Formation of wear resistant nanostructural topocomposite coatings on metal materials by ionic-plasma processing

D N Korotaev¹, E E Tarasov², K N Poleschenko³, E N Eremin⁴, E V Ivanova⁵

¹Siberian State Automobile and Highway University, 5, Mira ave., Omsk, 644080, Russia
²FGUP Federal Research and Development Centre “Progress”, 4, 5th Kordnaya street, Omsk, 644018, Russia
³Omsk Research Institute of Process and Engine Manufacture (RIPEM), 283, street of B. Khmelnytskyi, Omsk, 644021, Russia
⁴Omsk State Technical University, 11, Mira ave., Omsk, 644050, Russia
⁵Omsk Tank-Automotive Engineering Institute, 119, 14th military town, Omsk, 644098, Russia

E-mail: korotaevd99@mail.ru

Abstract. The article regards formation processes of activation and dissipative energy flows under ion-plasma sputtering of coatings. It proposes an advanced system of ionic-plasma processing with the activation of the processed surface by high voltage pulses of electric potential. The authors carry out the experimental evaluation of tribological properties for the samples with coatings and study the features of their surface damage. It is established that the topokompozitny coverings, containing high-disperse nanodimensional clusters reduce probability of brittle destruction during an initial stage of operation of tribocouplings. They show that the use of high-frequency pulse oscillations under ion-plasma sputtering decreases friction ratio and increases wear resistance of coatings.

1. Introduction
Reliability of current technology and the equipment of the machine-building complex depends first on service properties of the materials the details of friction units are made of. The increase in working capacity of surfaces of the metal products, operated under high loadings and speeds, is achieved by coating process providing the formation of optimum meso - micro - nanostructures of the materials. Consequently, the creation of new materials and wear resistant functional coatings, achieved by means of concentrated energy flows, is a promising direction of material science and tribology.
Vacuum ionic-plasma processing (VIPP) possesses wide technological capabilities of composite coatings formation for various service conditions of constructional parts for tribocouplings made of metallic. VIPP with nanostructural topocomposite coatings (NSTCC) application allows modelling the modified structure corresponding to the positive gradient code of mechanical properties in depth. It favorably influences tribotechnical characteristics of friction units [1, 2].

2. Problem statement
There are stress concentrators of a various scale which cause crack nucleation and fatigue failure initiation under ion-plasma sputtering at the interface «a hardened layer (coating) - a base material» [3]. Firstly, this is because when the coating is enlarged for more than 4-5 μm thick, significant
locked-up stresses can be formed in it. They lead to fast fracture under operating conditions of the product. Secondly, low coating adhesion with the base material can lead to stripping at the initial stage of the product operation. It sharply reduces its service life. Therefore, the development of minimization methods for sudden failures of products in the process of their operation is a major materials science problem.

On the contrary, the creation of nanostructural surface layers with the properties, changing in depth, provides the development of relaxational and adaptive processes under the tribocontact due to the size reduction of deformable volumes, stress decrease in their shearing stability and, consequently, level decrease in peak stresses under operating conditions of modified constructional parts [4].

Considering multifactoring of the phenomena proceeding in surface coatings of the materials under VIPP, it is appropriate their synthesis and analysis from the perspective of the thermodynamic approach [5]. It enables to analyze the influence of structural and technological parameters on adhesion strength of constructional parts with topcomposite coatings considering their adaptive behaviour under operating conditions [6].

The purpose of the given work is the creation and research of new nanostructural topcomposite coatings under structural modification of metal surfaces by the method of vacuum ion-plasma sputtering.

3. Materials and methods

Formation and comparative effectiveness research of nanostructural topcomposite coatings is carried out on the samples of different sorts of structural alloy steels with the use of two VIPP variants. In the work the results of the research for the structural alloy steel 15HGN2TA are given. In the first variant, the three-cathodic system [7] is used. In the second variant, the reflector of the ions is applied, besides the cathodic system. It enables to carry out ionic-plasma processing in the conditions of ionic assisting (fig. 1).

