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Standard Guide for Descaling and Cleaning Titanium and Titanium Alloy Surfaces[1]

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

1. Scope

1.1 This guide covers a cleaning and descaling procedure useful to producers, users, and fabricators of titanium and titanium alloys for the removal of ordinary shop soils, oxides, and scales resulting from heat treatment operations and foreign substances present as surface contaminants.

1.2 It is not intended that these procedures be mandatory for removal of any of the indicated soils but rather serve as a guide when titanium and titanium alloys are being processed in the wrought, cast, or fabricated form.

1.3 It is the intent that these soils be removed prior to chemical milling, joining, plating, fabrication, and in any situation where foreign substances interfere with the corrosion resistance, stability, and quality of the finished product.

1.4 Acid etching may be required following cleaning when the surface has an oxygen-contaminated layer or alpha case present.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are for information only.

1.6 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Processing Soil Removal

2.1 It is recommended that grease, oil, and lubricants employed in machining, forming, and fabricating operations on titanium and titanium alloys be removed by alkaline or emulsion soak-type cleaners and electrolytic alkaline cleaning systems. In the electrolytic system the work may be either anodic or cathodic polarity. Removal of these soils is recommended prior to heat treatment or application of acid treatment designated in 4.2. When electrolytic systems are employed, the voltage should be controlled to prevent the occurrence of spark discharge and subsequent pitting.

3. Blast Cleaning

3.1 Mechanical descaling methods such as sandblasting, shot blasting, and vapor blasting may be used to remove hot work scales and lubricants from titanium surfaces if followed by thorough conditioning and cleaning as described in Section 4.

3.2 The sand used for blasting should be a high-grade, washed, iron-free, silica sand. If carbon or low-alloy steel products are sandblasted in the same facility, the sand used for cleaning these products should not be used on titanium surfaces and a separate sand supply should be provided.

3.3 Roughening of exposed surface areas may occur from grit or shot if cleaning of the entire surface is accomplished by blasting. Partial cleaning for preserving the surface finish is to be preferred in conjunction with proper pickling procedures.

3.4 Blast cleaning that utilizes steel grit or sand containing appreciable amounts of iron should be followed by acid pickling to remove any embedded steel particles on the surface of the titanium.

3.5 Any abrasive or shot blast cleaning may induce residual compressive stresses in the surface of the material or titanium structure. Warpage may occur in sections that are subsequently chemical milled or contour machined.

3.6 In most cases, blast cleaning is not intended to eliminate pickling procedures completely. Abrasives will not remove surface layers contaminated with interstitial elements such as carbon, oxygen, and nitrogen. When these elements are present in excessive amounts, they are preferably removed by controlled acid pickling in accordance with 4.3.

4. Pickling and Descaling

4.1 Recommended post treatment of shot or abrasive blasted titanium surfaces may include acid pickling as described in 4.3.2, to ensure complete removal of metallic iron, oxide, scale, and other surface contaminants. If the product is to be chemical milled for the removal of the oxygen-contaminated layer, salt bath conditioning may be required to avoid selectively etched surfaces if configuration interferes with uniform blasting.

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4.2 Scale and lubricant residues developed on mill, foundry, forged, or fabricated titanium products usually require conditioning by one of the following commercial methods prior to final pickling as described in 4.3.2 to produce a completely scale-free surface.

4.2.1 Proprietary solutions of caustic-based compounds in tap water in accordance with the manufacturer's recommendation.

4.2.2 Molten alkaline-based salt baths operating at 750 to 850°F (399 to 454°C) in accordance with prescribed procedures.

4.2.3 Molten alkaline-based salt baths operating at 400°F (204°C) in accordance with prescribed procedures.

4.2.4 Oxides and heat tints developed below 1100°F (593°C) can frequently be removed by pickling in an acid solution composed of 10 to 20 volume % (150 to 300 g/L) of nitric acid (70 %) and 1 to 2 volume % (12 to 24 g/L) of hydrofluoric acid (60 % at 120°F (49°C)).

4.2.5 Forged and hot-worked titanium alloys that have the normal heat-developed scale mixed with graphitic or glass-type lubricants may be conditioned in a molten alkaline-based salt bath operating at 850°F (454°C) to effect complete solubility of the complex scale. Following conditioning, the part should be pickled as described in 4.3.2.

4.2.6 It is recommended that heat-treatable alpha beta and beta alloys in the solution-treated condition that have been processed at temperatures above 1100°F (593°C), with graphitic and molybdenum disulfide lubricant residues mixed with the heat-developed scale, be conditioned in a molten alkaline-based salt bath operating at 400°F (204°C). After conditioning, the part may be pickled in accordance with the treatment described in 4.3.2.

