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The application of an assisting gas plasma generator for low-temperature magnetron sputtering of Ti-C-Mo-S antifriction coatings on titanium alloys

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Abstract. The positive effect of assisting influence of high-density gas plasma formed by an independent plasma generator PINK on mechanical and tribological characteristics of Ti-C-Mo-S magnetron coating on titanium alloys at lowered to 350°C temperature of coating regardless of alloy structural condition was revealed by methods of calotest, nanorecognition, scratch testing and frictional material tests. The coating formed by means of a combined magnetron plasma method reduces titanium alloys friction coefficient in multiple times and increases wear resistance by two orders of magnitude. At the same time the mechanical properties of ultra-fine-grained titanium alloys obtained by nanostructuring do not deteriorate.

1. Introduction

Until recently methods of oxidation, nitriding and borating from gas and solid phase with alloy heating to high temperatures (800°C and higher) and long-term aging [1] have been commonly used to increase wear resistance of titanium alloys and decrease their adhesive and frictional interaction with a counter body in friction pair. However, the conventional high-temperature methods of surface modification mentioned above are inappropriate for alloys in nanostructured condition because of high probability of grain growth and loss of bulk stress-stain properties achieved by nanostructuring. At present the research of alternative methods of titanium alloys surface modification in particular by microarc oxidation, electro-explosive alloying with electron beam post irradiation or ion-beam surface treatment [2] is carried out. Methods of ion-plasma sputtering are also among modern methods of metals and alloys surface modification. Obviously, in order to obtain the adhesive strength of coating to substrate and the entire reaction behavior of ion plasma synthesis of necessary coating compounds it is essential to provide the sufficient diffuse atomic mobility of a deposited coating, which is usually achieved by substrate heating to high temperatures. Owing to the high heating of nanostructured alloys is inadmissible, the application of the assisting influence on a deposited coating by means of high-energy ions generated by an independent source is an advanced method to increase the diffusive atomic mobility of deposited coatings. The research carried out in the synthesis of wear resistant steel substrate coatings [3, 4] is an example of this technological method implementation. In the present work the technological method mentioned above and its potentialities in composite anti-friction Ti-C-Mo-S sputteringonVT6 titanium alloy in nanostructured condition are being investigated.

2. Materials and routine of experiment

The mechanical, physical and tribological investigations of properties of Ti-C-Mo-S coating deposited by the conventional method of magnetron sputtering and the combined method of magnetron sputtering with simultaneous assisting influence of gas-discharged plasma ions, formed by an independent plasma generator (magnetron-plasma method), were examined on VT6 ultra-fine-grained titanium alloy (hereinafter VT6ufg) with the compound of 5.5-7.0% Al, 4.2-6.0% V, Ti- the rest of it [5]. In order to compare the results, some identical investigations were carried out for VT6 and VT14 alloys in coarse-grained condition (VT6c и VT14c, respectively). VT14 alloy composition is the following: 4.5-6.3% Al, 0.9-1.9% V, 2.5-3.8% Mo, Ti - the rest of it [5]. Both alloys examined are alpha-beta titanium alloys. So as to form in VT6 titanium alloy blanks an ultra-fine-grained structure which decreases bulk strength, the method of severe plastic deformation (abc-pressing) with simultaneous decrease of blanks heating temperature in accordance with a stated procedure was used. After this treatment an average grain size was 0.7 μm. The strength properties of the initial VT6**c** alloy and VT6ufg alloy both after ion-plasma doping and/or sputtering were measured at indoor temperature with tensile test of a flat shoulder specimen [6]. The specimen-grid holders in the form of discs with 30 mm diameter and 4 mm thickness were made to carry out the comparative mechanical and tribological tests of initial alloys and deposited alloys. The deposition of Ti-C-Mo-S coating on titanium alloy substrates was produced by means of "Composit-3" [7] setup with a duomagnetron with two Ti-C-Mo-S sputtering targets and a PINK gas plasma generator [8] inside the working chamber. The targets were produced by self-propagating high-temperature synthesis (SHS) method with simultaneous compression of ignited alloy of a set composition from powder components. During the ion plasma spraying, argon was used as plasma-forming gas. Magnetron discharge capacity on each sputtering target was approximately 1500 W. Substrate accelerating potential was 200 V. Discharge current of an assisting plasma generator was regulated so that the substrate temperature did not exceed 350ºC. In all cases the method of ion plasma predoping of substrate by Ti, C, Mo, S atoms [9] was applied. The deposition time was 60 minutes. The research of mechanical and tribological properties was performed by means of calotest for measuring coating thickness, hardness measurement by nanorecognition on «NHT-S-AX-000X» device on 0,01 N loading, scratch testing on «Micro Scratch Tester» (CSEM) device with the registration of acoustic emission (AE) signal, friction coefficient testing during frictional material tests together with a counter-body, made of identical titanium alloy in the form of a cone with corner radius R=1mm or with a tempered steel rotating sphere HRC63. The tribological study was realized by means of «THT-S-AX000» (CSM) tribometer in accordance with the following order: a static indentor – rotating disk (pin-on-disc). In all cases the indentation load was 10N. The linear velocity of a counter body slippage was equal to 2 cm/s. The appraisal depreciation of surface coating of initial specimens and deposited ones was realized by «Micro Measure 3D Station» (STIL) by means of the calculation of averaged cross-section wear tack after the fixed friction testing cycle (3000 rotations). Comparative tribological tests were carried out on the same specimen, one side of which was in the initial condition and another was tested with Ti-C-Mo-S deposited coating. The optic microscopy of coating failure on calotest depressions and scratch track was conducted. The scratch test was carried out at the load leading to an explicit signal of acoustic emission.

