

Research of functional properties of nitride ion-plasma coatings

O Yu Vaulina, B B Ovechkin, A V Papchenko, E V Shvagrakova

National Research Tomsk Polytechnic University, Institute of High Technology Physics, 30, Lenin ave., Tomsk, 634050, Russia

E-mail: volgay@tpu.ru

Abstract. This paper considers the influence of ion-plasma coatings with the use of nitrogen (N), zirconium nitride (ZrN), titanium-aluminum nitride (Ti,Al)N and titanium nitride and zirconium nitride by-layer (TiN+ZrN – eight layers) on the properties of steel 65X13. The main functional properties of the coatings are determined: microhardness, nanohardness, Young's modulus and corrosion resistance. It is shown that all the types of coatings allow increasing the physical and mechanical characteristics of instrument steel 65X13. Hardness and wear-resistance, depending on the type of the deposited coating, increase from 1, 5 to 4 times, corrosion resistance increases by tens times.

1. Introduction

Development of mechanical engineering determines, mainly, the possibility to create and obtain instrument materials, available in sufficiency, which correspond to the range of requirements in the modern industry and new technologies. Creation of instrument materials, having the increased wear-resistance and high strength is a topical issue.

A traditional method of increasing the cutting tools properties by alloying is suppressed currently due to the deficit of some alloying elements. Therefore, an important applied task has arisen, which is to find new modes of additional tool processing. Ion-plasma coatings on the surface of cutting tools allow increasing their wear-resistance, when being in service, broadening the area of technological capabilities and decreasing the consumption of high-priced alloying materials as well as improving the quality of the machined surface of components. Wear-resistant coatings have become an integral part of the modern instrument materials, and the majority of modern plates are provided with one or another coating [1].

The aim of the work is to increase the microhardness and wear-resistance for steel 65X13, using the method of ion-plasma saturation of the surface N, ZrN, (Ti,Al)N and TiN+ZrN (8 layers).

2. Materials and methods

The samples of the investigation were the plates with the size 10×15×1 mm. The steel surface was prepared by abrading machining and polishing. Then, in the conditions of the innovation industrial company LLC “Composite coatings” (Tomsk, Russia) the surface of the samples was saturated with nitrogen (N), zirconium nitride (ZrN), titanium-aluminum nitride (Ti,Al)N and titanium nitride and zirconium nitride by-layer (TiN+ZrN – eight layers).

Structural investigations were conducted, using a metallographic laboratory microscope LaboMet-I. Microhardness was measured with the help of a microhardness tester PTM-3M. Nanohardness was



measured with a nanoindenter MTS G200. Tests for abrasion wear were conducted at a standard laboratory setup.

Nitrided materials have high surface hardness and strength, unchangeable at heating to 500...600°C. Nitrided steel is used for the production of cutting tools and the other components, working till wear as well as at high temperatures and corrosive environment.

The coating of zirconium nitride (ZrN) keeps a good combination of properties: high corrosion resistance and a low friction coefficient. It is used in various branches of industry.

The coating (Ti, Al)N increases wear-resistance, has good resistance to corrosion wear and high heat resistance. The instrument with the coating (Ti, Al)N is used in the situations, when heavy thermal loads are needed [1, 2]. During the work the deposition of the composition has been performed in a titanium-aluminum ratio 50/50.

It is known that the most effective method to prevent cracking in the surface layers of the instrument is to use multi-layer coatings, where “hard” layers interchange with the “soft” ones. For example, a central layer is iron (steel) and aluminum layers are disposed peripherically. At moving a crack will relax stresses in a soft aluminum layer, consequently, a life span of the given composition increases. Is it possible to make a similar kind of combinations for cutting tools coatings?

Evidently, a complete analog of the combination aluminum-iron-aluminum for cutting tools is impossible as far as the extreme layers of aluminum cannot withstand high cutting temperatures, which may reach 1200°C. But there are many coating materials that allow choosing the necessary interchangeability of the properties in a broad range [3].

In the frame of the multi-layer coating research the combination (TiN+ZrN – 8 layers) is offered, which, according to authors' opinion, allows increasing the wear-resistance and corrosion wear of the instrument.

3. Results and Discussion

The research was conducted, using instrument steel 65X13. The structure in the initial condition (condition as supplied) was a granular perlite with inclusions of carbides. Average microhardness was 220...240 N/mm².

The microhardness of the samples with deposited coatings was measured at the end surface from the surface to the centre of the sample, some tracks for one coating. The curves of microhardness dependencies according to the depth of the sample are presented in Figure 1.

