This is an pre-print of paper published by Elsevier in Tribology International, 82 PartB (2014) p.400-4006,

http://dx.doi.org/10.1016/j.triboint.2014.05.030

Wear resistant multilayer nanocomposite WC1-x/C coating on Ti-6Al-4V titanium alloy

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Abstract

A significant improvement of tribological properties on Ti-6Al-4V has been achieved by developed in this study multilayer treatment method for the titanium alloys. This treatment consists of an intermediate 2 μm thick TiC_xN_y layer which has been deposited by the reactive arc evaporation onto a diffusion hardened material with interstitial O or N atoms by glow discharge plasma in the atmosphere of $Ar+O_2$ or $Ar+N_2$. Subsequently, an external 0.3 μm thin nanocomposite carbon-based WC_{1-x}/C coating has been deposited by a reactive magnetron sputtering of graphite and tungsten targets. The morphology, microstructure, chemical and phase compositions of the substrate material after treatment and coating deposition have been investigated with use of AFM, SEM, EDX, XRD, 3D profilometry and followed by tribological investigation of wear and friction analysis. An increase of hardness in the diffusion treated near-surface zone of the Ti-6Al-4V substrate has been achieved. In addition, a good adhesion between the intermediate gradient TiC_xN_y coating and the Ti-6Al-4V substrate as well as with the external nanocomposite coating has been obtained. Significant increase in wear resistance of up to 94% when compared to uncoated Ti-6Al-4V was reported. The proposed multilayer system deposited on the Ti-6Al-4V substrate is a promising method to significantly increase wear resistance of titanium alloys.

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Keywords: Nanocomposite coating WC_{1-x}/C, Titanium alloy Ti-6Al-4V, Low wear coating, Diffusion hardening.

1 Introduction

Titanium alloys due to its low weight, high strength and good biocompatibility are widely used in aerospace, military, chemical and medical industries [1, 2]. However its poor wear resistance and low tribo-corrosion performance has severely limited their use in engineering applications where friction is involved [3, 4]. High coefficient of friction [5] and tendency to seizure and catastrophic wear drastically decrease the reliability of titanium based components. To alleviate the problem several coating systems has been proposed in recent years based on the following treatment and deposition processes: Physical Vapour Deposition (PVD) [6, 7], Chemical Vapour Deposition (CVD) [8], thermal oxidation [9], ion implantation [10, 11] and more recently Micro Arc Oxidation (MAO) [12]. In most cases another problem can arise that due to hard coating deposited on soft substrate will create so called ‘eggshell effect’ and such coating will have tendency to collapse under the load [13]. Therefore, an intermediate layer or hardened surface is often required to increase the tribological performance of coating system [14]. Attempts to overcome these disadvantages by the surface and near-surface zone hardening with use of interstitial C, N, or O atoms don't improve its tribological properties [15]. Different treatment methods can be used to increase tribological performance [16]. Tribological properties of hard coating deposited on soft substrates has been widely investigated. Ramalho et al. [17, 18] and Zaidi et al. [19] reported the results of tribological investigations of coatings on steel, whereas Schmitt and Paulmier [20] on aluminium alloys.

In this paper new multilayer coating system WC_{1-x}/C deposited by the reactive magnetron sputtering on a diffusion hardened Ti-6Al-4V alloy is developed and tribological analysis of friction and wear response of such tribosystem under severe fretting loading conditions are presented.

2 Experimental procedure

Experimental part of the study consists of two stages. The first one is coating deposition and the second is tribological analysis. New nanocomposite carbon-based

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WC_{1-x}/C coating has been deposited on a monocrystalline (100) 7N Si(p) wafer and on a diffusion hardened Ti-6Al-4V substrate coated with an intermediate TiC_xN_y gradient layer. The basic properties of the nanocomposite coating have been measured for the coating deposited on the Si substrate. Second stage of the study is devoted to tribological investigation (**Fig. 1**) carried out on nanocomposite coating deposited on the diffusion hardened Ti-6Al-4V titanium alloy.

