immersion is impractical, other methods of maintaining contact between the stainless steel surface and the solution within the specified temperature range for the specified time may be used.
- 6.1.1.1 Nitric 1—The solution shall contain 20 to 25 volume percent of nitric acid and 2.5 ± 0.5 weight percent of sodium dichromate dihydrate. The parts shall be immersed for a minimum of 20 min at a temperature in the range from 50 to 55° C [120 to 130° F].
- 6.1.1.2 *Nitric* 2—The solution shall contain 20 to 45 volume percent of nitric acid. The parts shall be immersed for a minimum of 30 min at a temperature in the range from 20 to 30°C [70 to 90°F].
- 6.1.1.3 *Nitric 3*—The solution shall contain 20 to 25 volume percent of nitric acid. The parts shall be immersed for a minimum of 20 min at a temperature in the range from 50 to 60°C [120 to 140°F].
- 6.1.1.4 *Nitric* 4—The solution shall contain 45 to 55 volume percent of nitric acid. The parts shall be immersed for a minimum of 30 min at a temperature in the range from 50 to 55°C [120 to 130°F].
- 6.1.1.5 *Nitric* 5—Other combinations of temperature, time, and concentration of nitric acid, with or without other chemicals, including accelerants, inhibitors, or proprietary solutions, capable of producing parts that pass the specified test requirements.
- 6.2 Water Rinse—Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, countercurrent, or spray washes, singly or in combination, with or without a separate chemical treatment for neutralization of the passivation media (see Section 9), with a final rinse being carried out using water with a maximum total solids content of 200 ppm.

Note 3—Each of the nitric acid solution treatments listed are recommended for different grades of stainless steel. See X1.3 or X2.1.

7. Treatments in Citric Acid Solutions

7.1 Passivation Treatment:

- 7.1.1 Stainless steel parts shall be treated in one of the following aqueous solutions and maintained within the specified temperature range for the specified time. Where immersion is impractical, other methods of maintaining contact between the stainless steel surface and the solution within the specified temperature range for the specified time may be used.
- 7.1.1.1 *Citric 1*—The solution shall contain 4 to 10 weight percent of citric acid. The parts shall be immersed for a minimum of 4 min at a temperature in the range from 60 to 70° C [140 to 160° F].
- 7.1.1.2 *Citric* 2—The solution shall contain 4 to 10 weight percent of citric acid. The parts shall be immersed for a minimum of 10 min at a temperature in the range from 50 to 60° C [120 to 140°F].
- 7.1.1.3 *Citric 3*—The solution shall contain 4 to 10 weight percent of citric acid. The parts shall be immersed for a minimum of 20 min at a temperature in the range from 20 to 50°C [70 to 120°F].
- 7.1.1.4 *Citric 4*—Other combinations of temperature, time, and concentration of citric acid, with or without other chemicals to enhance cleaning, including accelerants, inhibitors, or proprietary solutions, capable of producing parts that pass the specified test requirements.
- 7.1.1.5 *Citric* 5—Other combinations of temperature, time, and concentration of citric acid, with or without other chemicals to enhance cleaning, including accelerants, inhibitors, or proprietary solutions, capable of producing parts that pass the specified test requirements. Immersion bath to be controlled at a pH of 1.8 to 2.2.
- 7.2 Water Rinse—Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, countercurrent, or spray washes, singly or in combination, with or without a separate chemical treatment for neutralization of the passivation media (see Section 9), with a final rinse being carried out using water with a maximum total solids content of 200 ppm.

Note 4—For some grades of stainless steel, additional considerations may be recommended. See X2.3 and X2.5.

8. Treatments in Other Chemical Solutions, Including Electrochemical Treatments

- 8.1 It is recognized that the purpose of removal of all exogenous matter from a stainless steel surface, including the removal of free iron, can be accomplished by different media, with potential for benefits to be gained from use of proprietary skills and art, including proprietary passivation media. Such treatments may include externally applying an electrical potential on the stainless steel parts, as in the case of electropolishing. The suitability of such passivation treatments for use in meeting the requirements of this specification shall be determined by the capability of the processed parts meeting the specified test requirements.
- 8.2 Stainless steel parts shall be treated in a specified aqueous solution, with or without externally applied electrical potential, and maintained within a specified temperature range for a time sufficient for the processed parts to meet the specified test requirement.

8.3 Water Rinse—Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, countercurrent, or spray washes, singly or in combination, with or without a separate chemical treatment for neutralization of the passivation media (see Section 9), with a final rinse being carried out using water with a maximum total solids content of 200 ppm.

Note 5—See also Specification B912.