For all the deposited compositions it can be seen that the saturated surface layer has a higher microhardness than the centre. The microhardness near the surface along the ends of the machined samples is 300...420 N/mm², and it decreases in the distance 400...600 μm to the centre to 200...220 N/mm². Thus, microhardness of all the samples with ion-plasma coatings has increased in 1.3...2 times in comparison with the microhardness of the initial steel. The character of a transient area has a smooth junction from the reinforced layer to the matrix metal. The thickness of the modified layer in all the cases is, approximately, equal to ~ 500 μm. The TiN+ZrN (8 layers) coating has the highest microhardness.

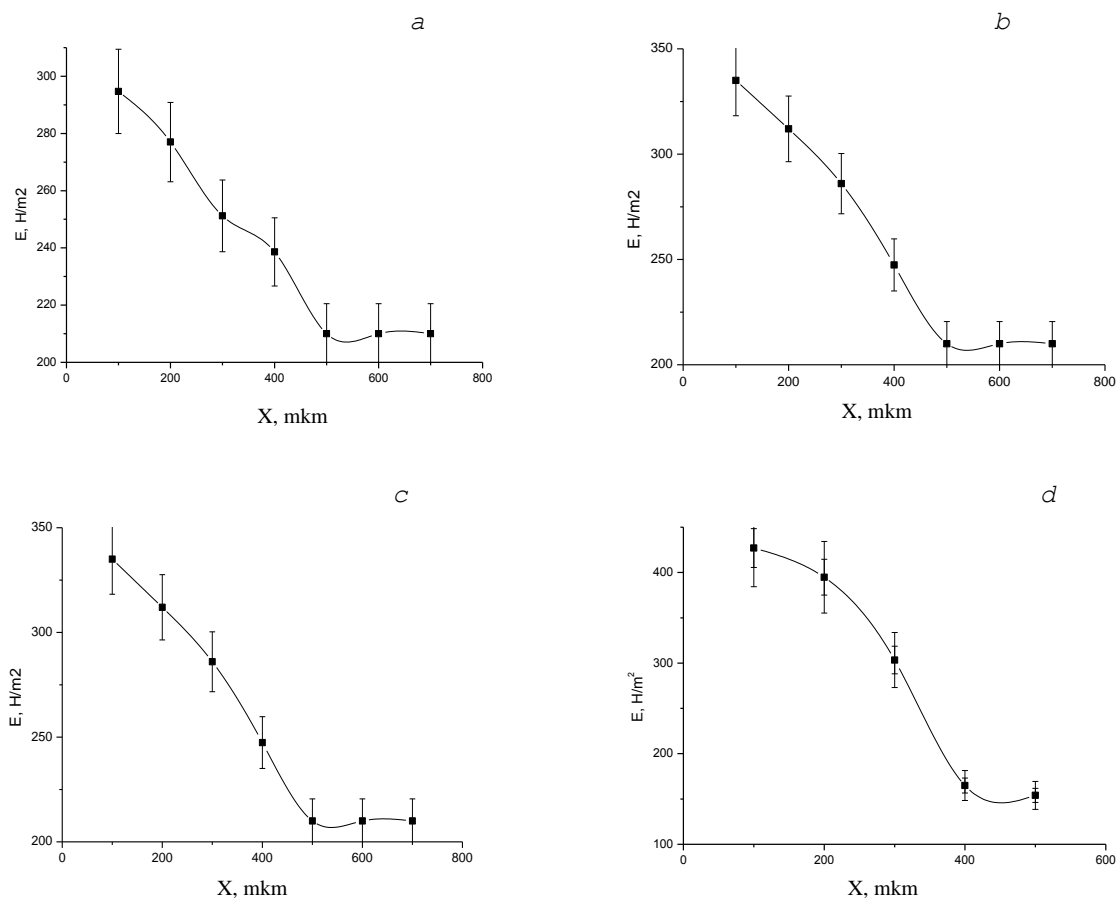


Figure 1. Microhardness dependence on the thickness of the samples with coatings: a – ZrN; b – N; c – (Ti, Al)N; d – TiN+ZrN

Hardness measuring of a thin surface layer at small loads was conducted with the help of the nanoindenter MTS G200. For each coating several measurements were made, and the curves of nanohardness dependence on the indenter penetration strength were plotted (Figure 2).

Also, the technology for nanohardness definition allows calculating Young's modulus automatically, which characterizes the material properties connected with the resistance to elastic yield. The obtained data of an average nanohardness and values for Young's modulus are presented in summary Table 1. It can be seen that the samples with the TiN+ZrN (8 layers) coating have the highest nanohardness, while the sample with the nitrated layer has the lowest one (after the initial steel).

Abrasion wear of the material in the work was defined at friction on the floated abrasive particles. Tests on wear-resistance were conducted, using the initial steel and steel 65X13 with all the types of investigated coatings. Wear tracks demonstrated the similar character for the initial steel with N-, ZrN-coatings. The surface of the tracks had a "streamy" structure; it had a metallic luster, what was typical for a brittle fracture. Wear tracks for (Ti, Al)N- and Tin+ZrN-coatings differed from the abovementioned tracks: a distinctive "cup-type" structure was revealed, the brushed character without any luster, which was usual for a ductile fracture. This fact was explained by the presence of lamellar constituents in these coatings.

