



Multi-step anodizing on Ti6Al4V components to improve tribomechanical performances

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ABSTRACT

This work presents the production and characterization of a controlled electrochemical modification of titanium surfaces, consisting of a two-step anodic spark deposition (ASD) process and a barrel finishing. The treatment was performed on titanium alloy Ti6Al4V, as most representative titanium alloy used in mechanical and aeronautical applications, where improved adhesion and tribological performances are required. FIB/SEM microstructural observations, nanoindentation, scratch, fretting tests and wear and friction tests were carried out both on laboratory specimens (planar pellets, 1 in. diameter) and on Ti6Al4V bolts of different shapes and sizes, to investigate the effect of a three dimensional shape on the correct execution of the electrochemical treatment and on the coating's mechanical performance. The formation of a double layered coating was observed, with a slightly decreased surface hardness and stiffness compared to the metallic substrate and increased scratch and fretting wear resistances, thanks to an excellent adherence to the substrate, an optimal value of the hardness to modulus ratio (H/E) and a marked decrease of the friction coefficient. The proposed coating procedure could be therefore a suitable solution for those applications where low friction, wear resistance under low load sliding contact and corrosion resistance are required.

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1. Introduction

Titanium and its alloys combine excellent mechanical resistance, good corrosion resistance and good maximum working temperature with affordable costs, specially taking into account the whole life cycle of the component and the consequent reduction of maintenance costs [1–4].

In contrast, titanium alloys present a major drawback, that is, their tribological behaviour, as they are characterized by low hardness, poor resistance to wear, high friction coefficient (either against a metal or ceramic counterpart) and limited load bearing capacity, which are ascribed to a low resistance to plastic shearing and limited work hardening behaviour [5,6].

In fact, titanium alloys present relatively low values of the H/E and H^3/E^2 ratios in comparison to steel, being H the hardness and E the elastic modulus. In particular, the H/E ratio (which can represent the elastic strain to failure) is a suitable parameter to predict resistance to sliding and/or abrasive wear, while the H^3/E^2 ratio is related to the contact yield pressure under normal load and can be a good estimation of the resistance to plastic deformation and load bearing capacity.

The increase of these parameters and the reduction of friction coefficient are therefore important issues for titanium alloys, e.g. by the implementation of a proper coating procedure.

This target is usually approached by the use of mechanical treatments [7,8] and coatings, where the optimal combination between substrate/coating properties (including H/E and H^3/E^2 ratios), which can be also achieved by the use of graded systems, is an issue of interest that is currently not completely solved.

As for coatings, the most commonly applied techniques are thermal and thermochemical diffusion [9–11], plasma nitriding, oxynitriding or carburizing [12–15], anodizing [16–21], chemical oxidation [22,23], laser or ion-beam surface alloying [24–27], thermal spraying [28–31] and Physical Vapour Deposition (PVD) [32–36]. Among these, the most frequently applied is nitriding, which increases the surface hardness and improves the resistance to wear without affecting the corrosion behaviour. Yet, nitride layers require a controlled atmosphere, and often induce a decrease in titanium alloy fatigue resistance due to premature fatigue cracks in the nitride layer [11,35,36].

Recently, the use of Duplex or multilayer-graded coatings has been proposed to induce a smooth transition of the mechanical properties from the compliant-ductile substrate up to the stiff-hard top layer. In this sense, an effective way is represented by a High Velocity Oxygen Fuel (HVOF) thermally sprayed WC–Co thick interlayer and a Cathodic Arc Evaporation (CAE) PVD thin top layer [32–34]. This system gives optimal configuration in terms of enhanced load bearing

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capacity and coating adhesion on Ti6Al4V substrates; yet, it is relatively expensive compared with other conventional coating techniques [32], and so could be effectively used only in a limited variety of industrial applications, e.g. high performance automotive engineering [34].

Previous works also report the efficiency of some anodic oxidation treatments in increasing the resistance of Ti6Al4V to corrosion in general, and specifically to fretting-fatigue phenomena [20,37,38]. The treatments which find most relevant applications are high voltage anodizing processes (Anodic Spark Deposition, ASD), which induce the formation of a thick ceramic coating with a rough, crater-like surface and chemical composition modified by the incorporation of electrolyte ions. This technique is highly productive, economic and ecological compared to other processes used to increase titanium alloy's surface hardness, since it does not require vacuum conditions or inert atmospheres. The films produced exhibit excellent bonding strength with the substrate; improvements are needed to increase hardness and wear resistance [39–42].

Despite the large amount of experimental works in this field, the achievement of the optimal compromise between an increased load bearing capacity and a reduction of friction coefficient is a complex problem not yet solved for titanium alloys and related coatings, as it involves chemo-mechanical surface interactions between the counterparts and the formation of complex tribo-layers evolving during wear. Several questions on the microstructure-properties-performances correlations for these coated systems are still unsolved: in particular, the influence of microstructure (grain size, porosity, defect distribution), surface hardness and coating's adhesion.

