Designation: B 481 – 68 (Reapproved 2003)$^{\epsilon 1}$

Standard Practice for Preparation of Titanium and Titanium Alloys for Electroplating[1]

This standard is issued under the fixed designation B 481; 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.

ϵ^1 NOTE—Section 3.1 was editorially updated in May 2003.

INTRODUCTION

Full utilization of the light weight and high strength of titanium is prevented by the tendency it has to gall and seize and by its lack of corrosion resistance at elevated temperatures. Frequently these limitations can be overcome by electrodepositing upon the titanium a metal with satisfactory properties. Titanium is an active metal that rapidly forms an adherent oxide coating in the presence of oxygen and water. This coating prevents the application of adherent electrodeposits by the more familiar preparative processes. For this reason, the special processes described in this practice were developed.

1. Scope

1.1 This practice describes processes that have been found to be successful in producing adherent electrodeposits of good quality on titanium and certain titanium alloys. Not all of the processes that have been reported as successful are described, but rather three basic ones that have had the widest use. A rather complete listing of the published work on electroplating on titanium is given in the list of references which appear at the end of this practice.

1.2 *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.* For a specific hazard statement, see 3.1.

2. Referenced Documents

2.1 *ASTM Standards:*

B 343 Practice for Preparation of Nickel for Electroplating with Nickel[2]

3. Reagents

3.1 *Purity of Reagents*—All acids and chemicals used in this practice are technical grade. Acid solutions are based upon the following assay materials (**Warning—**Use hydrofluoric acid with extreme care.):

Hydrochloric acid	37 mass %, density 1.184 g/mL
Hydrofluoric acid	60 mass %, density 1.235 g/mL
Hydrofluoric acid	71 mass %, density 1.260 g/mL
Hydrofluoric acid	100 mass %, density 1.0005 g/mL
Nitric acid	69 mass %, density 1.409 g/mL

3.2 *Purity of Water*—Use ordinary industrial or potable water for preparing solutions and rinsing.

4. Process No. 1

4.1 *Cleaning*—Remove oil, grease, and other soil by appropriate conventional processes such as vapor degreasing, alkaline cleaning, grinding, or blasting.

4.2 *Activating*—Activation may be done by chemical or electrochemical etching or liquid abrasive blasting. It is possible that all three processes will work equally well on pure titanium and all common alloys; however, only those for which each process has been demonstrated to be successful are given here. The suitability of a process for an alloy not listed should be experimentally determined before committing production parts.

4.2.1 *Chemical Etch*:

4.2.1.1 The following procedure is suitable for commercially pure titanium and for 6Al-4V, 4Al-4Mn, and 3Al-5Cr.

[1] This practice is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatingsand is the direct responsibility of Subcommittee B08.02on Pre Treatment.

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[2] *Annual Book of ASTM Standards,* Vol 02.05.

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4.2.1.2 *Pickle*—Immerse in the following solution, at room temperature, until red fumes are evolved:

HF (60 mass %)	1 volume and
HNO_3(69 mass %)	3 volumes

4.2.1.3 *Rinse*.

4.2.1.4 *Etch*—Immerse in the following aqueous solution for 20 min (Note that a special formulation is recommended for 3Al-5Cr alloy).

	Standard	3Al-5Cr
$Na_2Cr_2O_7 \cdot 2H_2O$	250 g/L	390 g/L
HF (60 % mass)	48 mL/L	25 mL/L
Temperature	82 to 100°C	82 to 100°C

NOTE 1—For platinum electroplating on commercially pure titanium, etching may be done by immersion for 5 min in hot (94°C min) concentrated hydrochloric acid followed by rinsing and platinum electroplating **(6)**[3]

4.2.2 *Rinse*.

4.2.3 *Electroplate*—Electroplate with chromium, with copper from an acid bath, or with nickel from either a Watts or sulfamate bath, or deposit nickel in an autocatalytic bath. If a deposit of some metal other than these three is desired, first apply a nickel coating with a minimum thickness of 1 µm followed by the desired final metal.