3. Experimental investigations results and their consideration

The evaluation of coating thickness by means of calotest showed the following. At the same deposition time in 60 minutes, thickness of coatings deposited with simultaneous assisting influence of gas-discharge plasma formed by an independent plasma generator is less than those deposited by the conventional method. The coating thickness on VT6ufg alloy deposited by the combined magnetron plasma method is 1.2 μm whereas by the conventional method the coating thickness is 2.5 μm (figure 1). On VT14c alloy these quantities are 1.7 μm and 2.9 μm, respectively. Quite evidently, it is connected with an additional amplification of spraying intensity deposited material atoms by ions of high-dense gas-discharge plasma argon. Besides, it should be mentioned that in contrast to the magnetron plasma coating failure (comparison figure 1a and figure 1b) the deposited coating formed by the conventional method of magnetron sputtering at moderate (about 350°C) substrate temperature delaminates into large scales around an indenter under calotest circular and load-bearing influence. The same pattern is typical for coatings deposited on VT14c alloy substrate as well.

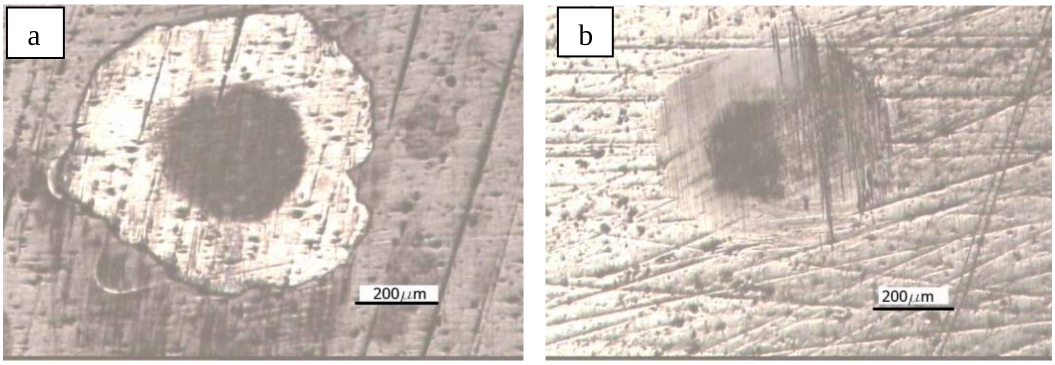


Figure 1. The images of calottes sections of Ti-C-Mo-S coating deposited on a substrate from VT6ufg nanostructured alloy by the conventional magnetron sputtering method (a) and by the magnetron plasma method with an assisting influence of gas discharge plasma from an independent source PINK (b) with indication of thickness index.

In figures 2 and 3 the typical scratch test results with emphasized signals of acoustic emission (AE) and distinctive images of scratch tracks in places of significant initial signals of AE are shown.

