Structure and phase changes in nanostructured compound coatings

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Abstract. The paper deals with an experimental study of the influence of temperature and plastic deformation on the structural-phase changes occurring at the border of the "nanostructured coating-supporting plate".

1. Introduction
It is possible to increase the wear-resistance of the cutting tool by changing the state of near surface properties, under which the most efficient cutting tool resists wear, especially at high temperatures. Moreover, the material of the tool in the body volume of the cutting tool should simultaneously have sufficient strength at compression and bending, shock loading and reversed stresses. Wide industrial employing of cutting tools with coatings allows [1–3]: to increase the resistance and reliability of tools; reduce the rate of consumption of expensive tool materials; extend the field of application and reduce the list of applied solid-alloy tools.

Today, a large number of experimental studies are carried out in the field of designing innovative multilayer multiplex coatings to improve the workability and performance properties of cutting tools with various applications, different composition, structure, architecture and properties of coatings. These tasks can be solved by employing multilayer composite nanostructured coatings. One of the most promising trends is alloying the coatings by different elements and combinations (aluminum, yttrium [1-2], etc.) considering the phenomena arising while operating.

2. Theoretical part
The reduction of particles up to 100 nm and less leads to an increase in the parameters of the crystal lattice [4]. The coatings applied to cutting tools allow increasing their wear-resistance by reducing the friction force [3], reducing deformation and cutting forces [3], changing the direction and intensity of heat flows, meanwhile the increase of wear-resistance of the tool with coatings is also explained by the screening effect of contact surfaces from external interferences [1].

At present, an increase in wear-resistance of the cutting tool can be achieved through the use of titanium and aluminum nitrides (Ti, Al)N coatings. Coatings such as (Ti, Al)N are characterized by unique combinations of properties: high hardness at high temperatures in the contact area together
with thermal and chemical resistance and low thermal conductance. An important advantage of (Ti, Al)N coatings is their tendency to oxidation and the formation of relatively stable surface oxide layers. A further increase in wear-resistance of (Ti, Al)N coatings during cutting may be achieved by bringing the grain refining to the nanoscale level (size of a grain less than 100 nm). This can be realized by alloying the (Ti, Al)N coatings on the tool material by means of magnetic-arc filtration (MAF).

3. Experimental studies

Studies [5-7] testify that the main influence on the hardness, elastic modulus and other physical and chemical properties of the coating is brought by parameters such as a grain size, constituents of the coating components, temperature of the supporting plate, ways of applying and possibility to form an amorphous phase from nitrides.

For experimental research, fast-changing multifaceted plates from hard-alloy titanium-cobalt compounds HF10, preliminary subjected to nitriding (for better adhesion of the coating and supporting plate) and having the (Ti, Al)N coating applied on them, were used. The coating thickness was measured by a Calotest device. The coating thickness was equal to $h_{\text{coating}} = 6.124 \mu m$.

The coating (Ti, Al)N was applied through filtration by a separator at a modified unit, such as NNV 6.6 – I1. The parameters of applying the coating with MAF are as follows: the arc current is 200 A, the nitrogen pressure is $5 \times 10^{-3} \text{Pa}$, the time is 30 minutes, the coil current is 20 A, the separator voltage is 16 V, the temperature of application is 340-370 °C. Heating of the supporting plate to the temperature of the coating application was performed by argon ions during their landing at a grid bias voltage of the supporting plate of 1 kV.

Nanocrystal coatings facilitate the formation of a protective layer of aluminum oxide on the surface. Coatings with a small grain structure have a greater length of grain boundaries. In this connection, a longer use of atoms is necessary for the outer diffusion of aluminum Al and inner diffusion of oxygen.

To study the chemical composition, the specimens were produced in the form of angle laps at an angle 5° to the upper part of the cutting plates. Before conducting the research, the patterns were skinned of the fat, then placed into the preparation chamber of the spectrometer and subjected to 5-minute pickling in the medium of argon ions. The pickling was performed under a gas pressure of $10^{-4} \text{Pa}$ and with a velocity of pickling of approximately 20 monolayers per minute. Such preparation of samples excluded the impact of accidental pollution on the results of analysis. The surfaces of the samples were studied at different depths, starting from the surface: either by stationary electron and ionic beams, or by scanning them in a chosen direction.

A set of samples was annealed at 700 °C under a pressure of 0.5 hPa for 20 minutes in ambient conditions to determine the possible impact of the atmosphere on the cutting tool.

These samples allowed us to study the heterogeneity of the composition at a shallow depth under the surface, including the developed films. In connection with this, the width of the studied area is increased by 9.57 times, i.e. $h_{\text{res}} = 9.57 \times h_{\text{coating}} = 58.61 \mu m$.

At the next stage of the experimental study, micro-hardness was measured according to the GOST 9450-76 standard on a Struers Duramin microhardness tester with the load of 5 H and a holding time of 10 s.

The values of microhardness were obtained at equidistance from the edge of the coating on an oblique loop. 12 values in the area of the coating and 12 more at the same distance were performed in the basis. According to the obtained microhardness values, graphs of the dependence $HV = f(h_{\text{res}})$ were plotted.

It is possible to see in the graph that a jump in hardness takes place on the border of the coating-supporting plate on the original sample. We can suppose that it comes from an increased concentration of nitrogen. Overviewing the graph of the sample hardness after annealing under pressure we may see the toughness annealing, however, therewith these values are higher than the hardness of the supporting plate. So as penetration into the base increases, the hardness value reaches the base hardness. This is apparently connected with structural changes occurring on the supporting plate.
At the next stage of the study, the angle laps were examined by a Tescan Vega3 scanning electron microscope at an accelerating stress of 20 kV and magnification by 5000 and 20000 times. Images were obtained with the help of a BSE detector (BSE – backscattered electrons), designed for obtaining images which contain information on variations in composition depending on the opposition in the charge number. Figures 3, 4 represent images of the original sample and the annealed one.

4. Conclusions
On the basis of the conducted research it is possible to make the following conclusions:
• an increase in the microhardness in the original sample on the “border” “coating-supporting plate” is probably connected with a high nitrogen content obtained by the nitrogenation of the surface before coating (figure 1);
• the release of stresses leads to leveling the coating properties in near-surface layers of the tool thereby increasing the workability of the cutting tool happens (figure 2).
• the change in microhardness occurs due to the deliberate implementation of the structural elements of the coating into the cobalt bond, which leads to an increase in the average value of microhardness in near surface layers (figures 3, 4).
Figure 3. The specimen (20 kV, BSE, 5000-fold increase): a – initial product; b – final product

Figure 4. The specimen (20 kV, BSE, 20000-fold increase): a – initial product; b – final product

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