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PROCESS FOR PLATING TITANIUM

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FIG.1











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3,647,647 **PROCESS FOR PLATING TITANIUM** Jules P. Winfree, Jupiter, Fla., assignor to United Aircraft Corporation, East Hartford, Conn. Filed Feb. 19, 1969, Ser. No. 800,549 Int. Cl. C23b 3/02, 5/08

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6 Claims

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ABSTRACT OF THE DISCLOSURE

A process for forming a smooth adherent nickel plate on the titanium alloys utilizing a controlled half-wave anodic etch in a quiescent acid bath to achieve selective key-holing of the substrate surface, followed by cathodi-15 cally applied nickel plating.

BACKGROUND OF THE INVENTION

The present invention relates to the plating of titanium 20 and, more particularly to the electroplating of the titanium alloys.

Because of its favorable strength-to-weight ratio, titanium is widely utilized in the aircraft industry and related aerospace fields. In low temperature applications, particu- 25 larly in static structural members, it may be utilized in an uncoated condition. In the more demanding environments, however, such as exposure at high temperature to dynamic corrosive environments where its high reactivity becomes a problem, or in applications involving impact ³⁰ dynamic abrasion or rubbing contact, the titanium alloys are normally coated. While paints, and organic or elastomeric coatings may provide suitable protection in some instances, metallic coatings are nevertheless essential in 35 many cases.

Recognizing the need for smooth adherent metallic plates on titanium, many investigators have proposed plating techniques wherein the desired surface protection has been provided with a hard nickel or chromium coating. Representative techniques are described in the patents to Foisel et al. 2,946,728 and Missel et al. 2,825,682.

As recognized in the industry, the basic problem encountered in the plated titanium alloys is the provision of an adequate degree of adhesion between the coating and 45 the basis metal to permit it to bear up under severe operating conditions. One of the early approaches, utilizing the knowledge gained with other materials, involved mechanically roughening the surface of the substrate to provide a mechanical or keyed-type bond with the basis metal. 50 However, this approach was never found to be entirely adequate in each case. In general, the faults could be traced to one or more of the following conditions;

(1) Lack of reproducibility;

(2) Surface contamination or pickup in the abrasion 55process as providing a fault in the coating or incipient failure site:

(3) Insufficient adherence with a slight surface roughening;

(4) A rough surface on severely abraded substrates 60 resulting in turn with rough surfaces on the finished coating, particularly with the thinner coatings. In bearing applications, the surface roughness is intolerable and, furthermore, inasmuch as certain component characteristics, such as stress corrosion and fatigue are adversely in-65 fluenced by surface roughness, such techniques have been viewed with some disfavor. Nor can the surface roughness problem be overcome merely by the application of thicker coatings for experience has shown that a depreciation in the fatigue endurance capabilities of the titanium alloys is 70 directly proportional to the plating thickness. It is known that surface roughening may be accomplished by anodic

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etching and that many of the drawbacks of mechanical abrasion may thus be obviated. However, even with the electrolytic surface preparation techniques, the problem of surface roughness and the adverse characteristics incident thereto will remain.

SUMMARY OF THE INVENTION

It has now been discovered that a unique electroetchelectroplate process for the titanium alloys may be utilized 10 not only to provide good adherence in a keyed-type bond, but also to provide a smooth surface layer in the coating in very thin thicknesses.

In summary, the present process involves a controlled, half-wave, direct current anodic etch in a quiescent acid bath to provide "key-holing," followed by cathodic electroplating of the desired metallic coating. Usually, the coating process is in turn followed by a heat treatment for stress relief, recrystallization and/or diffusion purposes.

In the preferred process, a titanium alloy substrate is etched to provide key-holing in an etch solution comprising a mixture of acetic and hydrofluoric acids; plated in a nickel sulfamate bath to a thickness of 0.0005-0.0002 inch; and heat treated in an inert atmosphere at about 1100° F.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates the evident "key-holing" of the substrate, clearly demonstrates the smoothness of the coating at a coating thickness of 0.001-0.002 inch.

FIG. 2 is a photomicrograph similar to that of FIG. 1 after a recrystallization heat treatment.

FIG. 3 is a photomicrograph similar in view to FIGS. 1 and 2 but of a specimen wherein the plating process was interrupted at about 1/3 the thickness desired. It clearly shows the nickel plate in the leveling process unique to this invention.

DESCRIPTION OF THE PREFERRED **EMBODIMENTS**

As previously described, the preferred process involves 40 surface activation and preparation of a titanium alloy substrate with a controlled anodic, half-wave, direct current etch in an acetic acid-hydrofluoric acid solution held motionless; a low current density sulfamate nickel plate, and an 1100° F. bake in an inert atmosphere.

Components formed from the MST 811 alloy (by weight, 8 percent aluminum, 1 percent molybdenum, 1 percent vanadium, balance titanium) were immersed, after cleaning, in an etch solution consisting of 87.5 percent glacial acetic acid and 12.5 percent aqueous (48%) hydrofluoric acid held at $160^{\circ}-180^{\circ}$ F. Immersion and subsequent withdrawal were made with the current off. Etching was performed using a half-wave D.C. current density of 10 amperes per square foot for 40 minutes in a motionless solution.