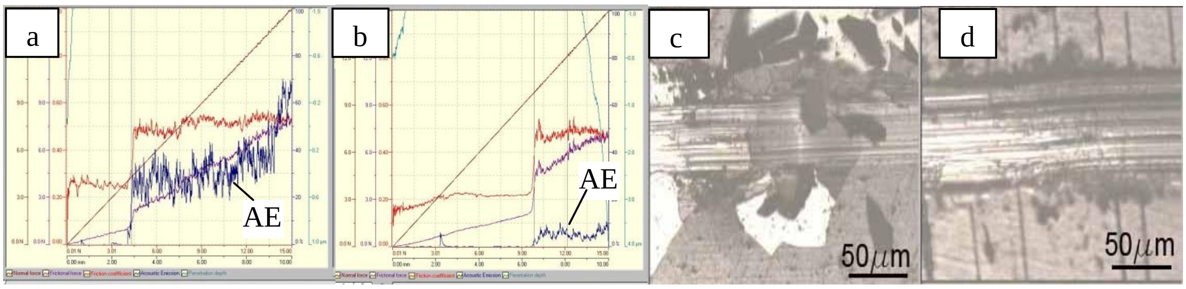


Figure 2. Load dependence of AE signal for Ti-C-Mo-S coating, deposited on nanostructured VT6ufg titanium alloy by means of the conventional method (a) and the combined magnetron plasma method (b) with images of coating failure peripherally a scratch track AE initial signal (c, d)

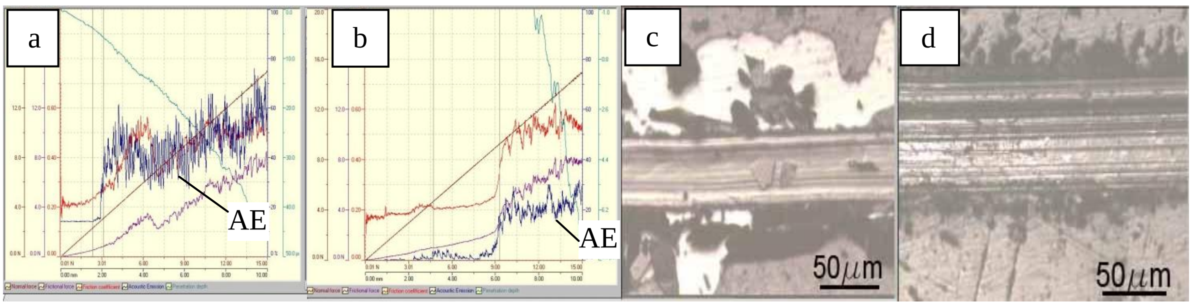
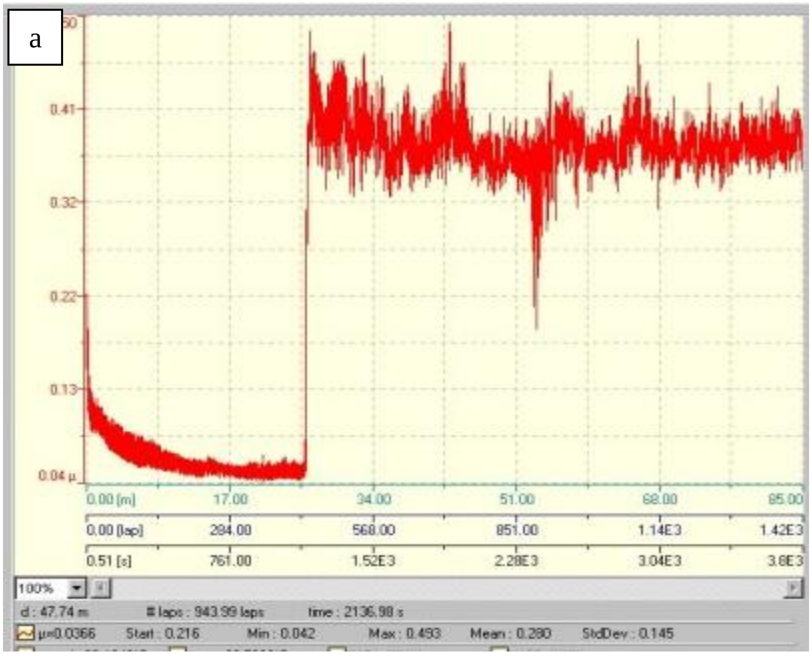


Figure 3. Load dependence of AE signal for Ti-C-Mo-S coating, deposited on nanostructured VT14c titanium alloy by means of the conventional method (a) and the combined magnetron plasma method (b) with images of coating failure peripherally a scratch track AE initial signal (c, d).