In addition, a study on the deposition efficiency, microstructural changes and related effect on the wear resistance of coatings on three-dimensional complex-shaped substrates has not been performed yet. This latter aspect appears to be the most critical issue in view of the technological transfer to industry of newly developed coating techniques for titanium alloys.

In view of this, the present work was aimed at investigating an ASD treatment involving a double anodizing step in alkaline electrolytes, which was described in a previous work [43], as it is considered particularly promising to induce remarkable improvements in fatigue and wear behaviour of titanium alloys.

Coatings were produced both on planar substrates and on 3D complex shaped specimens, consisting of bolts currently used as fasteners in aeronautical engineering, where corrosion and low load wear resistance are major requirements that need to be fulfilled both during the

period of storage of the components and during service. Characterization activities consisted of high resolution FIB/SEM microstructural observations, nanoindentation testing, micro-scratch testing and fretting wear testing.

2. Experimental details

2.1. Specimens anodizing

The surface of titanium alloy, Ti6Al4V, specimens was treated by applying the anodizing procedure formerly developed [43]. First, the specimen was anodized in a calcium glycerophosphate solution with a growth rate of 1 V/s, up to a cell voltage of 350 V. The specimen was then rinsed in fresh water and anodized in a sodium silicate alkaline solution with a growth rate of 1 V/s, up to a cell voltage of 350 V. All anodizing electrolytes were kept at a temperature of 5 °C. Finally, the specimen was subjected to a mechanical treatment of barrel finishing.

The treatment was performed both on laboratory specimens (planar pellets, 1 in. diameter) and on Ti6Al4V bolts for mechanical and aeronautical applications of different shapes and sizes (Fig. 1), with the main aim of investigating the effect of a three dimensional complex shape on the mechanical performance of the coatings, where coating's homogeneity (in terms of thickness and microstructure) is usually difficult to be achieved by conventional line-of-sight PVD techniques, especially in correspondence of the bolt threads, where the maximum contact stresses during tightening are expected.

2.2. Specimens preparation

The analytical techniques adopted to investigate the coating properties were X-ray diffraction (XRD), nanoindentation testing [44,45], adhesion and wear tests, as well as FIB/SEM microstructural analyses.

Planar pellets were tested after the surface treatment without further preparation steps, while bolts were prepared in order to perform mechanical testing: thin slides were obtained both on the lateral side of the stem and on the threads by metallographic cutting. Mechanical testing (indentation and scratch) was performed on the slice obtained from the stem, while the slices with threads on them were used for FIB/SEM microstructural analyses. XRD spectra were collected on planar specimens by using a Philips PW3020-Cu K α radiation instrument. Particular attention was conveyed to the angular range $2\theta = 20^\circ \div 30^\circ$,

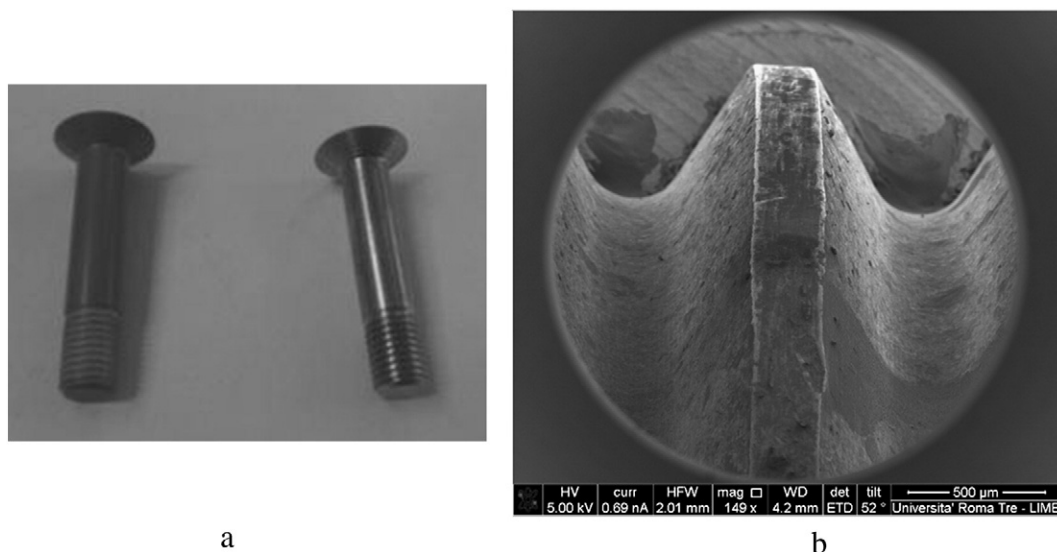


Fig. 1. Photograph of one anodized bolt (left) and the original bolt (right); SEM micrograph of one thread of the anodized bolt.

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