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## Ion nitriding and surface alloying of H13A hard alloy cutting tools

### ABSTRACT

*Ion-plasma coatings are widely used to increase the wear resistance of hard alloy metalworking tools. However, plasma coatings are practically not combined with other technological operations and do not improve the physical and mechanical properties of tool working elements. Hardening tools made of hard alloys is one of the promising directions that is created by layered structures on the surface. It provides a gradient distribution of physical and chemical properties between the wear-resistant coating and the base material.*

**Keywords:** cutting tool, carbide insert, zirconium, niobium alloy, electron-beam alloying, nitrided, tool failure.

### 1. INTRODUCTION

Modern automated machine-building production requires introduction of innovative technologies that can significantly increase the productivity of machining. Due to the current system the cutting tool is one of the crucial point. Research and industrial experience show that the largest share of failures in metalworking technological systems is associated by cutting tool failures that are catastrophic wear and breakage. Since tool failure usually occurs much earlier than the wear of parts and assemblies of technological equipment (machines, fixtures, etc.), it is precisely because of the tool. If there is no preventive replacement performance, the whole technological system will surely fail. When processing such alloys, due to the peculiarities of their physical and mechanical properties including cutting conditions, a significant amount of heat is released. The temperature level between contact zone of the tool and the workpiece increases sharply, that contributes to the activation of adhesion and diffusion processes. Additionally, it intensifies the wear of the working surfaces of the tools. As a result, the use of hard alloys as a tool material is not always possible, and the use of traditional high-speed tools is justified only at low cutting speeds [1].

The use of modern high-speed steels with better heat resistance, obtained by powder metallurgy, partially solves the problem of processing intensification. However, the introduction of new structural materials with increased heat resistance inevitably puts forward additional requirements to the tool material.

Today, tools are made of high-speed steels that includes various types of wear-resistant coatings based on nitrides of refractory metals. They are obtained by the method of physical deposition of a substance that are widely used. These coatings have high microhardness, low coefficient of friction, and inertness with respect to the material being processed [2,3]. However, the practice of operating a high-speed tool with a coating shows that the efficiency of such a tool is not the same in various technological operations of cutting. The successful implementation of wear-resistant coatings is hampered by the fact that, due to the large difference in physical and mechanical properties between the substrate and the coating. The high load intensively destruct the working surfaces of the tool that can be observed during plastic deformation. This disadvantage can be overcome by forming a certain transition layer. It can be obtained by chemical-thermal treatment preceding by applying a wear-resistant coating like ion nitriding that is widely used [4]. This process is called combined ion-plasma treatment. Use of it has created possibility to increase the durability of a high-speed tool by several times compared to a tool with a PVD coating [5,6].

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Nevertheless, it should be noted that the formed iron nitrides in the process of nitriding are not thermally stable enough. In order to prevent their thermochemical dissociation, it is necessary to limit the temperature when applying a wear-resistant coating. It is possible to increase the thermal stability of the near-surface layer of the tool directly adjacent to the coating by additional surface alloying of the nitrided hard alloy.

## 2. EXPERIMENTAL PART

The process task is based on the reliable integration into the surface layer of the tool of synthesized nitride compounds between metals of groups IV-V and nitrogen that is introduced into steel during preliminary chemical-thermal treatment. The metal is deposited in the form of a coating, for example, using magnetron sputtering. Then, an exothermic chemical reaction is initiated, carried out in the thermal explosion mode, by pulsed heating of the product surface [7]. In this case, it is possible to use a fairly large range of substances that are chemically active at high temperatures as reagents [5]. At the same time, other substances can be used as fillers or diluents, including those taking part in the synthesis as by-products of the reaction. The current process is not involved use of chemical nature reagents as the magnitude of the thermal effect, the conditions of heat transfer, phase kinetics or structural transformations.

In this article, we consider some compositions that are interesting from our point of view. The modified surface layers can actually be obtained by the considered method that increase the tool life. The low-alloyed H13A (ISO: SPGN 120308) hard alloy of medium heat resistance, in our case, during longitudinal turning of a heat-resistant nickel alloy KhN77TYuR (NiCr20TiAl).

### 2.1. Carbide cutting tool

As a cutting tool for the turning operation, replaceable cutting inserts (inserts) of a special design with various options for combined surface ion-plasma processing were used (Figure 1).



Figure 1. Hardened cutting inserts made of H13A alloy (ISO: SPGN 120308)

Slika 1. Kaljeni rezni umeci od legure H13A (ISO: SPGN 120308)

The plates were made from H13A hard alloy and subjected to standard heat treatment in salt baths.