During the testing of coatings deposited by the conventional magnetron sputtering, the AE signal initiation is observed at loads ≈3÷4 N for substrates both from VT6ufg and VT14c alloys (figure 2a and figure 3a). For both titanium alloys AE signal at maximal load of 15N is ≈60%. Load dependence of AE signal changes considerably during testing of coatings deposited by the combined magnetron plasma method. The AE signal initiation is registered at bigger for both alloys load (9-10N). The pick/maximum value of signal at load of 15N is 20-30% for substrate from VT14c alloy and not more than does not exceed 10% for substrate from VT6ufg alloy (figure 2b and figure 3b).In accordance with AE curve behavior, peripherally a scratch track there is considerable difference in coating failure inflicted by two mentioned methods. During the conventional method of magnetron sputteringVT6ufg substrate coating fails and delaminates into large scales at load of about 4.5 N (figure 2c). It is accompanied by AE signal increase. In case of the combined magnetron plasma method AE signal increase is observed at load of ≈10N and coating failure occurs with fewer amounts of particles (figure 2d). The similar pattern in coating failure and AE signal generation is observed during comparative scratch tests of coatings on coarse-grained VT14c titanium alloy deposited by means of two mentioned methods (figure 3c and figure 3d).

Hardness tests also demonstrated differences in two methods of sputtering. An averaged value of hardness for VT6ufg alloy substrate coating deposited by the conventional magnetron method is 6.5 GPa while by the combined magnetron plasma method (with assisting influence of gas-discharged plasma) is 7.1GPa. The maximum hardness value of coating deposited by the magnetron plasma method, in comparison with the conventional magnetron sputtering, was also registered on VT14c alloy (8.6 GPa comparatively with 7.9 GPa). From the total analysis of the above results we can conclude that bombardment of deposited material by gas ions of gas-discharged plasma (with higher plasma density than at magnetron discharge) from an independent PINK source notably increases a diffusion process. In its turn, it increases adhesive strength of the coating to the substrate, compound synthesis probability (solid carbide, in particular), has effect on structure and hardness of a deposited coating.

The comparative friction test also showed the advantage of Ti-C-Mo-S magnetron plasma sputtering in comparison with the conventional magnetron sputtering in friction pair both with a counter-body from identical titanium alloy and with a counter-body from SH-15 steel hardened to HRC63 (figure 4).