![Figure 1](image-url)

**Figure 1.** The scheme of the cathodic system with ionic assisting. Designations: K1, K2, K3 – cathodes; 1 – a template; 2 – a sample; 3 – a holder of samples; 4 – a reflector; 5 – a vacuum chamber; 6 – flows of ions.

In the first variant (mode I) coating formation is carried out from the flows of gas and metal plasma without impulse voltage input. In the activation mode the voltage across the underlayer changes in the range of 80-600V at current values of 100A. Coating deposition is carried out at the pressure of 900V with the current strength of 180A.

In the second variant (mode II) under activation across the underlayer there are high-voltage pulses of electric potential with the range of about 10kV, with the period of 10-20 μs and with the frequency of...
10-15 kHz under constant negative voltage of 1000V. In the course of the coating deposition on the underlayer the voltage impulse amplitude is about 5kV, the impulse period is about 50-60 μs, the frequency is of 15-25 kHz under constant negative voltage of 1000V. Besides, the reflector of the special design (fig. 1) and the grid pattern are used to receive multicharged ions in the stream. In both cases, the titanic alloy BT5 is used as the material for the cathodes. Nitrogen is used as a working substance.

Researches of changes of the surface morphology after various modes of ionic-plasma activation and coating deposition are carried out on the scanning probe microscope NTEGRA Prima in the mode of the contact atomic force microscopy (c-AFM). The research of wear resistance of the samples is carried out with the friction factor on the vibration shaker according to the scheme "a shaft- a shoe" under the following conditions: the pressure P = 4 MPa; the traversing speed V = 1 m/s; the testing time t = 60 mines; the vibration frequency f = 10 Hz; the displacement amplitude Ap = 0.2 mm.

4. Results and discussion

There are interacting processes of energy flows, ions or diffusing elements with the process material under ionic-plasma impact on the material surface. It is possible to present the process of formation NSTCC in the form of the scheme given in fig. 2 that is based on the three-cathodic system [7, 8], which is used earlier. There is a cathode sputtering with ions, metal and gas mixture production under the voltage generation between the cathode and the anode and in the process of the delivery of the working substance (gas) to the working chamber. It leads to primary surface sputtering or to ions deposition according to the energy of ions and the location of cathodes. There is a jump of the temperature spurring trouble processes of its crystal structure, mass transfer, defect and metastable structure (MS1, MS2) formation because of the energy deposition on the surface layer of the process material. The response of two processes simultaneously (both activation and dissipation) on VIPP leads to the disbalance of energy flows. The prevalence of the dissipative process leading to the ionic flows deposition on the surface with the formation of nanophase germs and their subsequent growth, promotes the nanostructural coating layers [8]. Thermodynamic processes of energy and mass transfer are developed at all stages of ionic-plasma impact.

![Figure 2. Structural diagram for producing nanostructural topocomposite coatings by ionic-plasma processing. Designations: VS1 – a voltage supply; VS2 – a high-voltage impulse supply; WS – working substance (gas); GD – a glow discharge; A – an anode (a process sample); K1, K2, K3 – cathodes; MS1, MS2 – metastable structures; SNC – a stabilized nanostructural surface layer](image-url)
condition; IF1, IF2, IF3 – ion flows generated by corresponding cathodes; Udf – dissipative thermal flows.

High density of the discharge power under VIPP and short duration of its active interaction period with the material keep from generating effective thermal dissipative channels. In this connection, there is a disbalance share of energy deposition, which prevents from relaxation and dissipation of discharge thermal energy.

In fig. 3 there are hypothetical dependences of power density of the energy flow under the activation process (curve 1), the thermal dissipative process (curve 2) and the current density of power consumption in the course of disbalance (zone 3 and curve 4). The charge power density reaches a maximum level at the end of the initial activation period \( t_a \), and then reduces to zero after the time \( t_p \).