2.1 Studied materials and coating deposition

Diffusion hardening process of the Ti-6Al-4V substrate with interstitial O or N atoms has been carried out with use of the glow discharge plasma in $Ar+O_2$ and $Ar+N_2$ atmosphere. Detailed description of this standard process can be found in a previous paper [21]. Increase of the surface micro-hardness, after diffusion hardening process, has been achieved from initial value of 350 $HV_{0.2}$ to 1000 $HV_{0.2}$. The intermediate 2 μm thick TiC_xN_y coating has been deposited onto the hardened Ti-6Al-4V substrate by the reactive arc evaporation from a pure Ti cathode in the reactor described in [21]. An external nanocomposite carbon-based 0.3 μm thick WC_{1-x}/C layer has been deposited by a reactive magnetron sputtering of graphite and tungsten targets in a vacuum chamber equipped with four magnetrons with circular targets (with diameter of 100 mm). Architecture of designed coating is presented in Fig. 1**.**

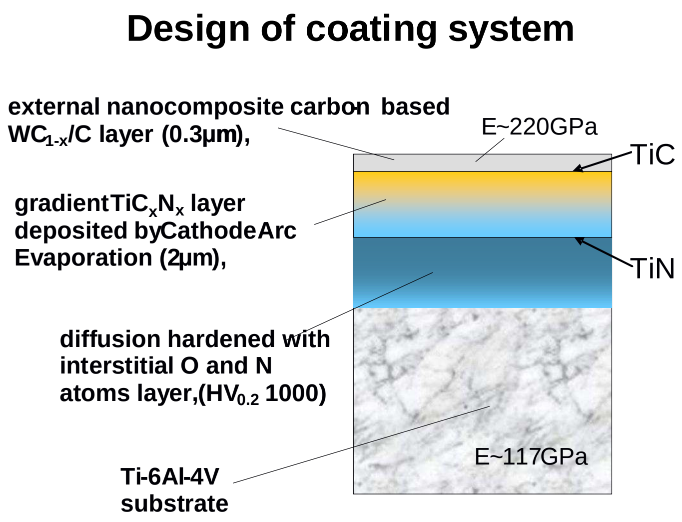

Fig. 1: Schematic diagram of developed multilayer coating system for titanium alloy.

The Ti-6Al-4V substrate has been placed in centre of a rotating table. In order to enhance ionization and increase reactivity of the plasma excited in the pure Ar, an additional electrode powered by 27.12 MHz RF generator and 0.6 kW linear amplifier has been mounted in the centre of the base plate of the vacuum chamber. The

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microstructure of nanocomposite WC_{1-x}/C coating deposited on the Ti-6Al-4V is presented in [22]. More detailed description of the device and process parameters can be found in [23]. As mentioned above, two different materials have been used as the substrates: pure monocrystalline (100) 7N Si(p) wafer, and a rectangular prism (6x18x18 mm) of Ti-6Al-4V titanium alloy.

Table 1: Parameters of reactive magnetron sputtering deposition process.

Process parameters	Substrate material	
	monocrystalline (100) 7N Si(p) wafer	Diffusion hardened Ti-6Al-4V with 2 µm TiC_xN_y intermediate gradient layer
Time of Deposition (ks)	3.8	3.9
Bias - Potential (V)	-50	-50
Bias - Current (A)	0.05	0.04
Ar flow (mm^3s^{-1})	470	420
Pressure (Pa)	0.03	0.025
Total power of 3 magnetrons with C targets (kW)	3.35	2.83
Power of magnetron with W target (kW)	1.15	0.8

2.2 Tribological analysis of friction and wear

Analysis of friction and wear behaviour for all: untreated, hardened and coated tribosystems have been performed on Ti-6Al-4V alloy substrates. The reciprocating tribotester with small amplitude sinusoidal displacements has been used. Tribological tests have been carried out in a configuration sphere/plane, where diameter of the sphere was 12.7 mm. In order to reduce wear of the sphere counterbody, an AISI 52100 sphere was used. The normal load in the contact has been kept constant during the tests and applied normal force was P=20 N. Displacement in the contact have been imposed by an electro-dynamic shaker. Four different amplitude levels of sinusoidal displacement have been used (25, 50, 75 and 100 µm) and therefore energy wear coefficient (α) of the system can be calculated [24]. Advantage of the energy wear approach is that it is quasi-independent of the used contact geometry and therefore more universal when used to predict or compare the wear kinetic on different systems. The test frequency was kept constant at F=10Hz. Test duration has been set to last for 10000 cycles. The schematic diagram of experimental device is presented in Fig. 2. During the tests, values of tangential force Q, normal force P and relative displacement δ have been recorded. All specimens have been ultrasonically cleaned in acetone and ethanol before the tests. Experiments were performed in laboratory conditions at constant temperature of 23+/-1ºC and relative humidity 43+/-5%.

