Tribological properties of ion-modified composite coatings

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Abstract. The paper presents the results of a study of one of the ways to increase the wear resistance of “duplex” coatings applied to cutting tools, which are due to preliminary diffusion saturation of the tool surface with nitrogen (known as ion nitriding) followed by physical deposition of a hard coating (Ti, Cr) N. The proposed coating also contains an additional layer with an impurity of ions, deposited on a preliminary nitrided surface of high speed steel before the deposition of a hard coating. Tests were carried out to evaluate the effect of these modified layers on the tool life of the HSS tool. The greatest wear resistance after “triplex” - treatment was achieved during ion implantation of titanium into a pre-nitrided surface. The coefficient of friction of the modified layer was studied at different contact temperatures. Ionic mixing contributes to the appearance of a thin surface layer with an amorphous-like structure, which prolongs the stage of normal wear, which significantly increases the tool life as a result of the self-organization process.

1. Introduction

The wear of cutting tools can vary over a wide range, flow through various mechanisms, but in all cases it is a consequence of friction. Each tribo-system is characterized by the formation of secondary structures (BC) on the friction surfaces, which represent some "third" body that carries out protective functions, limiting the propagation of interaction inside the rubbing bodies. VS formation and wear are associated with energy transformations during friction, which can be considered from the standpoint of nonequilibrium thermodynamics and self-organization. At the same time, the problem of the choice of contacting materials comes to the fore, which, with the adopted lubricating medium (or in the absence of lubrication) and a given friction mode, are able to adapt (adapt) to each other in the process of mutual movement, providing a decrease in entropy on the surfaces friction and increasing their wear resistance [1–6, 8].

A series of experimental studies of the wear resistance of multilayer wear-resistant coatings on a cutting tool have been carried out. Moreover, each layer of such a coating should be formed taking into account the change in the wear mechanism during the periods of running-in, normal (stable) and catastrophic wear. It is known [2, 3, 5, 6] that cutting tools made of high-speed steel usually operate under conditions of an increased stress-strain state of the cutting wedge and intense abrasive (abrasive) wear, where seizure associated with a build-up occurs when the tool interacts with the workpiece ... The inevitable wear of the coating leads to an intensification of the abrasion process, which is accompanied by a rapid increase in the intensity of wear of the tool material itself (substrate). Consequently, tool wear quickly enters its catastrophic stage. Extending the normal friction and wear phase is, however, practically feasible, even if gripping (abrasion) is a common problem for a
particular cutting process. However, prolongation can be achieved by applying an additional subcoat in a multi-layer “duplex” coating on the surface of the tool substrate. This layer, after a triple surface treatment of the cutting tool, combines the desired protective and anti-friction properties when interacting with a hard coating. The way these layers are created is through an ionic mixing process in the surfaces of HSS cutting tools.

2. Experimental materials and methods
The paper considers a new “triplex” coating. "Triplex" - processing was carried out in three stages. The R6M5 high-speed steel substrate was originally nitrided by the glow discharge method. Before applying a hard coating, the surface of the instrument was modified with an ionic mixture. Finally, the modified layer was coated with (Ti, Cr) N by physical deposition of coatings (PPD).

Ion nitriding of the high speed steel substrate was carried out in a special device for ion nitriding NSV 6 / 10-11 with a mechanical pumping system and containing heating sources in the form of a glow discharge and permanent heating elements. The technological parameters were as follows: the current density of the glow discharge was 3 A × m$^{-2}$, the bias voltage was 600 V, the nitriding time was 0.5 hours, and the gas pressure was 266 Pa. The composition of the gas is 25% N2 + 75% H2 (decomposed ammonia) and a temperature of 500 ° C.

The hard coating was applied using a standard cathode arc in the FOP process in an NNV 6.6-I1 device with a mechanical and diffusion pumping system. Two titanium cathodes and one chromium cathode were used. The distance between the sample and the cathode was 200 mm. The coating was applied by rotating the sample in the chamber. The parameters for coating were as follows: reactive gas (nitrogen) pressure - 3 × 10$^{-1}$ Pa, arc discharge current - 100 A, substrate bias voltage - 200 V, substrate current density about 15 A × m$^{-2}$, deposition temperature 500 °C.

Before the deposition of a wear-resistant hard coating, the samples after ionic nitriding were modified with an ionic mixture of five combinations of different elements. Modification was carried out on a high-energy ion setup (ion implantator) with an energy of approximately 40 keV at room temperature. The size of the chamber of the device, equipped with a turbomolecular pumping system, was 50 × 350 × 1000 mm. The distance between the samples and the plates used was about 10 mm. The five pairs of combinations of metals and non-metals were: Zr + N, W + C, W + N, Ti + N and Al + O. Such combinations for the ionic mixture were chosen to create layers with high wear resistance based on adaptation of the surface layer for the formation of protective layers or sun. Typical doses used were 8 × 10$^{17}$ ions / cm$^{2}$. The current density in this case was 50 mA / cm$^{2}$. Before ionic mixing of the studied elements, the surface of the elements was etched with argon ions.

Experimental studies of the temperature and force conditions of cutting and wear of these coatings were carried out when turning 40X carbon steel at a cutting speed of 70 m / min, a cutting depth of 0.5 mm and a feed of 0.28 mm / rev with the use of coolant in the form of 5% emulsion and without her. The wear of the edges of four-sided quick-change plates made of high-speed steel with multilayer coatings was studied. The frictional properties of the analyzed tribopair were determined using an adhesiometer, the design of which is described in the source [7].