The final wear-resistant coating nATCRo3 with microhardness HV50 = 345 MPa was applied on a Plaitit π311 machine. This coating is a combination of (CrTi)N adhesive layer, (TiAl)N gradient coating and (nc-AlTiCrN/a-Si3N4) multilayer nano composite coating. A two-phase nanostructured coating layer is located with an AlTiCrN grain size of up to 5 nm, at the amorphous Si<sub>3</sub>N<sub>4</sub> phase boundaries. It restrains the coagulation of grains of the main phase, both during coating deposition and during tool operation. Interfacial boundaries, that are a zone of intense energy dissipation, deviate the resulting cracks from the direction of propagation, partially or completely slow them down [2].

Some of the samples before applying the wear-resistant coating were subjected to the operation of alloying the near-surface layer. Processing was carried out in the RITM-SP facility, which is a combination of a source of low-energy in high-current electron beams (NSEB) RITM, and two magnetron sputtering systems in a single vacuum chamber. The installation allows the deposition of films on the surface of the desired product and subsequent liquid-phase mixing of the film and substrate materials by an intense pulsed electron beam [6,7]. The generation of NSEP includes the emission of electrons, the formation of a beam in a plasma-filled diode, and its transport in a plasma channel. The use of such a generation scheme makes it possible to obtain a beam of microsecond (about 5 μs) duration with a current density of up to 105 A/cm<sup>2</sup> at an accelerating voltage of 15 to 30 kV. In this case, the area of one-time processing is about 50 cm<sup>2</sup>.

The deposition of a thin layer of nitride-forming elements on the tool surface is possible (we used targets made of Zr and Nb70Hf22Ti8 alloy) before electron beam treatment that obtains multiphase structure. The outer layer is enriched with MN type refractory nitride phases. Due to the extremely high cooling rate, the nitride remains as a fine and homogeneously distributed in the final product. The surface layer is from 2 to 10 μm in modified structure, depending on the alloying composition.

### 2.2. Microalloying of pre-nitrided hard alloy with zirconium and niobium alloy

Figure 2a demonstrates the effect of a series of NSEB pulses with an energy density of 4.5 J/cm<sup>2</sup> and a duration of 5 μs on the surface of a nitrided sample which is made of N13A alloy. The thermal effect of the electron beam is sufficient for the upper layer of metal not only to melt, but for beginning of active evaporation that expose the carbide component. Irradiation with NSEB causes dissociation of iron nitrides, especially ε-phase, and a large amount of retained austenite is formed on the surface (Figure 3b).

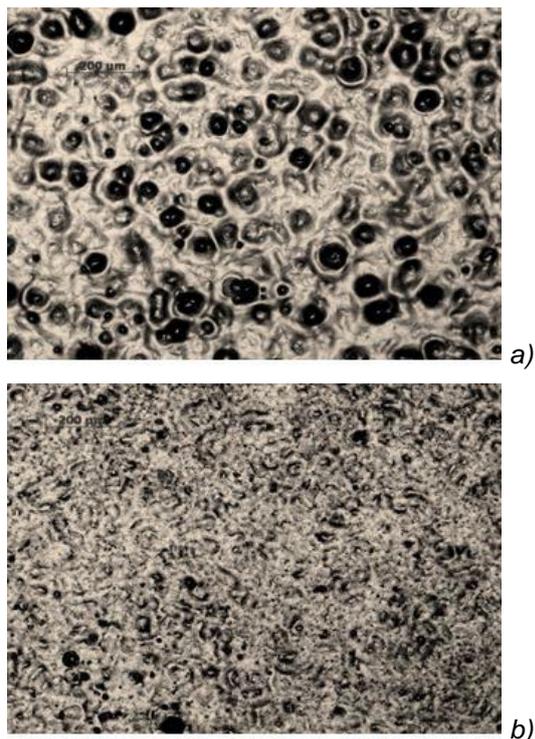


Figure 2. a) Structure of the surface of nitrated H13A hard alloy after exposure to NESP, b) The same after electron-beam alloying with zirconium

Slika 2. a) Struktura površine nitrirane tvrde legure H13A nakon izlaganja NESP, b) Ista nakon legiranja cirkonijumom elektronskim snopom

After depositing a thin (about 0.2  $\mu\text{m}$  thick) film (in this case, Zr) onto the samples using a magnetron sputterer and subsequent exposure to an electron beam, it is possible to initiate exothermic chemical reactions of the formation of the nitride phase. Due to the formation of a refractory nitride film on the surface, the evaporation of the metal is significantly reduced, and the structure becomes finely dispersed (Figure 2b). The formation of the nitride phase is confirmed by the data of X-ray diffraction analysis (Figure 3b). It should be noted that in the latter case, the content of retained austenite in the surface layer is noticeably lower.

At the same time, in our case, we are dealing with strain hardening caused by the passage of an elastic wave, which is generated under pulsed electron beam action. But due to the short duration of the process and the thermal inertia of the metal, compression and internal friction process happens, most likely, it will not be a physical factor that determines the behavior of the substance under such conditions. The main role in this case should be played by the mechanical activation of fast physical and chemical processes in the substance, which inevitably take place both in liquid and solid phases. The appearance of a melt leads to a sharp increase in the interfacial surface and an increase in the rate of the nitride formation reaction due to the additional release of energy during the exothermic reaction [5].