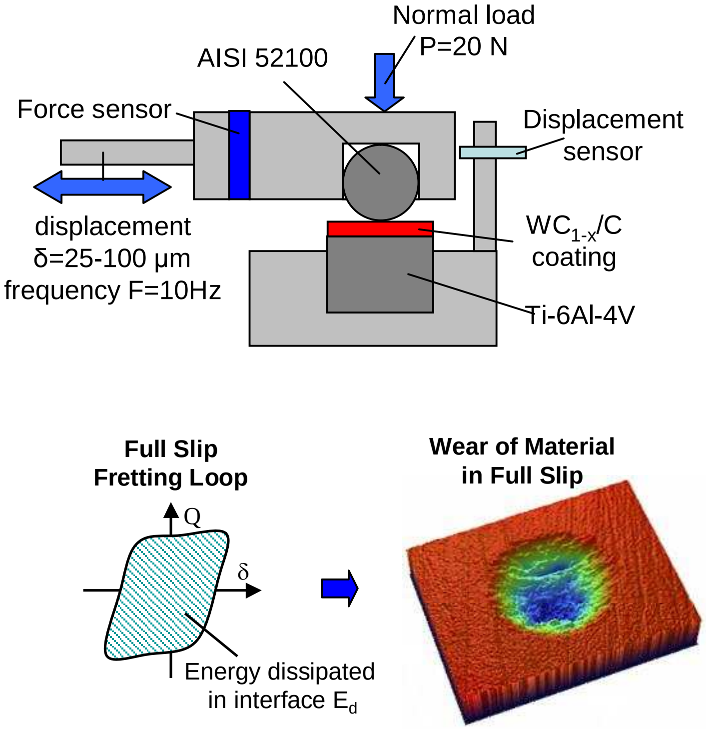

Fig. 2: Schematic diagram of experimental device for sphere/plane configuration, illustration of measured parameters and wear damage observed in gross slip regime [25].

Total amount of dissipated energy (E_d) during the test can be calculated by integration of fretting loops area over total number of cycles: $\sum_{1}^{N} E_d(Q,\delta)$. The wear volume of sphere (V_s) and plane (V_p) specimens have been measured by a 3D optical interferometer Veeco NT3300. To analyze friction behaviour, coefficient of friction calculated as ratio of tangential force amplitude and normal force ($\mu=Q^*/P$) can be analysed and average value of coefficient of friction for a given test can be calculated as: $\mu_a = \sum_{1}^{N} \frac{1}{N} \cdot \frac{Q^*}{P}$, where N is the total number of cycles. More detailed description of friction analysis can be found in previous publication [26].

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3 Results and discussion

Microstructure and mechanical properties of new nanocomposite carbon based WC_{1-x}/C coating have been analysed for coating deposited on a silicon substrate used as a reference material and tribological behaviour has been tested on the coating deposited on a diffusion hardened Ti-6Al-4V substrate.

3.1 Coating composition

An example of SEM morphology of cross-section fracture of WC_{1-x}/C coating deposited on the silicon substrate is presented in Fig. 3a. The thickness of coating is homogenous and is in the order of 0.3 µm. The AFM topography is presented in Fig. 3b, measured average roughness was R_a=1.35 nm. AFM analysis confirms that obtained surface is uniform and isotropic. For the coating system deposited onto Ti-6Al-4V substrate polished with 2500 grit abrasive sandpaper, the average roughness measured by 3D interferometer was R_a=95.6 nm. The characteristic "orange skin" surface can be observed only on SEM image presented in Fig. 10. This effect can be caused by an initial surface roughness (R_a=86.4 nm) and microstructure of the Ti-6Al-4V substrate.

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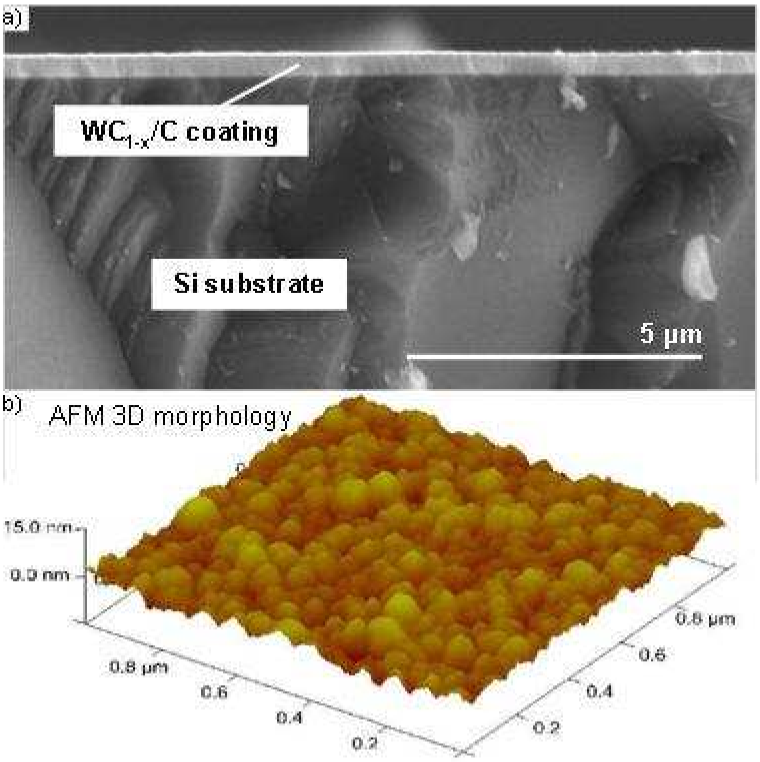