4.2.7 Abrasive methods such as wheel or belt grinding, segmented flapper wheels, and grit or shot blasting, when available, may be used when surface configuration is such that the scaled areas are readily accessible.

NOTE 1—Conditioning of titanium in high-temperature oxidizing salt can generate galvanic currents where the work is contacting ferrous base materials. Titanium is electrically positive or anodic with respect to these ferrous materials, having an open circuit potential of approximately 0.60 V. The resulting discharge from the rack to the work piece can result in surface over-heating and eventual ignition. This effect can be minimized by maintaining bath temperature at or below 850°F (455°C) and by providing titanium fixtures or aluminum insulation between the work and the fixture.

NOTE 2—Heavily scaled forgings or hot-rolled materials can be mechanically abraded to remove excess surface contamination prior to salt bath conditioning.

NOTE 3—Silicon-based protective coatings used to minimize scale formation during hot forming or annealing of titanium alloys are soluble in the molten salt bath. All of the coating should be removed prior to any acid treatment.

4.3 Following mechanical abrading or chemical conditioning, the material may be further treated to completely clean the surface in one of the following solutions:

4.3.1 After salt bath conditioning and water rinse, the titanium or titanium alloy may be immersed in a sulfuric acid solution to remove the converted scale product. It is recommended that the acid solution be maintained at 150°F (66°C) and a concentration of 10 to 40 volume % of sulfuric acid (95 % solution by weight). Final brightening may be accomplished by a short immersion in the acid solution of 4.3.2.

4.3.2 Material that has been mechanically abraded in accordance with 3.1 or chemically conditioned in accordance with 4.2.1, 4.2.2, or 4.2.3 may be finish cleaned by immersion in an acid solution composed of 10 to 30 volume % (150 to 450 g/L) of nitric acid (70 %) and 1 to 3 volume % (12 to 36 g/L) of hydrofluoric acid (60 % at 120°F (49°C)), maintaining a ratio of 10 parts nitric acid to 1 part hydrofluoric acid.

NOTE 4—Hot-formed or heat-treated titanium fabrications or assemblies that have a mixed surface contamination consisting of graphite or molybdenum disulfide with titanium oxides should be conditioned in a molten salt bath. The 400°F (204°C) temperature is preferred for these structures to avoid any thermally induced distortion.

NOTE 5—In salt bath or alkaline water solution conditioning, the titanium oxides present on the surface of the metal are chemically reacted to form sodium titanate. This material is soluble in the sulfuric and nitric-hydrofluoric acid pickles. The sulfuric acid solutions are not appreciably corrosive to titanium or titanium alloys and can be further inhibited by the addition of 0.25 to 1.0 % of cupric sulfate or ferrous sulfate.

NOTE 6—Most of the acid pickling following molten salt bath conditioning is accomplished in the sulfuric or nitric-hydrofluoric acid solution. The material is cycled through the salt bath, water rinse, and sulfuric acid until all of the scale has been completely removed. Final brightening is obtained by a brief cycle in the nitric-hydrofluoric solution (4.3.2).

NOTE 7—In the nitric-hydrofluoric pickling solution, the ratio of nitric acid to hydrofluoric acid is more important than the concentration of either of these two acids. When this ratio is maintained at 10 to 1, hydrogen absorption during pickling is minimized.

NOTE 8—In the processing of titanium mill products and fabrications, an oxygen-rich layer is unavoidable where there is a combined exposure to high temperatures and an oxidizing atmosphere. In the removal of this oxygen-rich layer or alpha case by pickling in strong solutions of nitric and hydrofluoric acids, it is extremely important that all residual oxide and scale has been removed to prevent preferential etching of the finished product.

NOTE 9—Hydrogen in excess of the specified amount may be removed by vacuum annealing of the cleaned work piece.

5. Inspection

5.1 Visual inspection of material cleaned in accordance with this guide should show no evidence of paint, oil, grease, glass, graphite lubricant, scale, abrasive, iron, or other forms of contamination.

5.2 Hydrogen absorption during the cleaning process should be minimized and well within tolerable limits if the procedures outlined are followed. Periodic monitoring of the cleaning system can be accomplished by processing samples of known hydrogen content through the complete system followed by chemical analyses. A hydrogen increase greater than 20 ppm over the original product analyses may be cause for replacing the acids or adjusting the composition to reduce the extent of hydrogen pickup.

5.3 Additional evaluation of product cleanliness may be obtained by chemical milling of an expendable sample test piece. It is recommended that approximately 0.001 to 0.002 in. (0.025 to 0.05 mm) shall be removed from each surface. After chemical milling, the surface should be uniformly smooth and bright with the absence of peaks indicative of residual scale or contamination.

6. Keywords

6.1 cleaning; descaling; pickling; titanium; titanium alloy