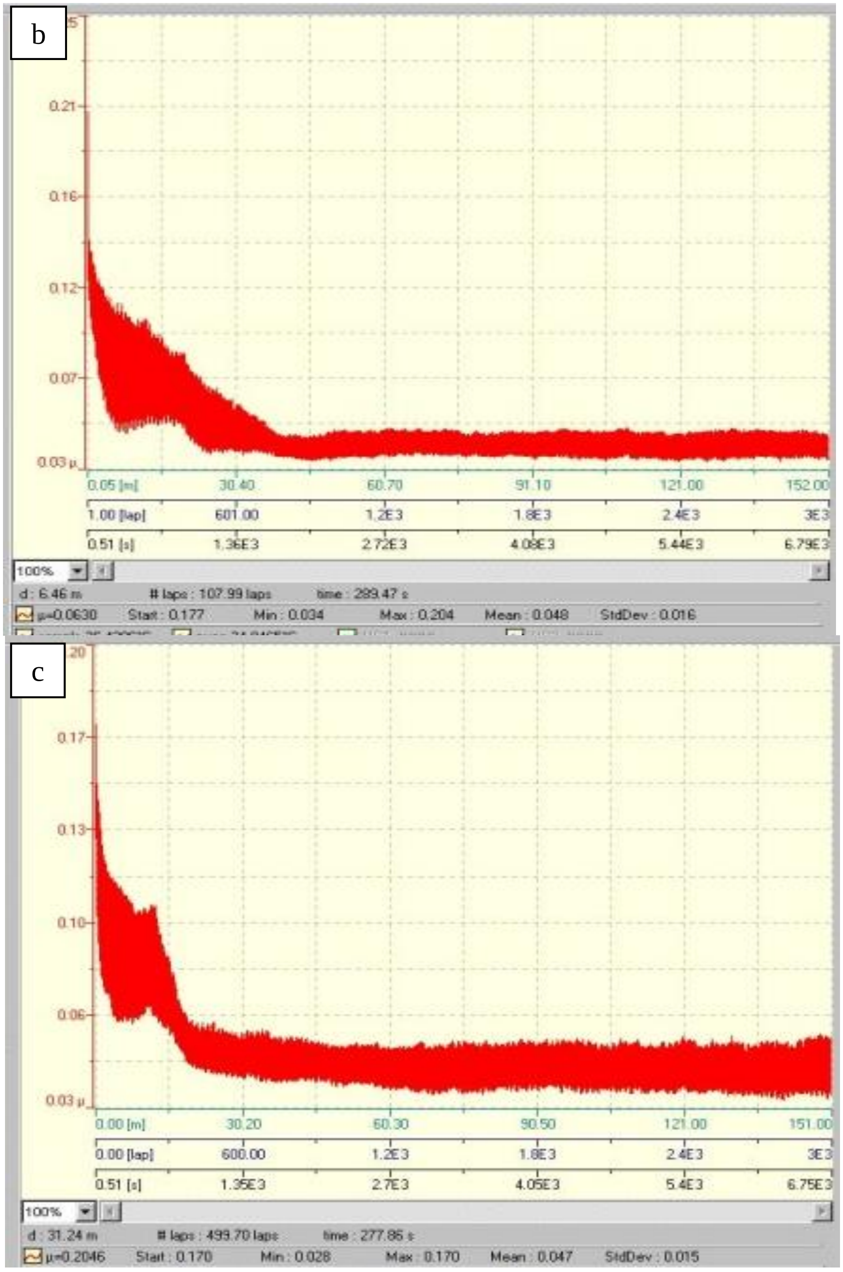


Figure 4. The dependence of friction coefficient μ from turning number of substrate-disc from VT6ufg alloy (friction path) for a coating deposited by the conventional magnetron sputtering method (a) and by the combined magnetron plasma method with an assisting influence of a gas plasma generator PINK (b, c). Counter body material is VT6ufg alloy with modified surface and SH-15 hardened steel (c).

The friction coefficient of coating deposited by conventional techniques after 400 revolutions of the disk increases sharply to 0.4 (figure 4a). Such kind of «a break» or coating delamination is connected with its weak adhesion (minor adhesive strength) with a substrate. It is correlated with the mechanical investigation results mentioned above. During the magnetron plasma sputtering method the minimal friction coefficient (about 0.045) remains while measuring (3000 rotations) when at friction test a counter-body from identical titanium alloy as well as a counter-body from hardened steel are used (figure 4b and figure 4c). The obtained results of friction tests bring out clearly a profound effect of assisting gas discharge plasma formed by an independent source PINK on strength and wear resistance of Ti-C-Mo-S coating. As it has been reported before [6], μ friction coefficient of titanium alloys decreases in multiple times while coating wear resistance increases by a factor of 10^2.

Control research of the effect of magnetron plasma coating deposited at temperature of 350°C on strength and ductility of an initial coarse-grained and nanostructured (ultra-fine-grained) VT6 titanium alloy were carried out. Control research did not reveal the decrease of the following characteristics (table 1).

Table 1. The effect of Ti-C-Mo-S magnetron plasma coating on ultimate strength (σ_B) and percentage extension before failure (δ) of VT6 alloy in coarse-grained and ultra-fine-grained condition.

Alloy condition	Strength and ductility initial indexes		Strength and ductility indexes after coating magnetron plasma sputtering	
	σ_B, MPa	δ, %	σ_B, MPa	δ, %
coarse-grained	900±10	10.5±0.5	With coating 920±10	With coating 9.5±0.4
ultra-fine-grained	1040±10	10.5±0.5	With coating 1070±10	With coating 13.0±0.3

4. Conclusion

Additional (assisting) influence of high-density gas-discharged plasma formed by an independent PINK source on substrate and deposited material has the potential to increase mechanical and tribological properties of Ti-C-Mo-S deposited magnetron coating at lower temperature of a substrate and to use this combined method of coating synthesis for nanostructured alloys on the basis of titanium, in particular.

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