Fig. 3: Characterization of the new WC_{1-x}/C coating deposited by reactive magnetron sputtering on the silicone substrate, a) SEM image of coating fracture and b) AFM morphology of coating surface [23].

A standard XRD phase analysis of the coating has been carried out and the spectrum of the diffracted beams is plotted in Fig. 4. Chemical compositions of the coatings measured by EDX technique is 53 (at.%) of C and 47 (at.%) of W.

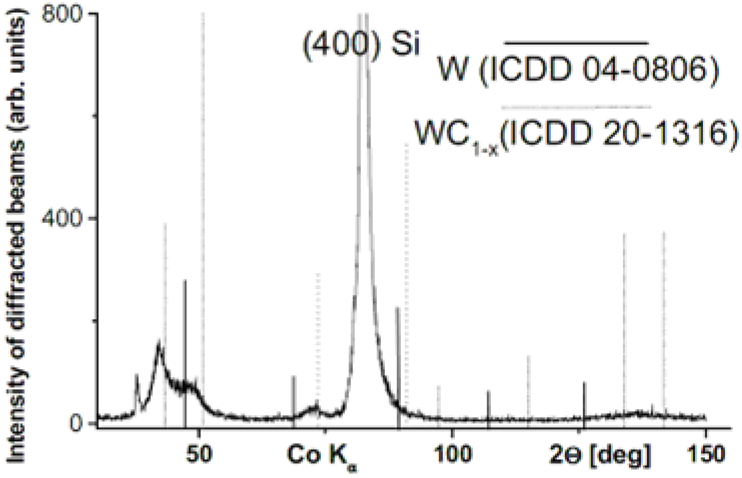

Fig. 4: Results of X-ray phase analysis of WC_{1-x}/C coating deposited on the silicone substrate, nanocrystalites of WC_{1-x} phase in an amorphous carbonaceous a-C matrix [23].

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3.2 Friction and wear resistance analysis

The friction and wear analysis have been performed for classical sphere/plane contact configuration at small displacement amplitudes (25 - 100 µm). The reciprocating tribotester have been used to carry out tribological experiments. Examples of results for coefficient of friction (COF) evolution during the test are presented in Fig. 5. The highest value can be noted for untreated substrate, slightly lower and very similar evolution can be observed for both the hardened and the coated tribosystems. Proposed multilayer system does not decrease the coefficient of friction significantly, which can be very suitable for many practical applications where the tangential forces or shear stress have to be transmitted through the contact (i.e. screw joints, rotor blades …). An average value of coefficient of friction μ_a for all tested conditions is summarized in Table 2.

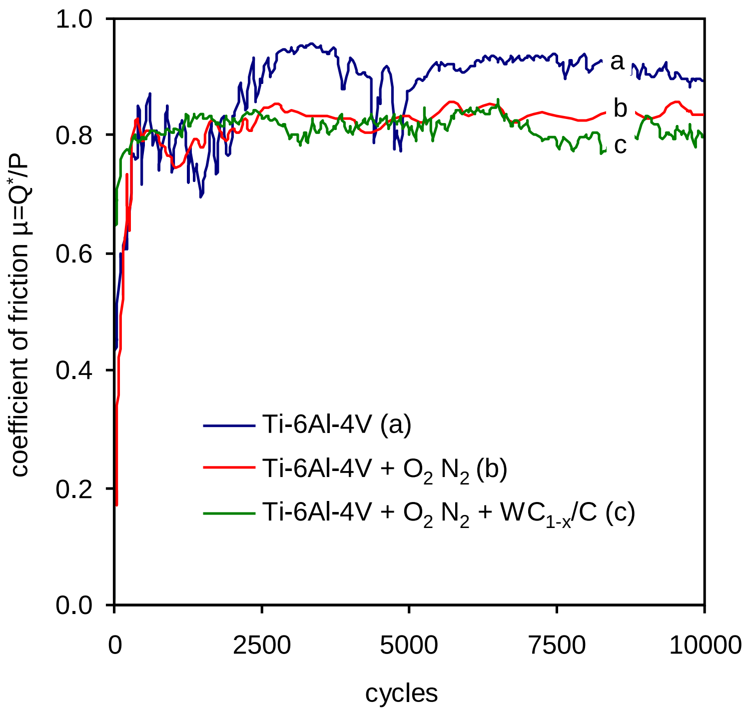

Fig. 5: Analysis of friction evolution for untreated (Ti-6Al-4V), hardened (Ti-6Al-4V + O_2 N_2) and coated (Ti-6Al-4V + O_2 N_2 + WC_{1-x}/C) in sphere/plan contact configuration, displacement amplitude δ^*=50 µm.

