Producing multilayer composites based on metal-carbon by vacuum ion-plasma method

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Abstract. The possibility of changing the coating properties and perspective of forming a compound having the unique mechanical properties, high hardness and high corrosion resistance is of particular interest to multilayer coatings based on metal-carbon composition. To investigate the mechanisms of formation of coatings and to demonstrate technological possibilities synthesis technology of multilayer composites, the titanium cathode and the silicon-graphite cathode were made. Coatings microhardness analysis showed that in forming the multilayer structure, microhardness increases by 15–20 %, and when forming a multilayer composite, microhardness increases by 60–65 %.

In most cases, when the parts are exposed to an aggressive environment, such as wear abrasion, corrosion and erosion, exposure to high temperatures, the destruction begins from the surface. To protect the surface, a method of vacuum-plasma deposition of coatings is most prevalent. This method is characterized by the possibility of obtaining surface layers with special physical and mechanical properties, and helps to protect the parts during the entire operating life. The suggested method has an almost complete universality of the technological process, high productivity, high reproducibility of parameters on each operation and almost complete lack of environmental problems [1–3].

There are several main reasons hindering the widespread implementation of ion-plasma coatings in friction units of machines and mechanisms, in particular aviation ones.

First, for friction units of aircraft components, we need to provide wear resistance and corrosion protection, which is not always achieved when the protective layer thickness is several micrometers (3...5 μm) produced by traditional technologies.

Secondly, it is necessary to ensure sufficient fatigue strength of the parts with coating. It can be achieved by a reduction of residual stresses. Therefore, we need to increase the thickness of the coating, to create therein transition layers or multilayer structure [1].

With this in mind, we carried out studies on the creation of multilayer coatings of Ti–C system. In this case, multilayering provides a smooth reduction of residual stresses on thickness, a reduction of porosity and defects in the coating, an increase the integrated thickness of coating without sacrificing operational characteristics and an ensuring a smooth transition from soft base to solid functional layer [2, 3].

The possibility of changing the coating properties and perspective of forming a compound having the unique mechanical properties, high hardness and high corrosion resistance is of particular interest to multilayer coatings based on metal-carbon composition.
For the deposition of coatings with improved properties, the technology of coating multilayer composite has been developed in relation to the modernized industrial plant NNV 6.6-I1, which includes: a preliminary surface preparation; ion cleaning of the surface; applying a multilayer composite (carbon–metal).

Plant lay-out for coating a multilayer composite is shown in figure 1. The preliminary surface preparation is used to remove from the surface different kinds of pollution. Then the parts are placed in the vacuum chamber and pressure is generated. Preliminary ionic purification is carried out by a non-self-maintained high-current diffusion discharge generated by plasma source PSIC, the use of which gives the additional acceleration of ions and acceleration of diffusion processes in the near-surface layers of the treated parts.

![Plant lay-out of NNV 6.6-I1 modernized for applying the multilayer composites](image)

**Figure 1.** Plant lay-out of NNV 6.6-I1 modernized for applying the multilayer composites:
1 – vacuum chamber; 2 – anode; 3, 4 – cooled cathodes made of sprayed material, titanium and graphite-doped silicon, respectively; 5 – table; 6 – part; 7, 8 – power supply.

The next step of technology is a cleaning the surface by the metal ions. It is carried out in a vacuum chamber at a certain pressure in an inert gas argon (Ar) atmosphere. The accelerating voltage is not less than 1100 V is applied to the substrate at an arc current of 40–50 A. Duration of procedure is 1–2 min till the termination of microarcs occurrence. Cleaning also allows preheating the parts before coating.

Step of technology related with the formation of the multilayer composite (carbon–metal) occurs during the serial deposition of layers of metal and carbon from plasma generated by electric arc plasma sources arranged at an angle to the substrate surface.

To investigate the mechanisms of formation of coatings and to demonstrate technological possibilities synthesis technology of multilayer composites, the titanium cathode (the Russian brand VT-1.0) and the silicon-graphite cathode were made. In the process of coating deposition, substrate evenly rotates on its axis and on the axis of the table together with the plant.

To determine patterns of synthesis, the samples were coated by the following types: a single-layer Ti–C coating, a multilayer Ti–C–Ti coating and the multilayer composite. A layer of titanium was the first and the last layers of each coating.

Investigations on adhesion strength of multilayer composites and multilayer coatings showed that the coatings have excellent adhesion to a base material. It was obtained by the study of the diamond pyramid's print scaled up by using an optical microscope. Lack of cracking of the material with coating near the impact zone of the indenter is proof of adhesive strength.

Coatings microhardness analysis showed that in forming the multilayer structure, microhardness increases by 15–20 %, and when forming a multilayer composite, microhardness increases by 60–65 %.
Corrosion resistance of samples with coatings was determined by gravimetric method. Corrosion resistance of the multilayer composite (Ti–C) is higher by 20–22% than that of samples with multilayer coating and by 25–30% than that of the uncoated samples (see table 1).

Table 1. The corrosion rate of samples with coatings.

| Type of coating       | Mass, g | Square of samples, m^2 | Corrosion rate, g/m^2·h |
|-----------------------|---------|------------------------|------------------------|
| Initial               | Initial | 4.39355                | 4.38625                | 0.0222379                |
| Multilayer coating    | 3.76240 | 3.75635                | 0.0015198              | 0.0184301                |
| Multilayer composite  | 4.39755 | 4.39245                | 0.0155361              |

Investigations of the electrode potential to determine the effectiveness of the multilayer composite Ti–C as protective coatings were carried out by the conventional method of observing the changes of the samples potentials in time. We used the block of universal voltmeter with a range of 7–35 V. The electrolyte was a 5% solution of NaCl. Reference electrode was silver chloride (AgCl) half-cell, immersed in a solution of KCl (saturated).

The electrode potential of multilayer coating applied to samples of steel is higher by 20–25% than the electrode potential of the sample coated with submicrocrystalline (SMC) structure (steel of the Russian brand 13H11N2VMF). Consequently, the surface of the multilayer composite Ti–C is more passive, which indicates its higher corrosion resistance. This can be explained by an increase of the number of boundaries between the layers. It inhibits corrosion processes at the boundary between medium and metal and prevents the penetration of external corrosive environment deep into coatings (figure 2).

![Figure 2. Electrode potential. Substrate 13H11N2VMF: 1 – initial state; 2 – multilayer coating; 3 – multilayer composite.](image)

Analysis of the samples with a multilayer composite Ti–C, obtained by vacuum ion-plasma method showed that the coatings deposited using plasma assisting provide higher protection against corrosion.
Furthermore, improved corrosion properties can be explained by increased density of the coating, restructured coatings and reduced residual stresses in the coating.

It was found that in comparison with a multilayer coating, the microhardness of coatings with SMC structure is higher by 30–50 %, electrode potential is lower by 20–25 %, the corrosion resistance of the multilayer composite (Ti–C) is higher by 20–22 % than that of samples with multilayer coating and by 25–30 % than that of the uncoated samples that is associated with an increase in the content of carbide and titanium carbosilicide in a multilayer composite.

References
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