9. Rinsing and Neutralization

- 9.1 The chemical reactions of the passivating media on the surface of the stainless steel shall be stopped by rinsing of the stainless steel part, with or without a separate neutralization treatment.
- 9.2 The suitability of a neutralization procedure is determined by the capability of the processed parts meeting the specified test requirements. (See Note 6.)

Note 6—The selection of medium and procedures for a neutralization depends of the chemistry of the passivation and on economic considerations. An example of a neutralizing treatment would be immersion of the part for a minimum of 30 min in a solution of 5 % NaOH at 70 to 80° C [160 to 180° F], followed by a water rinse.

10. Post-Cleaning Treatments

- 10.1 Although the passive metal oxide film characteristic of stainless steel will form spontaneously in air or any other oxygen-containing environment, the supplier shall, when specified by the purchaser, apply a chemical treatment that will accelerate the formation of the passive metal oxide film on a chemically clean stainless steel surface. Examples of mediums that serve to accelerate the formation of the passive film but do not contribute to the removal of free iron from the stainless steel surface are aqueous solutions of sodium dichromate or peroxide.
- 10.2 When specified by the purchaser, within one hour after the final water rinse as required in 6.2, 7.2, or 8.3, all ferritic and martensitic steel parts shall be immersed in an aqueous solution containing 4 to 6 weight percent of sodium dichromate at a temperature in the range from 60 to 70°C [140 to 160°F] for a minimum of 30 min, followed by a rinse in accordance with 6.2, 7.2, or 8.3. The parts shall then be thoroughly dried.
- 10.3 The purchaser may specify other post-cleaning treatments.

11. Finish

11.1 The passivated parts shall exhibit a chemically clean surface and shall, on visual inspection, show no etching, pitting, or frosting resulting from the passivation procedures.

12. Testing Agency

12.1 When required, the purchaser shall be permitted to perform such inspections as necessary to determine that the testing agency is capable of performing the specified test.

13. Lot, Frequency of Testing, and Selection of Test

13.1 *Definition of Lot*—A lot shall consist of one of the following, at the option of the supplier:

- 13.1.1 The passivated parts of similar alloy and manufacturing methods that are pretreated and passivated in a single day or within a time frame that will ensure consistent passivation results;
- 13.1.2 The passivated parts of the same product of one size from one heat in one shipment; or
- 13.1.3 When few parts are involved, the passivated parts from an entire production run.
- 13.2 Unless a greater frequency of testing is specified by the purchaser, one test per lot shall be sufficient.
- 13.3 One of the tests listed in Section 14, or more when specified on the purchase order, shall be performed on each lot of stainless steel parts to verify the effectiveness of the passivation treatment. It is important to note that not all of the following tests are suitable for all grades of stainless steel. (See Note 7.)
 - 13.3.1 *Practice A*—Water Immersion Test (see 14.1),
 - 13.3.2 *Practice B*—High Humidity Test (see 14.2),
 - 13.3.3 Practice C—Salt Spray Test (see 14.3),
 - 13.3.4 Practice D—Copper Sulfate Test (see 14.4),
- 13.3.5 *Practice E*—Potassium Ferricyanide-Nitric Acid Test (see 14.5),
 - 13.3.6 Practice F—Damp Cloth Test (see 14.6), and
- 13.3.7 Practice G—Boiling Water Immersion Test (see 14.7).

Note 7—Some of the tests may produce positive indications not associated with the presence of free iron on the stainless steel surface. An example would be application of Practice C on some lesser-alloyed martensitic or ferritic stainless steels.

14. Verification Tests

- 14.1 Practice A—Water Immersion Test
- 14.1.1 This test is used for the detection of free iron or any other anodic surface contaminants on stainless steel.
- 14.1.2 The sample representing the lot of passivated parts shall be alternately immersed in a non-rusting tank of distilled water for 1 h \pm 3 min and allowed to dry in air for at least 1 h. If the tank is metallic, the parts shall not be in contact with it; a polymeric support should be used. This cycle shall be repeated a minimum of twelve times.
- 14.1.3 The tested sample shall not exhibit rust or staining attributable to the presence of free iron particles embedded in the surface.
 - 14.2 Practice B—High Humidity Test
- 14.2.1 This test is used for the detection of free iron or any other anodic surface contaminants on stainless steel.
- 14.2.2 The test shall be performed using a humidity cabinet capable of maintaining the specified test conditions.
- 14.2.3 The sample representing the lot of passivated parts shall be subjected to 97 \pm 3 % humidity at 35 to 40°C [95 to 105°F] for a minimum of 24 h.
- 14.2.4 The tested sample shall not exhibit rust or staining attributable to the presence of free iron particles embedded in the surface.
 - 14.3 Practice C—Salt Spray Test
- 14.3.1 This test is used for the detection of free iron or any other anodic surface contaminants on stainless steel.