Ion-plasma modification of surface of ultrafine-grained titanium alloys: effect of substrate on coating properties

*R R Valiev 1,2, Iu M Modina 1, Yu M Dyblenko 1, I P Semenova 1

1Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, 450008, Russia
2Saint Petersburg State University, St. Petersburg State University, 7/9 Universitetskaya nab., St. Petersburg, 199034 Russia

E-mail: rovaliev@gmail.com

Abstract. This article is focused on research of properties of vacuum-plasma coatings Ti+TiVN and V+TiVN applied to the surface of titanium alloys Ti-6Al-4V and VT8M-1(rus.) (Ti-5.7Al-3.8Mo-1.2Zr-1.3Sn) in ultrafine-grained (UFG) states obtained by two different severe plastic deformation (SPD) techniques - equal-channel angular pressing (ECAP) and rotary swaging (RS). The influence of substrate UFG structure on coatings service properties improvement has been established. It was demonstrated that complex hardening of titanium alloy including formation of bulk UFG structure and applying vacuum-plasma protective coating to the surface significantly increases resistance to erosive wear of the latter in comparison with the substrate having the initial coarse-grained structure.

1 Introduction

Two-phase titanium alloys have a vast field of application as constructional materials in aviation as well as engine manufacturing due to the most favorable ratio of strength, ductility, crack resistance, corrosion resistance and other characteristics that provide high weight efficiency, service life and reliability of gas turbine engine (GTE) products. Titanium alloy VT8M-1 (Ti–5.7Al–3.8 Mo–1.2Zr–1.3Sn) exhibits better properties in terms of heat resistance and thermal stability than popular Ti-6Al-4V alloy [1,2]. However, products and components of gas turbine engines may carry high cyclic, dynamic loads, or experience erosion and corrosion during operation and thus, the requirements to their structural strength tend to become more rigorous.

Processing by severe plastic deformation (SPD) has proved itself as one of the most efficient ways to considerably increase the mechanical and fatigue characteristics of metals and alloys by forming in such materials ultrafine-grained (UFG) structure with the grain size in submicron and nanocrystalline range with mostly high-angle grain boundaries (HAGB) [3-5]. At the same time a proper surface treatment can provide different properties, such as resistance to wear, erosion and corrosion and another way to protect materials in this paper deals with ion surface improvement by the deposition of vacuum-plasma
coating [6-8]. Therefore, combining the formation of UFG structure in the material and vacuum-plasma coatings on the surface can serve the purpose of complex strengthening of GTE parts.

2 Material and experimental procedure

The studies were conducted on titanium alloys Ti-6Al-4V and VT8M-1 (Ti-5.7Al-3.8 Mo-1.2Zr-1.3Sn) that are classified as hard-to-deform materials. Therefore, the rods of these alloys were quenched in water at 940 °C and annealed for 4 hours at 700 °C with cooling in air as a pretreatment for the formation of a duplex structure consisting of primary α-phase not exceeding 25% and a thin plate α+β structure, thus providing the conditions of plastic deformation [9].

Ultrafine-grained state was achieved by two techniques. The 30mm diameter Ti-6Al-4V alloy rods were subjected to equal channel angular pressing (ECAP) with 4 passes, route Bc at 750 °C (ε=2.7 with a strain rate of 4 mm s⁻¹), channel intersection angle ψ = 120°. The VT8M-1 alloy rods with 70mm diameter and 1000 mm in length were processed by rotary swaging with intermediate annealing at 750°C till the diameter was reduced to 60, 50, 40, 30 mm (ε~1.7, the strain rate exceeding 300 mm s⁻¹). These procedures are described in more detail in [10,11].

For high surface quality, Ti alloys with coarse-grained (CG) initial and UFG structures were subjected to mechanical grinding and further electrolyte-plasma polishing (EPP) [12,13] before coating deposition. EPP of the samples was conducted in a 5% electrolyte solution (KF and NH4F) at 70...90°C by pulsed electric discharges at U=320-350V and I=0.2...0.5 A/dm² for 5 minutes [13], which interacted with the tops of microroughnesses and removed them. As result, the samples acquired a characteristic mirror-like metallic luster. Further, the CG and UFG samples of Ti alloys were sprayed with TiVN coating with two versions of sublayer on the Ferry-WATT machine, where in the first case the substrate was treated with Ti, and in the second one – with V, the deposition time of sublayers and functional coating layers was 8 and 30 minutes respectively. Please, refer to [14,15] for more details of the coating deposition technique. The coating was certified by measuring the thickness of the formed sublayers/coating layers on the Calotest machine and the microhardness on the Struers Duramin machine at the applied load of 300g for 15s. Adhesion strength of the coating was tested by scratching at a continuously increasing load of 0.03 to 10 N/min at an indentation rate of 2 mm/min on a CSM Microscratch testing machine. Erosive wear testing was performed on the basis of the comparative test method according to ASTM G76-18 “Standard Test Method for Conducting Erosion Tests by Solid Particle Impingement Using Gas Jets”. This standard cover metal materials and coatings and establishes a method for their testing for abrasive wear in a solid particle stream.