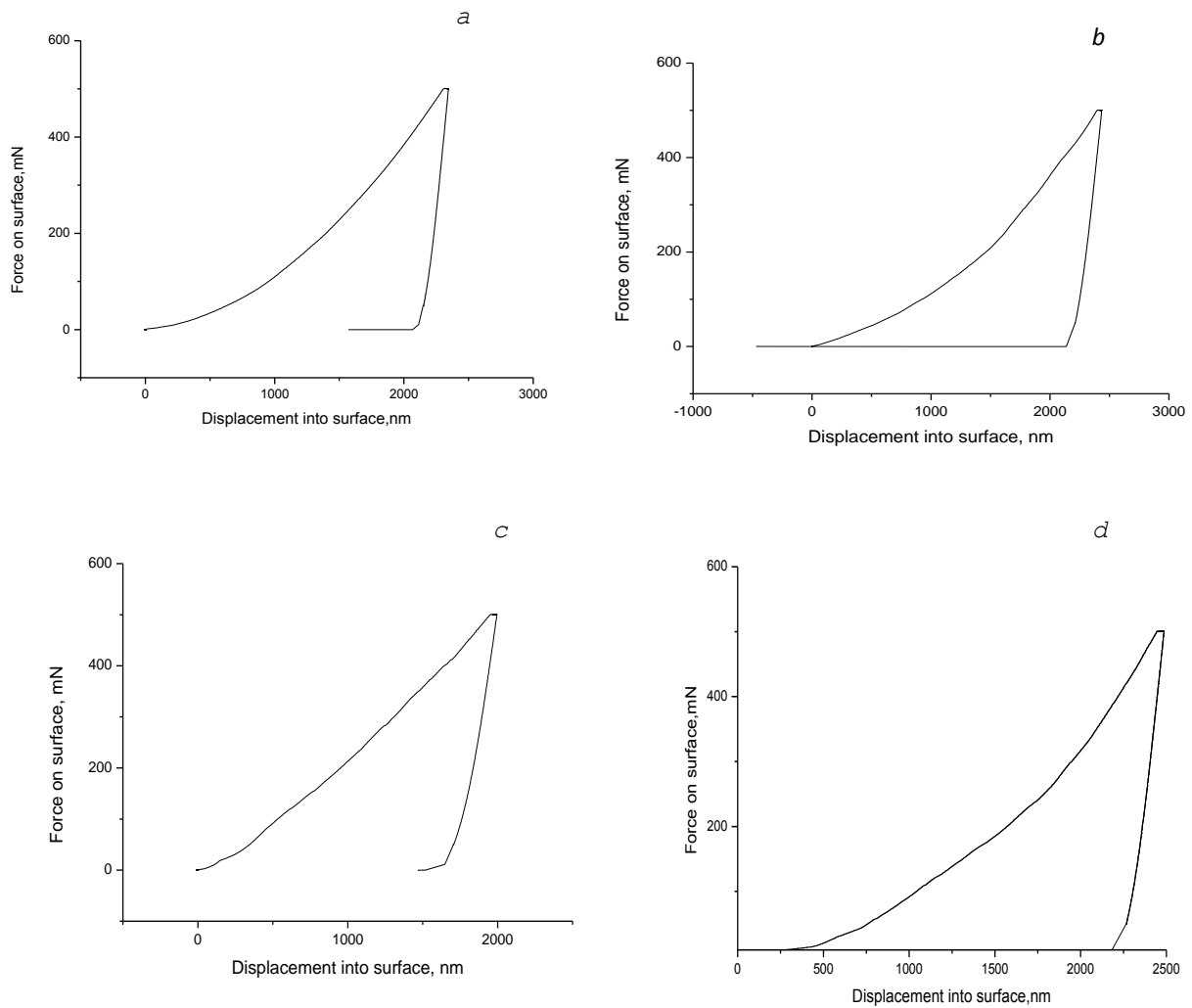


Figure 2. Nanohardness dependence on the indenter penetration strength into the sample: a – N; b – ZrN; c - (Ti, Al)N; d - TiN+ZrN

The obtained results of the tests for wear-resistance are presented as bar graphs in Fig.3 and in Table 1. It can be seen that the best results for wear-resistance a complex eight-layered TiN+ZrN coating has demonstrated.