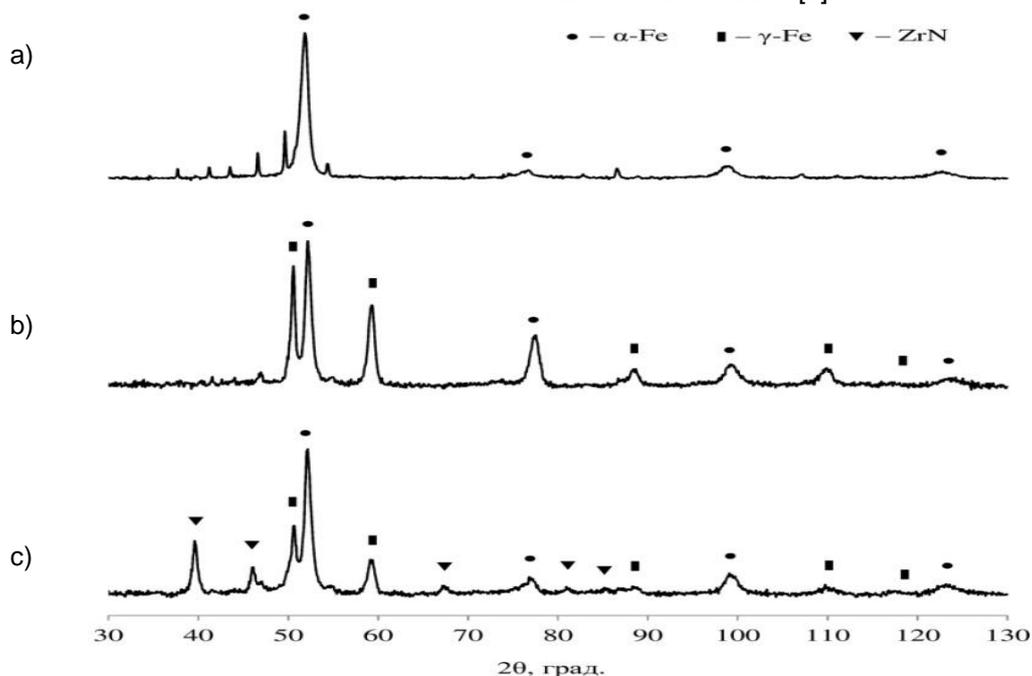


Figure 3. a) X-ray diffraction pattern (CoK $\alpha$ ) from the surface of a sample of nitrated H13A alloy, b) the same after exposure to NESP, c) the same after applying a Zr film to the surface before irradiation

Slika 3. a) Difrakciona slika rendgenskih zraka (CoK $\alpha$ ) sa površine uzorka nitrirane legure H13A, b) ista nakon izlaganja NESP, c) ista nakon nanošenja Zr filma na površini pre otvaranja.

### 3. RESULTS AND DISCUSSION

Multiple initiation of the process practically does not change the initial microstructure. In order

to finish the transformation completely, as a rule, a series of five or six NESP pulses is sufficient.

