The adhesion solidity, physico-mechanical and tribological properties of the coating of titanium nitride

L A Krivina, Yu P Tarasenko and Ya A Fel
Institute of problems of mechanical engineering of RAS, 603024, Nizhny Novgorod, Russia
E-mail: npktribonika@yandex.ru

Abstract. Influence of variable technological factors (arch current, fractional pressure of gas in the camera) on structure, physic-mechanical and tribological features of an ion-plasma coating of titanium nitride has been investigated. The adhesion solidity has been put to the test and the mechanism of destruction of a covering has been also researched by a sketch-test method. The optimal mode of spraying at which the formation of the nanostructured bar coating of TiN has been defined. The covering offers an optimal combination of physic-mechanical, tribological and solidity features.

1. Introduction
Nowadays, when abrasion of basic means of production and transport facilities in Russia reaches 80 %, the main task is to increase reliability and operability of various details of clusters and mechanisms in machine-building branch. In addition, increase of reliability and resource of further developed technique is also relevant.

In spite of the fact that thin coverings on the basis of refractory nitrides of the transitional metals are rather well studied and are widely used in many areas of human activity, scientific and practical interest in them does not fade away for the specific physic-mechanical properties – high hardness, a low friction coefficient, endurance, resistance to corrosion, etc. [1, 2].

2. Experimental technique
The ion-plasma coating of TiN was applied on the VU-2MBS installation on small-size thin-walled exemplars of steel on the modes given in the table 1.

| Technological parameters of spraying | Fractional pressure of gas in the building bag $P$, Pa |
|-------------------------------------|-----------------|
| Arch current $I$, A                | 0.04  | 0.06  |
|                                     | 160   | 160   |
|                                     | 140   | 140   |
|                                     | 120   | 120   |

The spraying of coating was realized in definite periods of time of processing in one production cycle: cleaning and activation of a surface of details with method of ionic bombing Ti⁺ in a periodic mode:
5 s (processing) + 5 s (pause), etc.; drawing an underlayer α-Ti – 10 min; TiN deposition – 50 min.

The microstructure of the coating of TiN was studied with a submicroscopy method on a raster microscope of VEGA/TESKAN.

Microhardness \( (H_c) \) of compositions “covering + basis” was measured on PMH-3 microhardness, theoretical calculations of the true microhardness of a coating were made according to the formula [3] that takes into account the influence of a substrate on measurement process:

\[
H_c = H_{\text{com}} + \frac{(H_{\text{com}} - H_b)\left[(2t/h + 3(t/h)^2)\right]}{1},
\]

where \( H_c \) – a microhardness of a coating; \( H_{\text{com}} \) – a composition microhardness; \( H_b \) – a substrate microhardness; \( t \) – thickness of a coating; \( h \) – dimpling depth.

Research of frictional features was carried out using laboratory installation of friction at dry sliding at loading 1 N on an indenter of sapphire.

Tests of endurance was carried out at the Echo installation at dry transversal contact of the rotating core (steel U10, HRC = 65) by diameter ~8 mm load of 3 N during various time. Endurance of the original material and coating of TiN was estimated in geometrical parameter – diameter of a spot of the depreciation.

Process of destruction and the adhesion solidity are analysed by a sclerometric method by Revetest RST sketch-tester. Tests were passed with use of a diamond spherical indenter. Test parameters: the growing load of the indenter (AL) – from 1 to 50 N, the speed of movement of the indenter – 1 mm/min, length of scratch (L) – 3 mm, the loading rate – 16.33 N/min, sensitivity of the acoustic emission – 5.

3. Results of the research

By method of a submicroscopy it has been ascertained that at all modes of spraying the coating with the bar structure of TiN grains (figure 1) is received. The tendency of formation of the nanostructured TiN grains with decrease of pressure of reactionary gas in the camera and current of an arch (figure 2(a)) has been revealed.

![Figure 1](image_url)

**Figure 1.** The nanostructured bar covering of TiN received by various modes of spraying, x10000.
The received dependences of modification of a microhardness of a coating TiN on arc current at different fractional pressure of gas in the camera showed that the maximal value $H_{\mu}^{50} \approx 19.4$ GPa of a covering is received at arc current of $I = 120$ A and is caused by the finely divided microstructure. Decrease of microhardness of a coating by increasing the grain size (figure 2(b)) is installed with increase of arc current and gas pressure in the camera.