In the tribosystems with high value of coefficient of friction, also high wear rate can be expected. Contact between Ti-6Al-4V and 52100 is characterized by high value of coefficient of friction similar to Ti-6Al-4V // Ti-6Al-4V contact with value around ~0.9 [27]. Wear analysis, also was conducted for untreated, hardened and coated contact configurations. In first case, for untreated Ti-6Al-4V // 52100 contact (Fig. 6), severe wear rate and exponential relationship between cumulated dissipated energy ΣE_d and wear volume of the plane V_p can be observed. These results confirm the investigation and results reported by Fridrici [28], where similar relation in Ti-6Al-4V // Ti-6Al-4V contact has been reported.

For uncoated tribosystem the wear observed on a sphere counterbody is decreasing for higher value of dissipated energy E_d (higher displacement amplitude δ^*). This effect is related to high adhesion of titanium alloy and material transfer phenomenon from the plane to the sphere specimen. Therefore, adhesive wear mechanism for uncoated system can be confirmed. This hypothesis can be supported by EDX analysis performed on the sphere (Fig. 10, Sphere T15). Titanium (Ti) particles has been detected in the fretting scar on the sphere sample, however on plane specimen (Plane T15) only traces of the iron (Fe) particles has been found. This confirms that transfer of material for an uncoated tribosystem has been observed from plane Ti-6Al-4V to the sphere 52100 material. This phenomenon leads to a very high wear rate of Ti-6Al-4V substrate.

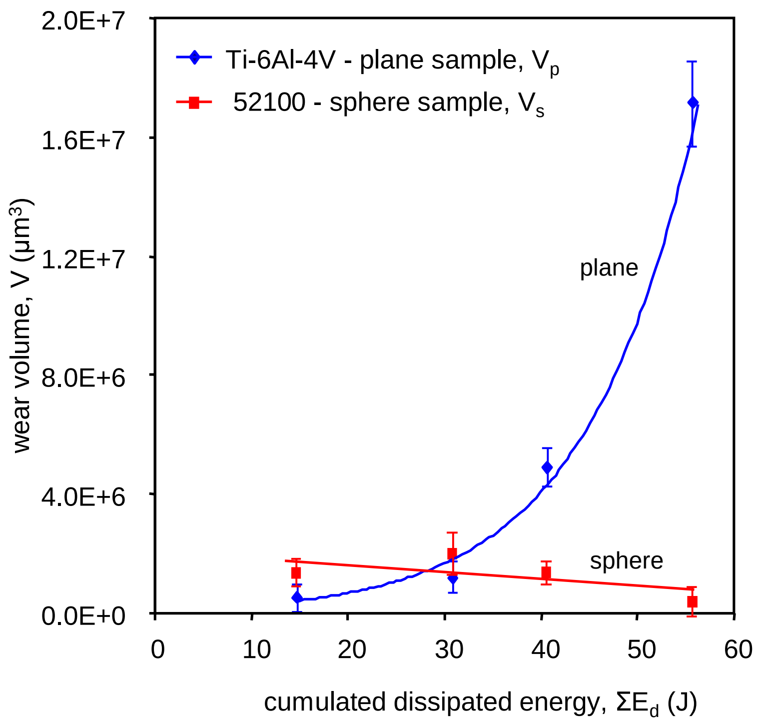

Fig. 6: Evolution of wear volume (V) as a function of cumulated energy dissipated in contact during the test ΣE_d, for uncoated Ti-6Al-4V // 52100 tribosystem.