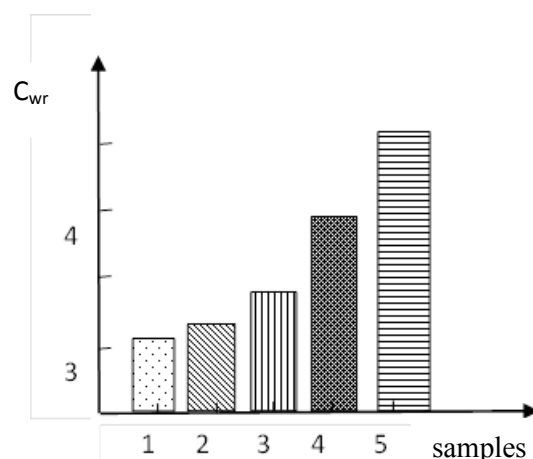


Figure 3. Wear-resistance coefficient (C_{wr}) of the investigated samples: 1 - 65X13, 2 – nitrided, 3 – ZrN, 4 - (Ti,Al)N; 5 - Tin+ZrN (8 layers)

Table 1. Summary data obtained as a result of research (E_{av} – Young’s modulus; H_{av} – average nanohardness; R_w – relative wear; h_i – relative mass change as a result of corrosion in $\Delta t_i=60\text{min}$)

Material	E_{av} , H/mm ²	H_{av} , H/mm ²	C_{wr}	h_i
65X13	232024	4066	1.1	0.33
N	232111	4352	1.3	1.92
ZrN	233927	4361	1.9	0.36
(Ti,Al)N	235723	5833	2.9	0.87
TiN+ZrN	235983	5935	4.2	0.03

Corrosion tests were carried out in the laboratory conditions in a 10%-solution of a nitric acid in water. The nitric acid is a corrosive environment for the majority of metals. The scheme of testing may be different: dipping and storage in a basin with the high humidity or dipping and storage in the atmospheric humidity. The last is the most suitable for testing the instrument steels with coatings.

Nitride coatings testing for corrosion resistance to the static effect of liquid was carried out according to GOST 9.308-85. By this method the samples were deposited in the corrosive environment for 5 minutes of each hour with the following storage in the atmospheric humidity during 50 minutes. The tests were conducted at a room temperature. At every turn we conducted the estimation of a sample mass loss per the unit of area, the mass was determined by weighing. Moreover, the external examination of samples had been performed before the first attributes of corrosion appeared. The obtained results are demonstrated in summary Table 1.

The best result of corrosion testing was shown by the TiN+ZrN coating, which for the whole time of corrosion testing did not lose the mass. The next result was the stainless steel 65X13. This steel is known by its high ability to resist influencing of various corrosive media, which include the influence of nitric acid. The samples with the coating ZrN (Table 1) showed reasonably good results, but the coating itself had disappeared after 20 minutes, and the other time only steel worked. The nitrided samples demonstrated the worst corrosion resistance in a water solution of nitric acid.

4. Conclusion

The possible methods of increasing the hardness, wear-resistance and corrosion resistance of instrument materials by means of alloying have a significant disadvantage connected with the deficit and expensiveness of alloying elements, and they have already been worked out, to a great extent. The paper shows that the application of new progressive technologies of ion-plasma coatings deposition may increase the operational characteristics of instrumental materials and obtain a significant economic effect.

The coatings N, ZrN, (Ti, Al)N and a multi-layer TiN+ZrN (8 layers) deposited on steel 65X13 by the ion-plasma method are investigated in the paper. It is observed that all the types of coatings allow increasing the physical and mechanical characteristics of instrument steel 65X13. Hardness and wear-resistance, depending on the type of the deposited coating, increase from 1.5 to 4 times, corrosion resistance in the case of a multi-layer coating increases by tens times.

The complex TiN+ZrN (8 layers) coating has demonstrated the best values of working properties. However, the other investigated coatings have showed the results, which are much higher than the initial steel 65X13 has.

Therefore, it is shown that the use of modern technologies and materials for ion-plasma coatings deposition may significantly improve the physical and mechanical characteristics of instrument steels and provide the high technical and economic indicators of machine building.

References

- [1] Tabakov V P and Chihranov A V 2010 Determination of mechanical characteristics of wear-resistant ion-plasma coatings based on titanium nitride *Proceedings of the Samara Scientific Centre of the Russian Academy of Sciences* **12** 4 292-97 (in Russian)
- [2] Brozdnichenko A N, Gorchakov S A, Ryazancev S S, Senkevich S V, Tiltse K L and Hinich I I. 2000 Influence of deposited TiN- and AlN-layers structure on the properties of a nanocomposite TiAlN coating. *Izvestia: Herzen University Journal of Humanities and Sciences* **15** 64-68 (in Russian)
- [3] Cirkin A V 2003 Development of a multi-layer coating for end cutters *Reporter of Ulyanovsk State Technical University* **3-4** 33-35 (in Russian)