![Figure 3. Energetic characteristics of the ionic flow under VIPP: 1 – Power density of the ionic flow for the activation process; 2 – Power density of the flow for the dissipative thermal process; 3 and 4 – disbalance of energy flow](image)

Potentialities of the dissipative thermal channel grow up according to the temperature field generation, however, during the initial period of the ionic-plasma impact its intensity is considerably behind the intensity of the activation process. The maximum activity of the dissipation thermal process is achieved when the peak of energy brought by the ion charge is passed. The balance between the energy of the activation process and the energy taken away by the thermal process is achieved at point A. Hence, the formation of metastable structures takes place in the period from zero to \( t_d \) when a dissipative thermal process cannot balance the input charge energy. The disbalance of energy flows under VIPP is defined by the area of the shaded zone 3, and the time dependence for power density of power consumption for the disbalance neutralisation can be presented by curve 4, as the difference between the ordinates of curve 1 and curve 2 in the time span from 0 to \( t_d \).

Thus, in the system two energy flows are set and operate simultaneously: 1 – an activation flow of the input energy, 2 – a dissipative flow, which has a thermal nature and is accompanied by energy and mass transfer processes. The simultaneous development of the two energy flows leads the system to the stabilized nanostructural condition (SNC).

According to the results of the thermodynamic analysis of activation and dissipative flows, developing under VIPP, it is required to provide the stabilization of the nanostructural condition for the coating. For this purpose, it is necessary to increase the values of power density of energy deposition at the
expense of voltage and current increase, because the achievement of their maximum values is limited by technological opportunities of the unit. For the solution of the problem, a cathodic system is developed. The scheme of this system is presented in fig. 1.

The analysis of the given electron microscope images (fig. 4) demonstrates that the surface activation before the coating process in the mode of ionic assisting leads to the development of high-dispersity nanosized formations (fig. 4, a) and to the increase in their density in comparison with the activation without the accelerating voltage supply(fig. 4, b).

![Figure 4](image_url)

**Figure 4.** Surface images of the nanostructural topocomposite coating after ionic-plasma processing: 
a) - voltage with the amplitude of 10 kV, the duration of 10-20 μs, the frequency of 10-15 kHz under constant negative voltage of 1000V; b) – the voltage across the underlayer of 180V, the current strength of 100 А.

Fig. 5 presents the given changes of the friction ratio \( f \) in the course of the surface wear process.

![Figure 5](image_url)

**Figure 5.** Friction ratio dependence on the testing time of steel samples 15HGN2TA with nanostructural topocomposite coatings: 1 – the voltage across the underlayer of 80V, the current strength of 100A; 2 – the voltage across the underlayer of 100V, the current strength of 120A; 3 – the voltage across the underlayer of 120V, the current strength of 130A; 4 – the voltage across the underlayer of 150V, the current strength of 150A; 5 – the voltage with the amplitude of 5 kV, the duration of 10-20 μs, the frequency of 10-15 kHz, the negative voltage of 1000V; 6 – the voltage with
the amplitude of 10 kV, the duration of 10-20 μs, the frequency of 10-15 kHz, the negative voltage of 1000 V.

The analysis of the quoted results suggests that the greatest decrease in the friction ratio is achieved involving the cathodic system, given in fig. 1 under the surface activation by means of high-voltage impulses supply across the underlayer. Moreover, the increase in the amplitude of impulses from 5 to 10 kV has a great effect on the decrease in the friction ratio (dependences 5 and 6 in fig. 5).

5. Conclusion

1. The development of the structural diagram for nanostructural topocomposite coatings is proposed based on the analysis of activation and dissipative processes under forming functional coatings by ionic-plasma processing.
2. The system of ionic-plasma processing including a cathodic complex with ionic assisting and the reflector of the special design are developed for the efficiency upgrading of the ion-plasma sputtering method and for the generation of the stabilized condition of nanostructural coatings. It enables to receive topocomposite surface layers with the structure, containing high-dispersity nanosized formations, reducing the probability of fracture failure of coatings during the initial operating period for modified structural components of tribocouplings.
3. The result of the tribological tests is that the greatest decrease in the friction ratio is achieved involving the advanced cathodic system under the surface activation in the course of the formation of nanostructural topocomposite coatings by means of high-voltage impulses supply across the underlayer.

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