3 Results and discussion

3.1 Microstructure of SPD-processed alloys Ti-6Al-4V and VT8M-1

Two-phase titanium alloys after heat treatment had duplex structure, the average size of primary α-phase was ~2.7 μm and its volume fraction equaled to 25%, and included the interlayers of the plate-like (α+β) structure (Figure 1a).
Figure 1. Microstructure of Ti-alloy (a) after heat treatment; and after subsequent SPD-processing: (b) Ti-6Al-4V by ECAP; (c) VT8M-1 by RS.

SEM and TEM analysis (Figure 1b, c) demonstrated the complete transforming of plate-like structure into a globular one after processing by ECAP and RS, with the grain and subgrain size (α+β) of 0.48 and 0.25 µm respectively. The size of the primary α-phase equaled to ~3 µm and was close to equiaxial, but some grains after RS processing were elongated in the direction of straining with the length up to 7 µm. The features of formation of these UFG structures are discussed in more detail in [11].

3.2 Architecture and properties of vacuum-plasma protective coatings Ti+TiVN and V+TiVN

Figure 2. SEM image of UFG substrate + deposited coating: (a) Ti-6Al-4V + Ti+TiVN; (b) VT8M-1 + V+TiVN.
Peculiarities of the formation of these coatings were earlier investigated in our works [14,15]. The same coating structure was deposited on the samples of these alloys in coarse grained states. Table 1 presents the average values of the microhardness for CG and UFG Ti alloys and coatings deposited on these substrates. As is seen, the coating hardness on the UFG alloy is higher than on the CG one, which speaks to the fact of the substrate hardness producing influence on that of the coating [16].

**Table 1.** Properties of vacuum-plasma coatings Ti+TiVN and V+TiVN on CG and UFG substrates of titanium alloys

|                  | CG Ti-6Al-4V + Ti+TiVN | UFG Ti-6Al-4V + Ti+TiVN | CG VT8M-1 + V+TiVN | UFG VT8M-1 + V+TiVN |
|------------------|------------------------|-------------------------|--------------------|---------------------|
| Coating thickness, h, µm | 5.4                    | 5.5                     | 6.0                | 6.2                 |
| Coating hardness, MPa   | 10000±500              | 12000±500               | 16500±1000         | 26300±1000          |
| Substrate hardness, MPa | 3200±100               | 4050±100                | 2860±150           | 3350±160            |
| Critical load during cohesive fracture of the coating, Lc1, N | 3.9±0.12               | 4.5±0.10                | 5.0±0.15           | 5.1±0.10            |
| Critical load during adhesive fracture of the coating, Lc2, N | 6.9±0.20               | 14.2±0.30               | 7.6±0.2            | 18.1±0.15           |

As is seen in this work (Table 1), the adhesive strength and physico-mechanical properties of the applied coatings are considerably influenced by the substrate grain refinement. The experimental data obtained during the adhesive strength tests by scratching showed that the formation of UFG structure by grain refinement leads to an increase of the critical load to fracture (Lc2) by 2 and 2.5 times for coatings with sublayer of Ti and V respectively as well as to the enhancement of the critical load during adhesive fracture of the coating (Lc1). The effect of UFG structure on the hardness and adhesion strength of the coating is clearly of a general nature. This effect may be related to an increase in the crystallization rate of the coating material and its vacuum-plasma deposition on the UFG substrate, which leads to a decrease in the grain/crystal size in the coating [15]. The use of a V sublayer while vacuum-plasma protective coating TiVN deposition leads to increased erosion resistance due to the enhanced viscosity of vanadium [17]. Figure 3 demonstrates the results within 11 cycles of tests for erosive wear of vacuum-plasma protective coating V+TiVN on titanium alloy VT8M-1 in CG and UFG states as well as uncoated samples. The obtained data prove a sample from ultrafine-grained titanium alloy VT8M-1 with vacuum-plasma coating V+TiVN deposited on its surface to be the most resistant to abrasive wear.
Figure 3. Dependence of mass loss of titanium alloy VT8M-1 samples in CG and UFG state with V+TiVN protective coating on erosive wear test cycle.

The results of erosion tests testify to the deposition of vacuum-plasma protective coating V+TiVN on titanium alloy VT8M-1 in CG and UFG states significantly reduces mass loss of the sample (Table 2) and twice increases erosion resistance of the material as compared to uncoated CG alloy. A substrate with a UFG structure is even more efficient for achieving this phenomenon, which is evident by the increased adhesion strength of the coating (Table 1).

Table 2. Erosive wear testing results of titanium alloy VT8M-1 in CG and UFG states with vacuum-plasma coating V+TiVN

|                | CG VT8M-1 | UFG VT8M-1 | CG VT8M-1 + V+TiVN | UFG VT8M-1 + V+TiVN |
|----------------|-----------|------------|-------------------|-------------------|
| Total average mass loss, \( \Sigma \Delta m_{\text{average}} \), g\( \times 10^{-6} \) | 8585      | 8444       | 5931              | 4286              |
| Comparative erosion resistance, K | 1.0       | 1.02       | 1.45              | 2.0               |

Thus, the data on high hardness and enhanced adhesive and abrasive properties of the coating on titanium alloys in UFG state by means of severe plastic deformation processing techniques provide evidence that these coatings with sublayer V and Ti are quite effective and promising for application as constructional materials in aircraft engine building that involve extreme environment and in particular must perform under the harsh conditions of abrasive treatments.

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