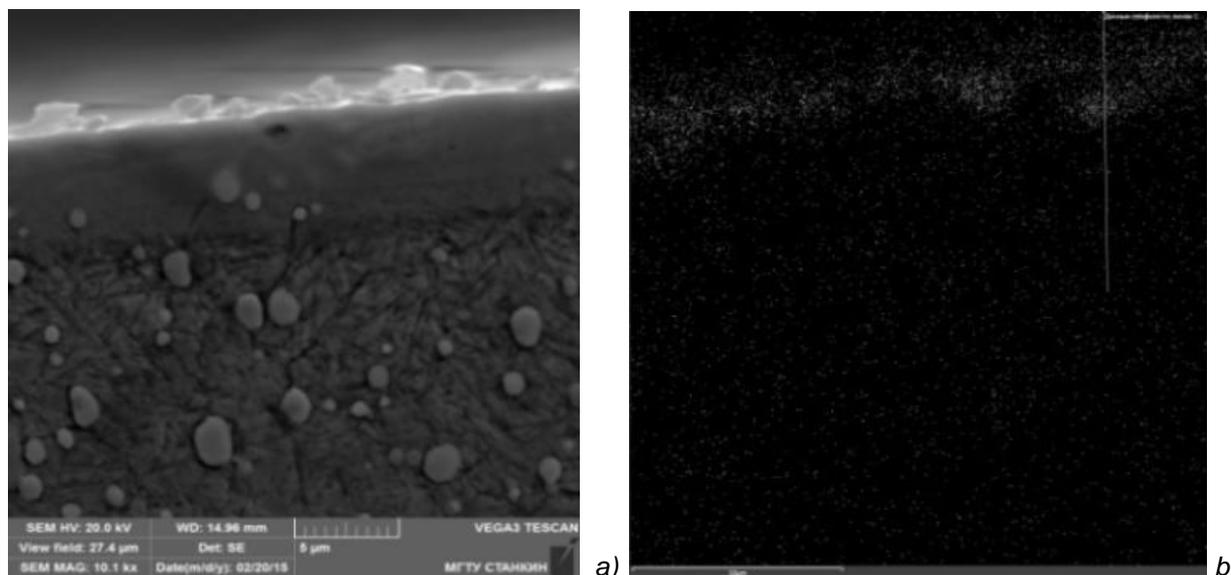


Figure 4. Surface layer of H13A hard alloy modified with zirconium nitride. a) SEM image of the etched section, b) map of zirconium distribution over the section

Slika 4. Površinski sloj od tvrde legure H13A modifikovane cirkonijum nitridom. a) SEM slika urezanog preseka, b) mapa distribucije cirkonijuma po preseku

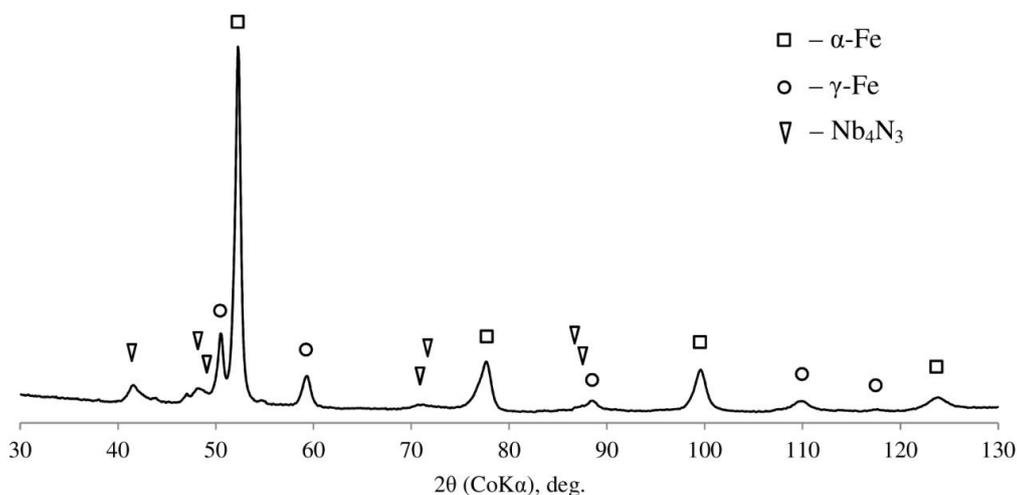


Figure 5. X-ray diffraction pattern (CoK $\alpha$ ) from the surface of a sample of nitrided H13A alloy after applying NbHfTi alloy film to the surface before irradiation

Slika 5. Difrakcioni uzorak rendgenskih zraka (CoK $\alpha$ ) sa površine uzorka nitrirane legure H13A nakon nanošenja filma od legure NbHfTi na površinu pre zračenja

The distribution of zirconium near the surface is shown in Figure 4b. It can be seen from the Figure 4 that Zr is detected in a near-surface layer with about 2  $\mu\text{m}$  thickness. But the thickness of the zone of influence of processing by a pulsed electron beam is 5-6  $\mu\text{m}$  (Figure 4a). The large input energy density and short interaction time give reason to expect the formation of rapidly quenched

layers with a more uniform and finely dispersed structure and an improvement in the service characteristics of the near-surface layer due to secondary hardening with the formation of a martensitic-carbide structure in high hardness. In addition, during the subsequent deposition of a wear resistant coating, the tool is subjected to at least two hours of tempering at a temperature

about 450°C, which reduces the residual austenite content in the modified layer and facilitates the removal of residual stresses.

A similar picture can be observed upon irradiation of a thin film of a niobium-hafnium alloy deposited on a metal surface. The diffraction

pattern (Figure 5) shows reflections corresponding to the Nb<sub>4</sub>N<sub>3</sub>(γ) phase (t116, a = 0.4385 nm, c = 0.4320 nm) based on niobium. The lattice periods of the nitride agree well with the reference data; apparently, other elements dissolved in the nitride do not significantly affect its crystal structure.