The phase composition of the FOP coating layer and the nitrided substrate was investigated using XRD. The chemical and phase compositions of the surface of the modified layers, as well as the contact area of the cutting tool and the workpiece, were studied using Auger - electron spectroscopy (OES). Extended fine structure analysis of electronic energy loss spectra (EELFAS) and secondary ion mass spectrometry (SIMS) were performed using a VG ESCALAB MK2 spectrometer. Scanning spectroscopy (OES) was used to analyze the surface composition of a wear chamfer on an oblique cut of the cutting tool surface at various stages of wear.

3. Results and discussion
The data obtained from the experimental tests of the wear resistance of cutting tools at various combinations of ion mixing are presented in table 1. The efficiency of ion modification - the relative period of tool life was determined by comparing the cutting time required to achieve a certain level of,
wear (the wear criterion of the cutting wedge according to of the rear surface for the conditions of finishing and semi-turning \( h_{\text{rad}} = 0.3 \text{ mm} \), on tools with “triplex” and “duplex” coatings. Eight cutting tests were carried out for each type of surface modification. The scatter of the results of determining the tool life was approximately 8 - 10%. They show the effect of ionic mixing on the tool life under the cutting conditions under study. At the same time, the operating temperature was within 500 ° C. When using coolant, the temperature decreased by 100 ° C with a corresponding increase in the tool life. The data presented in table 1 show that the best wear resistance after “triplex” surface treatment is achieved in an ion-modified layer containing Ti and N.

**Table 1.** The relative service life of a cutting tool with a triplex coating.

| Mixing elements | Relative durability period |
|-----------------|---------------------------|
|                 | No cooling | Refrigerated |
| Al + O          | 3.0        | –            |
| Ti + N          | 4.0        | 2.5          |
| Zr + N          | 0.53       | –            |
| W + N           | 0.4        | –            |
| W + C           | 1.33       | –            |

**Figure 1.** Frictional properties of cutting tools made of high-speed steel with a modified surface at different temperatures: ○ - Steel 40X - R6M5 (ion nitriding + coating (Ti, Cr) N); □ - Steel 40X - P6M5 (ion nitriding + ion mixing (Ti, N) + coating (Ti, Cr) N).
The results of field experiments to assess the friction parameters \( \tau_{nn}, p_{rn}, \tau_{nn}/p_{rn} \) at different temperatures are shown in fig. 1. It can be seen from this figure that the use of wear-resistant coatings with triplex treatment (ion nitriding + ion mixing (Ti, N) + coating (Ti, Cr) N) significantly reduces the shear strength \( \tau_{nn} \) of adhesive bonds and the complex parameter \( \tau_{nn}/p_{rn} \) practically in the entire investigated range of contact temperatures. This is also facilitated by the use of cutting fluid. In general, the results obtained confirmed the prediction about the improvement of the frictional properties of the contact between the tool and the processed materials when using "triplex" coatings.

To explain why ionic mixing increases the tool life several times, it is necessary to consider from the standpoint of tribology and synergics. It is known that thin protective films are formed on the surface as a result of the process of “self-organization” during friction [2]. These protective films are oversaturated with solid solutions of oxygen that comes from the environment. An amorphous-like structure can also be formed by reaction with the tool material. These types of Ti-O films are formed in the upper layers of the surface of the investigated TiCrN coating obtained by the FOP method. The initial amorphous-like surface structure found after ion mixing is similar to the structure of films formed on the surface by friction. The rapid formation of protective films is most likely when the hard coating interacts with the ion-mixed layer during cutting. Ionic mixing improves the process that naturally develops in the tribosystem in the process of self-organization and ends with the formation of stable protective films. These films delay the transition to the catastrophic stage of tool wear so that the tribosystem can return to a stable wear state again. Finally, due to these effects, the service life of cutting tools made of high speed steel, made using the technology of "triplex" coating, increased by 4 times.

4. Conclusion

In this work, some ways to improve the "duplex" coatings obtained by the FOP method and applied by double treatment of the surface layer of the tool were considered. This process includes diffusion saturation with nitrogen, known as ion nitriding of high speed steel tools, and hard (Ti, Cr) N plating by FOP. The coating contains an additional substrate modified by ion mixing, and the ion mixing was carried out on a high-speed steel surface pre-saturated with nitrogen ions. Such multilayer coatings allow to increase the tool life by 3-4 times. This is achieved primarily by extending the normal wear phase.

The effect on the tool life of five pairs of elements, which were added in the form of an ionic mixture to the main surface of a high-speed steel substrate, was investigated. The best wear resistance was obtained for a “triplex” coating with a substrate containing an ionic mixture (Ti + N).

The best effect of increasing the tool life is achieved when the ion-modified layer forms an amorphous-like structure with a lower coefficient of friction, an improved ability to accumulate elastic deformation energy, which prevent deep damage to the surface. This is realized when a layer modified with a Ti + N mixture by ionic mixing is applied. During cutting, oxygen-containing tri-films are intensively oxidized on the surface of the ion-modified layer, protecting the cutting tool from wear. This extends the stable normal wear stage and significantly increases the tool life.

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