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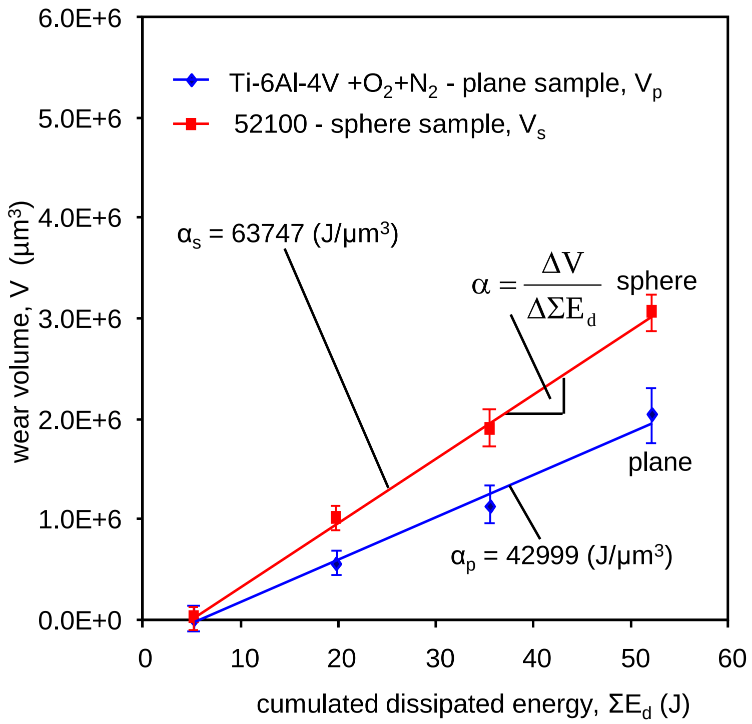

Fig. 7: Linear evolution of wear volume (V) as a function of cumulated energy dissipated in contact during the test ΣE_d, for the diffusion hardened Ti-6Al-4V alloy against 52100 sphere.

The results of wear analysis for diffusion hardened titanium alloy (Ti-6Al-4V + O_2 N_2) is presented in Fig. 7. In this case, linear evolution of wear volume can be observed for both, plane and sphere specimens. The strengthened material presents much better wear resistance, while keeping the coefficient of friction at similar level. Wear mechanism change from adhesive to abrasive, some layer of Fe particles was detected on titanium sample, but there is no transfer of titanium material onto the sphere specimen. For this linear evolution a wear rate coefficient based on energy description can be calculated for the plane α_p=42999 J/μm^3 and for the sphere counterbody α_s=63747 J/μm^3. One can note that, evolution of wear for the plane specimen does not intersect the origin of the graph, this phenomenon is related to the energy required to activate the wear degradation process. This phenomenon is relatively common in this kind of tribological test and can be observed for different materials [25] and for different surface treatments [29]. It is usually associated with the tribologicaly transformed microstructure of material layer, but related physics is still not fully understood and is yet to be unveil.

The third case is hardened titanium alloy with the transitional gradient coating (TiC_xN_y) and an external nanocomposite WC_{1-x}/C coating deposited by reactive magnetron sputtering of graphite and tungsten targets. The results of wear analysis

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are presented in Fig. 8. Significant improvement of more than an order of magnitude in wear rate value, have been achieved for the plane material with energy wear coefficient α_p=2393 J/µm^3. Also further reduction in wear rate of sphere counterbody have been observed with α_s=36266 J/µm^3. This reduction could be attributed to thick layer of Fe atoms adhered on plane specimen (Fig. 10 Plane TW12) which slows down the abrasive wear kinetics. In this case, there is no transfer of Ti particles on the sphere specimen (Fig. 10 Sphere TW12). Wear mode in this case also changed from adhesive to abrasive due to higher hardness (1000 $HV_{0.2}$) of titanium material after hardening treatment and better resistance to shear stress.