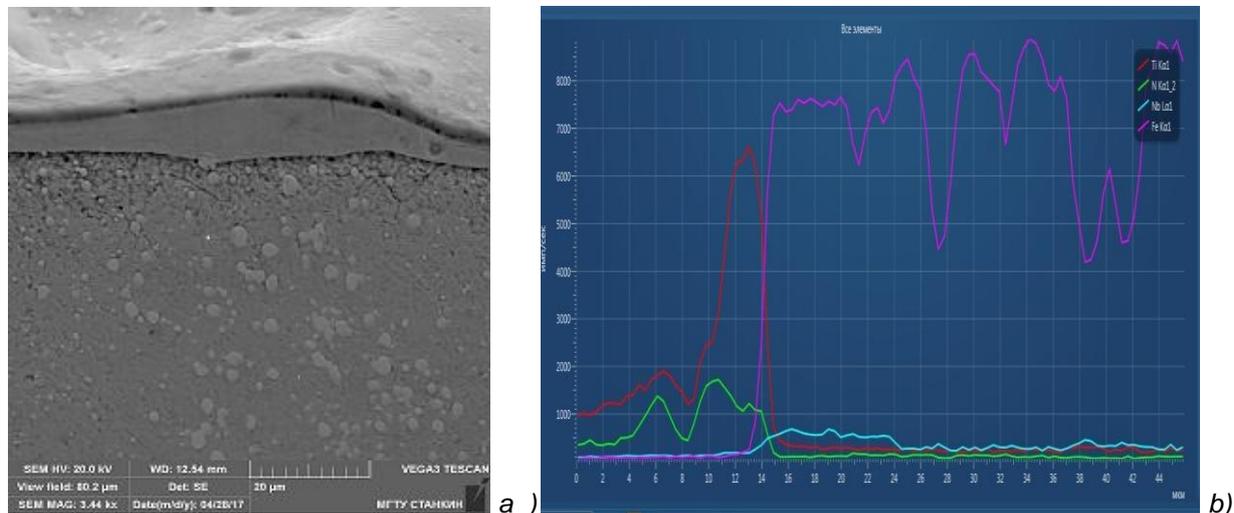


Figure 6. Sample from H13A alloy after combined surface treatment: ion nitriding + microalloying of Nb70Hf22Ti8 alloys + wear-resistant coating, a) SEM image, b) distribution of some chemical elements in the near-surface layer

Slika 6. Uzorak legure H13A nakon kombinovane površinske obrade: jonsko nitriranje + mikrolegiranje legura Nb70Hf22Ti8 + premaz otporan na habanje, a) SEM slika, b) distribucija nekih hemijskih elemenata u prizemnom sloju

Figure 6 shows the SEM image of a section from a sample of H13A alloy after microalloying with a niobium-hafnium alloy, on which a wear-resistant coating was applied. In contrast to the case of doping with zirconium, niobium and hafnium are distributed over the entire depth of the near-surface layer modified by an electron beam to

a depth of 10 μm (Figure 6b), which can be attributed to the fact that the mixing of metals in the melting zone. But in the case of a niobium alloy, it occurs at a higher temperature. In our case, it will apparently be limited by the evaporation temperature.

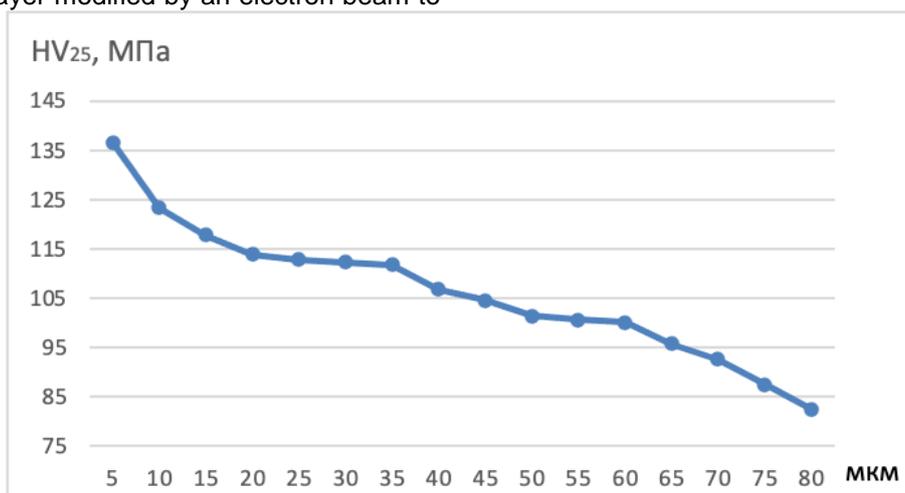


Figure 7 Change in microhardness with respect to the depth of the hardened zone on a transverse section from a sample of H13A alloy after combined processing.

Slika 7. Promena mikrotvrdoće u odnosu na dubinu očvrstne zone na poprečnom preseku od uzorka legure H13A posle kombinovane obrade

Measurement of microhardness on a transverse section shows the presence of a hardened zone up to 80  $\mu\text{m}$  deep (Figure 7). At the same time, at a depth of up to 50  $\mu\text{m}$ , the microhardness of HV25 exceeds the microhardness of the base by at least 15 MPa and it is about 100 MPa. The increase in microhardness can be associated with the influence of residual tensile stresses formed during pulsed heating. The influence of complex surface treatment on the cutting ability of the tool

The studies on the wear of H13A alloy plates showed that electron beam alloying in combination with the operation of applying a wear-resistant coating can have a significant effect on the tool wear process. Durability tests were carried out on the operation of turning a heat-resistant alloy at a cutting speed  $v=100$  m/min, feed  $s=0.5$  mm/rev, depth of cut  $t=1.5$  mm. As a failure criterion, the amount of wear along the flank surface of 0.3 mm was chosen. The test results are shown in Figure 8.