![Graphs](image)

**Figure 2.** Dependences of cross sectional size of the bar grains TiN (a), microhardness (b), relative change of friction coefficients (c) and endurance (d) of coating of TiN on arc current at different fractional pressure of gas in the camera.

Application of the nitride coating promotes decrease of the friction coefficient of the processed surface. Spraying of the TiN coating at the “$U = 140$ V, $P = 0.04$ Pa, $I = 120$ A” mode leads to decrease of a friction coefficient of an effective area of a steel detail in ~1.4 times (figure 2(c)).

It has been ascertained that the titanium nitride coating received at arc current of 120 A (figure 2(d)) have the greatest endurance (in ~4 times).

Analyzing results of the adhesion tests it is possible to mark out various threshold values of ultimate load leading to various types of destruction. Three stages of destruction are typical for coating with a microhardness of ≤13 GPa (figure 3) and two stages are typical for coating with a microhardness ≥15 GPa (figure 4).

At the first stage (load of an indentor to ~13 N) the indentor penetrates monotonous into a coating, at the same time the frictional force poorly increases, and amplitude of an acoustic emission remains invariable. The indentor leaves smooth slight marks on a coating. Sliding of a diamond indentor on a covering takes place with very low friction coefficient.

At the second stage an increase in amplitude of an acoustic emission and change of an inclination of curves of a frictional force and friction coefficient take place. At scratching with loadings more than 13–15 N chevron cracks appear at the bottom of scratch, cleavage of separate flakes (a cohesion destruction of a covering occurs) and islet detachment is observed on edges of scratches. Increase in loading over 20 N leads at first to the local and then permanent cleaving of a covering. The permanent cleaving of a covering leads to growth of amplitude of a signal of an acoustic emission, a frictional force increase monotonously up to 10 N. At further increase in a frictional force a fast attrition of a coating occurs.
Figure 3. Panoramic shot of the scratch (x200) and curve changes of signals of different sensors at scratching the composition "TiN – steel" at the increasing loading: AL – the size of normal loading ($P$); DP – depth of penetration of an indentor ($h$); FF – value of a frictional force ($F_N$); AE – value of the acoustic emission ($A_i$); CF – value of a friction coefficient ($\mu$) for TiN received by the mode $I = 160$ A, $P = 0.04$ Pa.

At the third stage penetration of an indentor deep into of substrate material is observed, at the same time amplitude of a signal of an acoustic emission decreases, and the frictional force (to 40 N) and a friction coefficient rise steeply.

Figure 4. Panoramic shot of the scratch (x200) and curve changes of signals of different sensors at scratching the composition "TiN – steel" at the increasing loading: AL – the size of normal loading ($P$); DP – depth of penetration of an indentor ($h$); FF – value of a frictional force ($F_N$); AE – value of the acoustic emission ($A_i$); CF – value of a friction coefficient ($\mu$) for TiN received by the mode $I = 120$ A, $P = 0.04$ Pas.
On the basis of results of the researches the optimal mode of spraying of a coating of TiN is chosen: \( P = 0.04 \text{ Pa}, \ I = 120 \text{ A}, \ U = 140 \text{ V} \) at which the nanostructured bar coating of TiN with an optimal combination of physic-mechanical, tribological and solidity features is formed.

4. Conclusion

The received nanostructured coating of TiN has been tested and implemented at many enterprises of machine-building branch for increase of efficiency and reliability of various details of machines: PA “Tochmash” (city Vladimir), SK «KMZ» (city Kovrov) – a limitative slideway of the gas centrifuge [4]; SK «KUZNETSOV» (city Samara) – the rods which are a part of the vibration isolations system of the RT-2 exercise machine for using on the space station ISS; MV “VOSCHOD” (city Pavlovo) – nipples for the aircraft equipment; various enterprises of Kazakhstan – plunger couples for the «KAMAZ» cars, the compressor shovels of high pressure restored for DZh59L3 engines.

References

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