Subsequent to etching, the articles were cleaned in nitric acid for about 20 seconds; rinsed; cleaned in an alkali bath; rinsed; and recleaned in nitric acid and rinsed.

Plating was done in a nickel sulfamate bath at 115°-140° F. The parts were immersed with the current on but at a reduced density of 5-10 amps. per sq. foot. Plating to the desired thickness, usually 0.0005-0.002 inch was accomplished using a current density of about 35-50 amps. per square foot.

After cleaning, the components were then baked at 250°-300° F. in air for one hour, followed by baking in argon at about 1100° F. for one hour.

The plate achieved by the above process was compared with a previously utilized method of surface treatment involving dry abrasive blasting and hydrochloric acid etching. In the prior method occluded abrasive particles and excessive cold working were noted. The nickel plate

achieved by the above process was clearly superior in terms of plate adhesion and uniformity and displayed a remarkably clean interface. The good adhesion was attributed to the key-holing effect noted particularly in the photomicrographs of FIGS. 1, 2 and 3.

FIG. 3 illustrates the leveling process associated with the electroetch-electroplate process disclosed, with the nickel plate clearly replacing the etched areas and completely filling the same at very thin thickness, resulting finally in the smooth coating of FIGS. 1 and 2.

The improved processing affected the fatigue capabilities of the coated component less than expected. It was known that the fatigue properties were directly proportional to plating thickness and testing with the prior art plating methods had indicated a substantial fatigue capability depreciation. The unexpected results in improved fatigue capabilities were attributed to both the smooth coating surface and the excellent plate adhesion.

Similar processing was also undertaken with the titanium—5 percent aluminum—5 percent vanadium—5 percent molybdenum alloy and the titanium—6 percent aluminum—4 percent vanadium alloy.

Fatigue testing was conducted with a variety of turbine engine blades plated in their contact areas to various thicknesses with nickel. Testing was performed at 900° 25 F. with short root blades tested with a simulated centrifugal load of 30,000 pounds and a 20,000 p.s.i. airfoil stress. Long root blades were tested at a 35,000 pounds root load and 25,000 p.s.i.

In addition to the corrosion-resistance benefit resultant 30 from the plating of the titanium alloys, compressor blading processed according to the present invention has been utilized to increase the frequency of resonance of such blading and thus to take it out of the critical range. This was demonstrated in actual engine testing. 35

The unique properties of the plate were achieved only with the electroetch-electroplate combination with electroetching being performed in a quiescent bath. The nonelectrolytic plating methods do not appear suitable for the thin coating as, for example, in hardware subject to 40 fatigue. Principally, the non-electrolytic plating methods appear unsuitable in these applications in that the selfleveling feature of the coating is not provided.

As may readily have been seen, the present invention provides a convenient method of attaining smooth, adherent plates on the titanium alloys in very thin thicknesses. While the invention has been described in detail with reference to certain examples and preferred embodiments, these are intended to be illustrative only. It will be understood that the invention is not limited to the exact details 50 described, for obvious modification will occur to those skilled in the art.

What is claimed is:

1. The method of providing smooth, adherent metallic nickel plating on the titanium alloys which comprises: 55 204-37 R, 49

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- anodically electroetching the clean substrate alloy surface by half-wave direct current in a quiescent acetic acid-hydrofluoric acid bath to provide key-holing thereof;
- cathodically electroplating the etched surface in a sulfamate nickel bath to a thickness of at least 0.0005 inch;
- and heat treating in inert atmosphere the plated alloy to effect plate-substrate bonding.
- 2. The method according to claim 1 wherein:
- the titanium alloy consists essentially of, by weight, about 8 percent aluminum, 1 percent molybdenum, 1 percent vanadium, balance titanium.
- 3. The method according to claim 1 wherein:
- the plating thickness is about 0.0005 to 0.002 inch.
- 4. The method according to claim 2 wherein:
- the plated alloy is heat treated at about 1100° F. in an inert atmosphere.

5. The method of providing smooth, adherent nickel 20 plating on the titanium alloys containing 4-10 weight percent aluminum which comprises:

- anodically electroetching the clean alloy surface with half-wave, direct current at a current density of about 10 amperes per square foot in a quiescent acetic acid-hydrofluoric acid bath held at a temperature of about 160°-180° F.;
- cathodically electroplating the etched alloy to a thickness of 0.0005-0.002 inch at a current density of about 35-50 amperes per square foot in a sulfamate nickel bath at a temperature of about 115-140° F.; and
- heat treating the plated alloy at about 1100° F. in argon to effect plate-substrate bonding.
- 6. The method according to claim 5 wherein:
- prior to the 100° F. heat treatment, heat treating the plated article at about 250°-300° F. for about one hour.

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