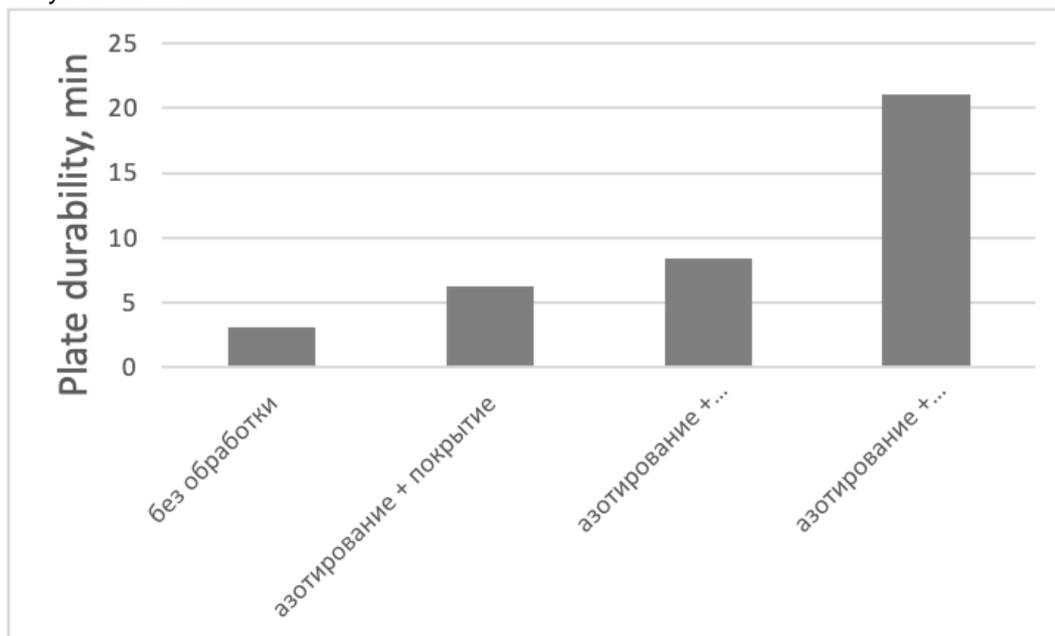


Figure 8. Tool life of inserts with combined surface treatment (min).

Slika 8. Vek trajanja alata umetaka sa kombinovanom obradom površine (min).

When unprocessed tool is cutting, the characteristic place of wear occurs on the top of the insert (Figure 9).

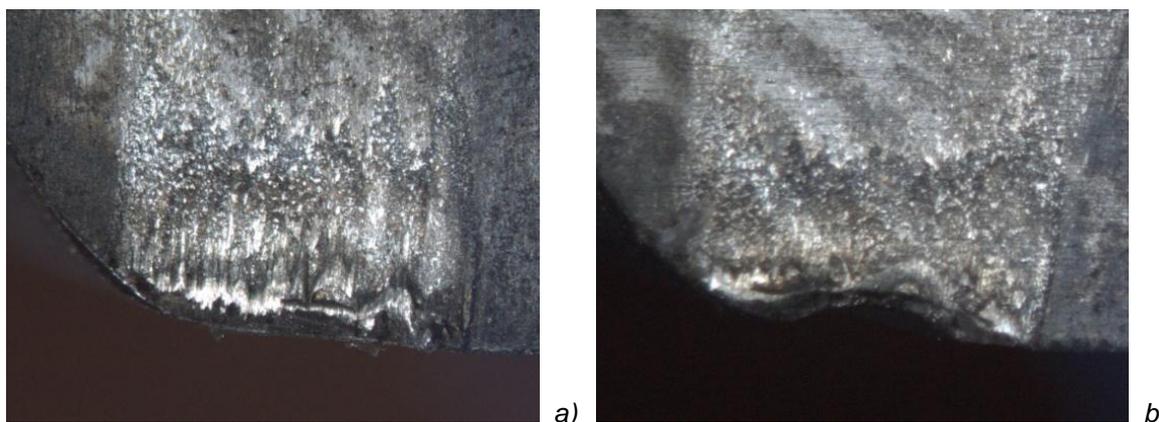


Figure 9. Wear of an H13A alloy plate during turning,  $v = 100$  m/min,  $s = 0.5$  mm/rev,  $t = 1.5$  mm, a) after 1 min, b) after 3 min of cutting.

Slika 9. Habanje ploče od legure H13A pri struganju,  $v = 100$  m/min,  $s = 0,5$  mm/obr.,  $t = 1,5$  mm, a) posle 1 min, b) posle 3 min sečenja.