4.2.4 *Heat Treat*:

4.2.4.1 The adhesion of the electrodeposit is mechanical and, therefore, although of a relatively high order of magnitude, it may be less than adequate. If a higher degree of adhesion is desired, use nickel as an intermediate coating and heat treat. This causes interdiffusion of the nickel and titanium and produces a metallurgical bond. The heat treatment can be performed after all electroplating is applied or immediately after the nickel electroplating. This later approach is used in certain cases, for example, when undesirable diffusion can occur between the nickel and the subsequent deposit.

4.2.4.2 Heat treat in an inert gas atmosphere (for example, argon) for 1 to 4 h at 540 to 800°C. The exact time and temperature should be selected by subjecting electroplated test pieces to adhesion or performance tests or both.

4.2.4.3 If the heat treatment is performed before applying the subsequent deposit, the nickel will have to be activated before continuing the electroplating. Methods of activation are given in Practice B 343.

5. Process No. 2

5.1 *Clean*—See 4.1.

5.2 *Electrochemical Etch*:

5.2.1 The following procedure is suitable for commercially pure titanium and 4Al-4Mn alloy. The adhesion produced is purely mechanical but sufficient to pass a bend test and heating in a gas flame.

5.2.2 *Pickle*—Immerse in the following solution at room temperature until red fumes are evolved:

HF (60 mass %)	1 volume and
HNO_3(69 mass %)	3 volumes

5.2.3 *Rinse*.

5.2.4 *Etch*—Immerse in the following solution and make anodic, raising the current above the operating value until local chemical attack of the metal is stopped as evidenced by the cessation of gassing. Then reduce the current to the operating value and etch anodically at 5.4 A/dm^2 for 15 to 30 min.

HF (anhydrous)	15 mass %
H_2O	6 mass %
Ethylene glycol	79 mass %
Temperature	55 to 60°C

5.2.4.1 The formulation in 5.2.4 is equivalent to the following volumetric formulation.

HF (71 mass %)	19 volumes and
Ethylene glycol	81 volumes

5.2.4.2 The water content must not be too high; therefore, less concentrated grades of hydrofluoric acid cannot be substituted for the 71 % grade. The solution or part should be mildly agitated. The cathodes may be carbon, nickel, copper, or other materials not attacked by the solution.

5.2.4.3 Remove the part while the current is still on.

5.2.4.4 Excessive current densities will produce electropolishing and inadequate current densities will permit local chemical attack. Both conditions will result in lack of adhesion of the electroplating.

5.2.5 *Rinse*.

5.2.6 *Electroplate*—Electroplate with copper from an acid bath or copper from a cyanide bath preceded by a cyanide copper strike, with cadmium from a cyanide bath, with silver from a cyanide bath, or with nickel from a Watts bath.

6. Process No. 3

6.1 *Clean*—See 4.1.

6.2 *Liquid Abrasive Blasting*:

6.2.1 The following procedure is suitable for commercially pure titanium, 3Al-5Cr, 5Al-2Cr-2Mo, 7Al-5Cr, 2.5Al-16V, 4Al-4Mn, 2Fe-2Cr-2Mo, 28Cr-1.5Fe, 6Al-4V, and 3Al-13V-11Cr.

6.2.2 *Blasting*—Blast all surfaces with a water-abrasive slurry until a uniform appearance is achieved. The grit may be as coarse as 100 mesh or as fine as 1250. The finer grits require somewhat more time but do not cause roughening of the surface or dimensional changes. The grit should be used exclusively for blasting titanium to avoid imbedding of contaminants such as iron that can cause local failures in the coating.

6.2.3 *Electroplating*—Deposit nickel from either an electrolyte or autocatalytic bath. A minimum thickness of 1 µm is required when the nickel is used as a base for subsequent deposits.

6.2.4 *Heat Treatment*:

6.2.4.1 Heat treat in an inert gas atmosphere (for example, argon) for 1 to 4 h at 540 to 800°C. The exact time and temperature should be selected by subjecting electroplated test pieces to adhesion or performance tests or both.

6.2.4.2 The heat treatment can be performed after all electroplated coatings have been applied or immediately after the nickel electroplating. This latter approach is used in certain cases, for example, when undesirable diffusion can occur between the nickel and the subsequent deposit. If the heat treatment is performed before applying the subsequent deposit,

[3] The boldface number in parentheses refers to the list of references at the end of this standard.

the nickel will have to be activated before continuing the electroplating. Methods of activation are given in Practice B 343.

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