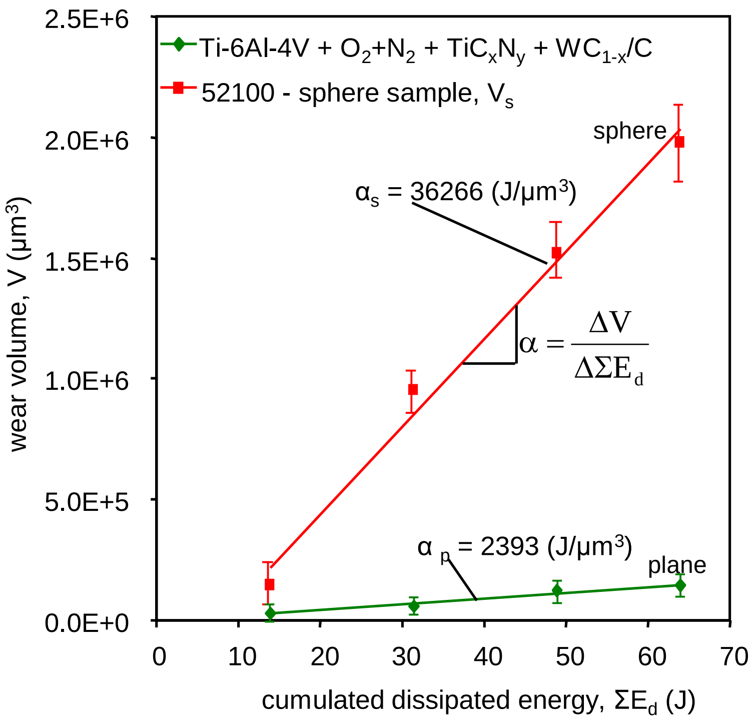

Fig. 8: Linear evolution of wear volume (V) as a function of cumulated energy dissipated in contact during the test ΣE_d, for the multilayer system on Ti-6Al-4V alloy with external nanocomposite carbon-based WC_{1-x}/C layer against 52100 sphere.

For large displacement amplitude (δ^*=100 µm) the relatively thin coating system of 0.3 µm of WC_{1-x}/C and 2 µm of TiC_xN_y have been worn and the Ti-6Al-4V substrate has been reached. However, for the smaller amplitudes of displacement the maximum depth of wear measured on plane surfaces remains lower than 1.8 µm suggesting that only external nanocomposite coating was removed due to very severe contact conditions. Therefore, it is important to note that all three processes: diffusion hardening with interstitial O and N atoms, intermediate TiC_xN_y gradient

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coating and top layer of nanocomposite WC_{1-x}/C coating are required to guarantee such a significant wear resistance improvement as achieved in this study. Results of wear analysis taking into account total wear volume ($V_t=V_p+V_s$) observed on the plane and on the sphere counterparts have been presented in Fig. 9 and summarized in Table 2. For coated tribosystem a significant reduction in the wear rate of 94% in comparison to uncoated Ti-6Al-4V can be observed for $\delta^*=25$ µm displacement amplitude and 99% for $\delta^*=100$ µm. This spectacular wear rate reduction is a result of multilayer treatment combining substrate diffusion hardening, the intermediate TiC_xN_y coating and the top nanocomposite WC_{1-x}/C coating deposition.

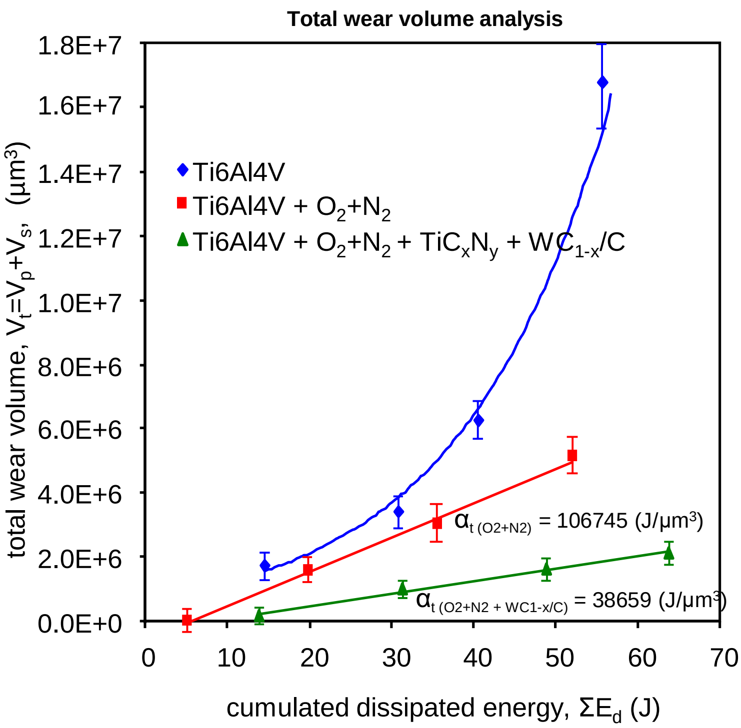

Fig. 9: Evolution of total wear volume ($V_t = V_p + V_s$) as a function of cumulated dissipated energy ΣE_d.