It is known that a gradual increase in temperature in the zone of direct contact leads to catastrophic wear along the flank surface, which eventually reaches values at irreversible processes in the hard alloy begin [8,9]. The delayed flank wear on the combination tool is ion nitriding to a depth of about 40  $\mu\text{m}$ . Subsequent deposition of a wear-resistant coating can be explained by the fact

that the near-surface layer created under the coating has increased hardness combined with higher heat resistance and better resists microplastic deformations (Figure 10). The shape stability of the cutting edge is increased, that reduces the level of internal stresses in the wear-resistant coating. This, apparently, slows down the processes of softening near the rear surface.

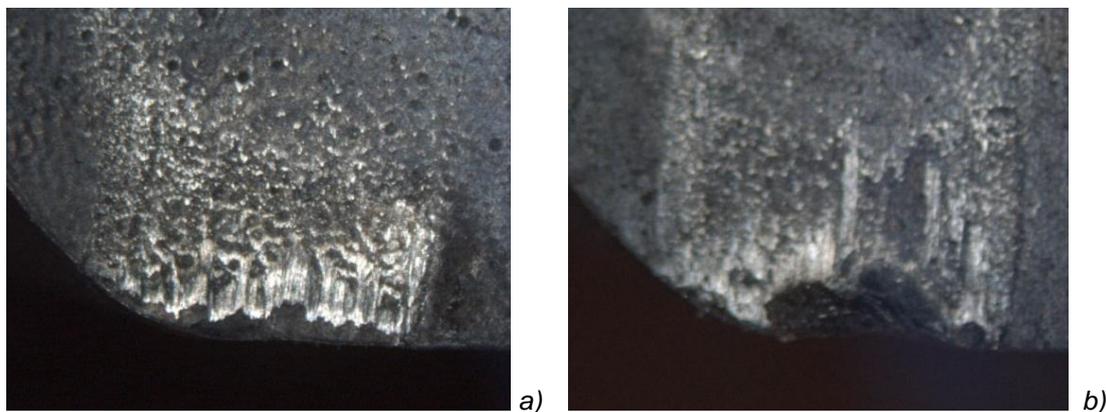


Figure 10. Wear of an H13A alloy plate,  $v = 100 \text{ m/min}$ ,  $s = 0.5 \text{ mm/rev}$ ,  $t = 1.5 \text{ mm}$ , a) after 3 minutes, b) after 6 minutes of cutting.

Slika 10. Habanje ploče od legure H13A,  $v = 100 \text{ m/min}$ ,  $s = 0,5 \text{ mm/obr.}$ ,  $t = 1,5 \text{ mm}$ , a) posle 3 minuta, b) posle 6 minuta sečenja.

