Complex modification of the surface of high-speed steel in low-temperature high-density plasma

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Abstract. A technological process has been developed for complex modification of the surface of tool materials in low-pressure discharges, including low-temperature nitriding and subsequent deposition of multilayer coatings based on intermetallic compounds of the Ti-Al system. The results of production tests of metal cutting tools processed by the developed complex technology are presented.

1. Introduction

Today, the aircraft engine industry is actively increasing the requirements for structural materials in order to improve the basic characteristics of the engine: increased thrust, increased efficiency, reduced fuel consumption. In this connection, the working temperature in gas turbine engines increases and hard-to-use heat-resistant alloys are increasingly used. The low machinability of these alloys is determined by their physico-mechanical properties [1–4]. Given this trend, the requirements for metal-cutting tools are also increasing (cutters, cutters, slotting tools, etc.).

To solve the problem of increasing the durability of metal-cutting tools used in the aviation industry from high-speed steels and hard alloys, a method for complex surface modification in low-temperature high-density plasma has been proposed, including ion nitriding and subsequent formation of multilayer nanostructured coatings based on intermetallic compounds of the Ti-Al system synthesized in reactive gases O, C, N environments. Studies of the effect of low-temperature nitriding and subsequent formation multilayer coatings based intermetallic Ti-Al system to change the microhardness of the surface layer and the wear resistance of tool materials [5–7].

The aim of this study is to investigate the influence of ion nitriding on the adhesive properties of TiAl(O,C,N) coatings.

2. Methods of conducting experiments

The investigated material was instrumental high-speed steel R6M5. The chemical composition of the metal is given in table 1.

| C   | Si  | Mn | Ni | S | P     | Cr   | Mo   | W   | V   | Co | Fe |
|-----|-----|----|----|---|-------|------|------|-----|-----|----|----|
| 0.82–0.9 | <0.5 | <0.5 | <0.4 | <0.03 | 3.8–4.4 | 4.8–5.3 | 5.5–6.5 | 1.7–2.1 | <0.5 | other |

TiAl(O,C,N) coatings were deposited using an NNV-6.6-I1 vacuum installation. Ion nitriding in the arc discharge was also carried out in this installation. Samples from materials of R6M5 were placed in a vacuum chamber (figure 1). The sputtering parameters changed within the following ranges: the
pressure in the chamber was \( P = 10^{-1} - 10^{-2} \text{ Pa} \); the arc current was \( I = 60 - 120 \text{ A} \); and the processing time was 60 min. The coatings were formed by the simultaneous sputtering of two single-component Al and Ti cathodes upon rotation of the table around its axis. Schematic of sample arrangement during deposition of the coating by arc plasma discharge was present in previous study. As the working gas were nitrogen and argon (50% N\(_2\) + 50% Ar).

To study the influence of the ion nitriding on the adhesive characteristics of the coatings, the samples were exposed to ion nitriding and subsequent deposition of TiAl(O,C,N) coatings. Types of technologies is given in table 2.

| Sample | Technology                        |
|--------|-----------------------------------|
| 1      | Ion nitriding (15 min.) + TiAl(O,C,N) |
| 2      | Ion nitriding (30 min.) + TiAl(O,C,N) |
| 3      | Ion nitriding (45 min.) + TiAl(O,C,N) |
| 4      | Ion nitriding (60 min.) + TiAl(O,C,N) |

To study the physico-mechanical parameters of the samples, such as adhesive resistance and wear resistance, the installation Scratch Tester by CSM Instruments was used (figure 2). The research technique is to use a diamond conical indenter with a radius of curvature of 200 \( \mu \text{m} \), which moves along the sample under study with increasing load. A computer was used to register the applied load \( (F_n, H) \), the penetration depth of the indenter \( (P_d, \mu \text{m}) \) and acoustic emission \( (AE, \text{dB}) \), which reflected the destruction, delamination or spalling of the coating. After that the load was released and the indenter moved in the opposite direction to measure the restored scratch depth \( (R_d, \mu \text{m}) \). The tests were performed in the following conditions: the indenter was increasingly loaded at 0.3–30 N; the indentation rate was 2 mm\( \cdot \text{min}^{-1} \); the scratch’s length was 5 mm; the load application rate was 11.88 N\( \cdot \text{min}^{-1} \); frequency of the discrete signal was 60 Hz, and the power of the acoustic emission was 9 dB. The minimum (critical) loads \( L_C \), at which first cracks appeared, were specified in the tests. Microhardness studies were carried out on the installation Struers Duramin (figure 3).
3. Experimental results and discussion

Table 3 shows the microhardness results of all samples before ion nitriding, followed by coating deposition and after.

| № of technology | Microhardness before ion nitriding and coating deposition HV₀.₂ | Microhardness after ion nitriding and coating deposition HV₀.₂ |
|-----------------|---------------------------------------------------------------|---------------------------------------------------------------|
| 1               | 790                                                           | 980                                                           |
| 2               | 820                                                           | 1020                                                          |
| 3               | 820                                                           | 1220                                                          |
| 4               | 830                                                           | 1050                                                          |

Figure 2. Scratch adhesion test schematic.

Figure 3. Struers Duramin.
According to the results of microhardness measurement, it can be seen that the highest results were shown by the sample with technology number 3, but other samples also showed an increase of microhardness.

Figure 4 shows the results of tests of the coating for adhesive strength. In the scratch test when the maximum load (30 N) was reached by optical microscopy no detachments and cracking of the coatings were detected, which is evidence of high adhesion of the applied coating to the substrate.

![Figure 4](image)

**Figure 4.** Results of the sclerometric tests: a) Technology No. 1; b) Technology No. 2; c) Technology No. 3; d) Technology No. 4.

The analysis of results (figure 4) showed that the penetration depth of the indenter under the maximal load equal to 30 N and after its removal was different in steels with different time of ion nitriding. As a result of scratching of samples with the multi-layer coating it was revealed that the penetration depth of the indenter under the load in case of technology No. 1 made 13 µm, in case of samples with technology № 2 was ~ 10 µm. The ion nitriding made before deposition of the coating allowed decreasing considerably the depth of the indenters penetration, and with increased the time of ion nitriding from 15 to 45 minutes penetration depth was decreases from 13 to 8 µm. But further increasing time of ion nitriding don’t decrease a penetrations depth. The results of measurements depth of the track (2–3 µm) after removal of the loading showed that neither shearing nor damage of the coating occurred under the maximal loading (figure 4).

According to the results of sclerometric tests of influence of the ion nitriding to adhesion strength, it was established, that technologies No. 3 and 4 showed the best characteristics.

4. Conclusion
The analysis of scratch tracks’ images revealed that the critical load under which microcracks start to appear in the coating was 18 N for the samples with ion nitriding for 15 minutes. Increase time of the
operation of ion nitriding prior to deposition of the multilayer coating allowed increasing the critical load up to 22 N for the samples times of 45 and 60 minutes.

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