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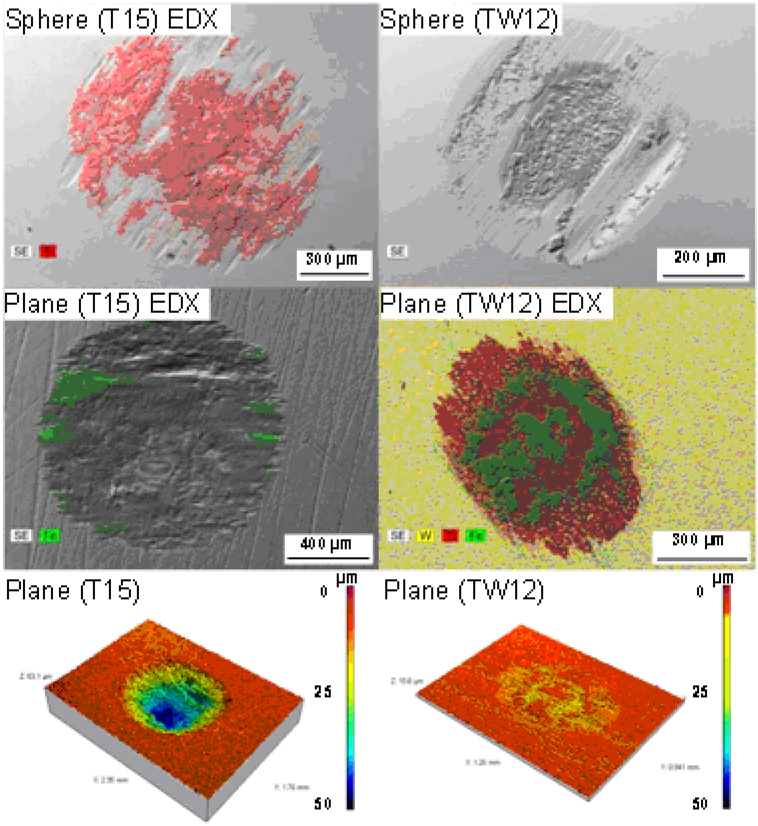

Fig. 10: Wear scar: SEM images and 3D morphologies of uncoated Ti-6Al-4V // 52100 tribosystem and multilayer system on Ti-6Al-4V alloy with an external nanocomposite carbon-based WC_{1-x}/C layer against 52100 sphere (displacement amplitude δ*=100 µm).

Table 2: Wear and friction results of experimental tribological analysis.

Sphere material 52100 (D=12.7 mm), Frequency F=10 Hz, Normal force P=20 N							
Test No.	Plane Material	Displ. amplitude	Averaged COF $\mu_a = \sum_1^N \frac{1}{N} \cdot \frac{Q^*}{P}$	Cumulated Dissipated Energy	Wear Volume of sphere (V_s) and plane (V_p) specimens		Maximum wear depth on plane specimen
		δ^* (µm)		ΣE_d (J)	V_s (µm^3)	V_p (µm^3)	H_{max} (µm)
T18	Ti-6Al-4V	25	0.85	14.657	1.212E+06	5.117E+05	6.1
T17		50	0.87	30.939	2.259E+06	1.165E+06	10.7
T16		75	0.85	40.620	1.376E+06	4.891E+06	20.6
T15		100	0.84	55.782	3.695E+05	1.716E+07	36.3
TN43	Ti-6Al-4V +N_2 O_2	25	0.74	5.216	2.00E+04	1.09E+03	0.59
TN44		50	0.82	19.826	1.00E+06	5.46E+05	3.1
TN45		75	0.89	35.688	1.90E+06	1.09E+06	8.1
TN46		100	0.88	51.175	3.05E+06	2.06E+06	10.0
TW19	Ti-6Al-4V +N_2 O_2	25	0.75	13.842	1.456E+05	3.062E+04	1.7
TW14		50	0.81	31.406	9.511E+05	5.842E+04	1.6

TW13	+WC_{1-x}/C	75	0.84	48.969	1.522E+06	1.210E+05	1.8
TW12		100	0.81	63.867	1.976E+06	1.432E+05	1.9

4 Conclusions

The new multilayer system of nanocomposite WC_{1-x}/C coating deposited on a diffusion hardened Ti-6Al-4V has been developed. Tribological performance of this new system has been investigated under severe fretting contact conditions and the results of friction and wear analysis are reported.

Developed multilayer coating system for titanium alloy consists of the following layers:

1) diffusion hardened layer with interstitial O and N atoms obtained by glow discharge plasma in the atmosphere of Ar+O2 and Ar+N2,
2) intermediate layer of 2 µm thick TiC_xN_y coating deposited by reactive arc evaporation,
3) external layer of 0.3 µm thick nanocomposite carbon-based WC1-x/C coating deposited by the reactive magnetron sputtering of the graphite and tungsten targets.

Based on the result of experimental analysis, the following conclusions can be formulated:

- Significant reduction of wear rate of titanium material (up to 94% less wear volume observed) has been achieved by developed multilayer coating system,
- Adhesive wear mechanism observed for untreated surface changed to abrasive wear mechanism after diffusion hardening by O_2 and N_2 atoms.
- Similar coefficient of friction was maintained after the coating system deposition and only small drop of about 10% was observed to average value of μ_a=0.8 for the coated titanium alloy // 52100 system.

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