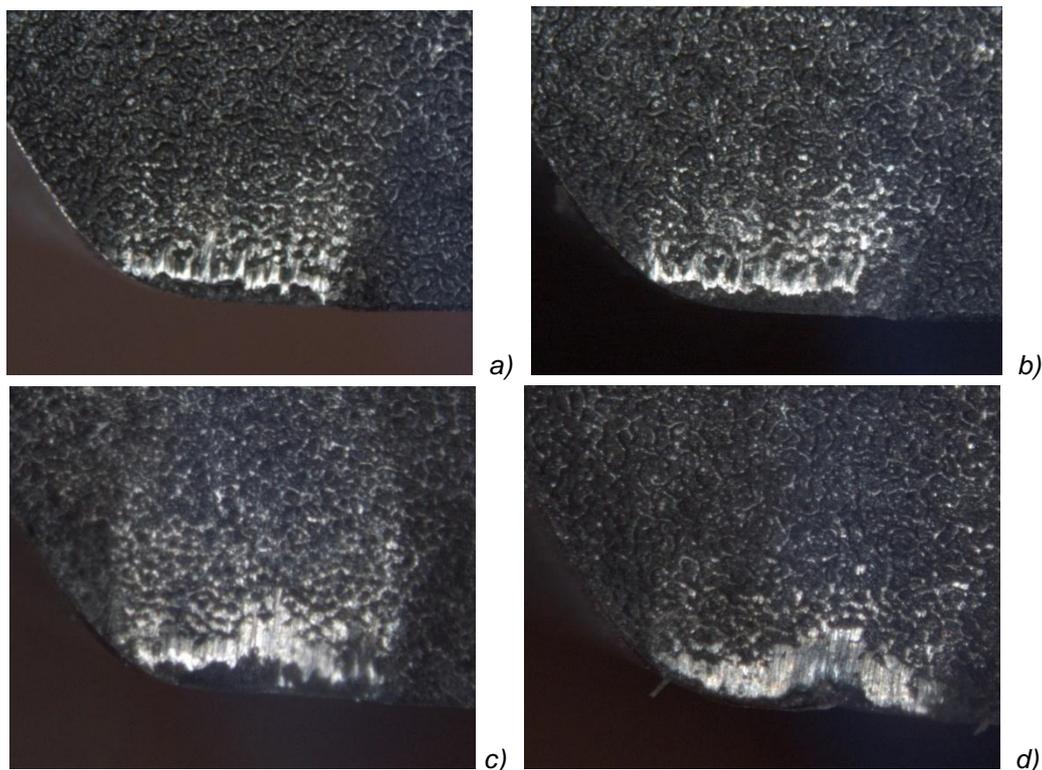


Figure 11. Wear of H13A alloy plate (nitriding + microalloying with Nb70Hf22Ti8 alloy + coating),  $v = 100 \text{ m/min}$ ,  $s = 0.5 \text{ mm/rev}$ ,  $t = 1.5 \text{ mm}$ , a) after 6 min., b) after 10 min. c) after 15 min. d) after 21 minutes of cutting

Slika 11. Habanje ploče od legure H13A (nitriranje + mikrolegiranje sa legurom Nb70Hf22Ti8 + premaz),  $v = 100 \text{ m/min}$ ,  $s = 0,5 \text{ mm/obr.}$ ,  $t = 1,5 \text{ mm}$ , a) posle 6 min., b) posle 10 min. c) posle 15 min. d) nakon 21 minuta sečenja

For tools with a wear-resistant coating and pre-treatment, including ion nitriding and surface alloying, there is a noticeable blocking of wear development at the tip, which significantly slows down the onset of catastrophic wear (Figure 11). This can be attributed to the fact that the near-surface modified layer is chemically passive and reduces adhesive interaction with the material being processed. Zr, Ti, Nb and Hf nitrides form stable and strong oxides. As a result, the characteristics of contact processes change, that significantly reduces the power of the source of heat release near the cutting wedge of the tool.

With a coated tool without microalloying, after the base is exposed, the friction conditions on the flank surface are increasingly approaching those that are characteristic of an uncoated tool. For a tool with complex machining, even after a breakthrough of the coating, the modified layer continues to perform its protective functions, which is reflected in the tool wear pattern. The complex machining significantly inhibits the formation of a wear hole on the rake face.

It should be noted that microalloying of the alloy with a niobium-hafnium alloy gave a much greater effect than microalloying with zirconium. This confirms that the hardening of the alloy in our case was achieved to a greater extent due to the alloying of the surface layer, in particular, with niobium and hafnium nitrides, and not only due to pulsed surface electron beam hardening of the hard alloy tool.

It should be noted that the processing time of the tool with surface alloying is about 15 minutes without taking into account the time of vacuuming the working chamber. This determines the low cost of the process and allows for a full load of an industrial machine for applying medium-sized wear-resistant coatings with a duty cycle of 4 to 5 hours using a single surface alloying plant.

#### 4. CONCLUSION

Obtained experimental results indicate following possibilities.

Firstly, the layer of hard alloy tool is obtained by modified surface alloying. This process is achieved by initiation of exothermic chemical reactions between the pre-nitrided base and the thin film deposited on it. At the same time, the formation of new phase components was found in the products.

Secondly, the formation of a structure in the near-surface layer of the material is provided by a pulsed nature that acts in the microsecond range. Dependence of the modified layer thickness on the

accelerating voltage is markedly extreme. Irradiation with insufficient energy in the beam is unable to initiate the process and its excess leads to the evaporation of most of the film. The treatment should be carried out in such a way that the coating thickness is approximately half of the electron penetration depth into the substrate material, that is, in our case, about 200 nm.

And the third, tool life of H13A (ISO: SPGN 120308) insert increased with combined machining, according to the power cut criterion when cutting a heat-resistant alloy. If we compare with the original plates without treatment, it was up to 100% wear. However, in our case it was decreased 25% wear of tool and it showed up to 75% wear, comparing to plates with only a wear-resistant coating. A significant increase in flank tool life was about 400-500% when compared with inserts without treatment, and up to 40% when compared inserts with only a wear-resistant coating.

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## IZVOD

### JONSKO AZOTIRANJE I POVRŠINSKO LEGIRANJE ALATA ZA REZANJE TVRDE LEGURE H13A

*Jonsko-plazma premazi se široko koriste za povećanje otpornosti na habanje alata za obradu metala od tvrdih legura. Međutim, plazma premazi se praktično ne kombinuju sa drugim tehnološkim operacijama i ne poboljšavaju fizičko-mehanička svojstva radnih elemenata alata. Kaljenje alata od tvrdih legura je jedan od perspektivnih pravaca koji se stvara slojevitim strukturama na površini. Obezbeđuje gradijentnu distribuciju fizičkih i hemijskih svojstava između premaza otpornog na habanje i osnovnog materijala.*

**Ključne reči:** Rezni alat, karbidni umetak, cirkonijum, legura niobijuma, legiranje elektronskim snopom

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