Application of titanium nitride and titanium aluminonitride as intermediate layer for diamond-like coatings of steel parts

I A Buyanovskii¹, V D Samusenko¹, V A Levchenko²

¹ Department of Friction and Wear, Mechanical Engineering Research Institute of the Russian Academy of Sciences, 4 M. Kharitonyevskiy Pereulok, Moscow, Russia, 101990
² Faculty of Chemistry, Lomonosov Moscow State University, GSP-1, Leninskie Gory, Moscow, Russia, 119991

samusenkovd@gmail.com

Abstract. The physomechanical and tribological characteristics of DLC coatings was investigated, with using TiN and AlTiN hard coatings as intermediate layers. The tests were carried out both with dry friction and in the environment of PAO-4. It was found that intermediate coatings provide good adhesion of the DLC to steel; the best result was obtained with the AlTiN coating. The main factor determining antifriction properties in the friction pair “steel – coating” with boundary lubrication is tribological properties of coatings.

1. Introduction

Diamond-like coatings (DLC) have good protective and tribological properties. They also have good prospects for application in friction units requiring high reliability and antifriction properties [1], but often due to high internal stresses in diamond-like coatings there is a problem with adhesion to the substrate [2]. For this reason, multilayer coatings are used, which retain their high tribological properties due to the upper layer of diamond-like coating and have good adhesion due to intermediate layers. Generalized results of tribological tests of diamond-like coatings using titanium nitride [3] and titanium aluminum nitride [4] as intermediate layers are considered in this paper.

2. Materials and research methods

2.1. Samples

The properties of a diamond-like coating were investigated; titanium nitride (TiN) and titanium aluminum nitride (AlTiN) were used as intermediate layers. Cylindrical rollers 8x8 mm (D x L) made of 100Cr6 steel were used as samples for coating deposition. The investigated coatings were deposited to cylindrical surfaces of rollers by the vacuum-arc method of coating deposition (PVD-technology) [5].

Samples with deposited TiN and AlTiN coatings with a thickness ~ 1 μm were made; some of these samples were subsequently coated with a layer of DLC with a thickness of 0.3…0.5 μm. As a result, samples were obtained with TiN and AlTiN coatings, and with two-layer TiN-DLC and AlTiN-DLC coatings.
Measurements of nanohardness, elasticity modulus and adhesion of coatings were carried out on a nanohardness tester NanoScan-4D. Hardness was measured in accordance with recommendations of GOST R 8.748-2011. The Berkovich triangular diamond tip was used as indenter. The maximum loading force was 10 mN (the number of indents in the series is at least 30). Time of loading and unloading was 10 s. The results of measurements of the samples surface characteristics are shown in table 1. It can be seen that the nanohardness of samples with DLC is approximately 4 times higher than that of steel and approximately 2 times higher than that of the coatings that were used as intermediate layers.

Roughness of samples was measured on the optical profilometer S Neox along a profile with length of 100 μm along the generatrix. The results of roughness measuring are shown in table 1. Based on the results, the layers of titanium nitride and titanium aluminum nitride used to adhesion improving have almost no effect on the surface roughness. The application of DLC reduces the surface roughness.

| Characteristics of samples. |
|-----------------------------|------|------|-------|------|------|
| Arithmetic average roughness | Ra (nm) | TiN | TiN+DLC | AlTiN | AlTiN+DLC |
| Indentation depth (nm)      | 184 ± 16 | 120 ± 24 | 104 ± 14 | 138 ± 12 | 114 ± 12 |
| Hardness H (GPa)            | 9.4 ± 1.5 | 22 ± 7 | 49 ± 13 | 18.0 ± 2.4 | 41 ± 9 |
| Elastic modulus E* (GPa)    | 250 ± 31 | 400 ± 100 | 530 ± 110 | 264 ± 40 | 440 ± 80 |

The test procedure for evaluating the adhesion consists in making scratches with varying load and registration minimum force at which the coating delaminate from the substrate (ASTM C 1624). Delamination was determined by an optical image. Each sample was subjected to 3 scratches with a load from 10 mN to 2 N. The length of the scratch was 1 mm. The load was increased linearly. Photographs of scratches on the surface of samples with x10 magnification are shown in figure 1.

On a sample with TiN coating (figure 1 (a)) no delamination of the coating was observed. On a sample with TiN-DLC coating (figure 1 (b)) the destruction of the coating is stably observed at the load of 200 mN. Destruction of the coating takes place in the middle of the TiN layer. Chipping in the titanium nitride layer occurs at the depth of 0.3…1 μm from the surface of the coating.

On a sample with AlTiN coating (figure 1 (c)) no delamination of the coating was observed on two scratches. On the third scratch the coating began to delaminate at the load of 100 mN near the edge of the sample, which is probably due to defects during deposition in this zone. On samples with AlTiN+DLC coatings (figure 1 (d)) no delamination was observed. On the surface of the sample coated with AlTiN+DLC visible defects formed during deposition.
2.2. Tribological tests

Tribological tests were carried out on the friction test machine KT-2 according to the “rotating ball - three rollers” friction scheme [4]. Standard balls with diameter of 12.7 mm made of steel “IIIX-15” (100Cr6), and rollers 8x8 mm, on the cylindrical surface of which the investigated coatings were applied as samples for testing. The machine spindle with a ball clamped in it under the load of 108 N is pressed against the cylindrical surfaces of rollers folded into an equilateral triangle, which are installed in a mandrel in a plane perpendicular to the spindle axis. The spindle speed with the ball was 1 min⁻¹. The test duration for each set of samples was 60 min. During the study, values of the frictional moment are recorded, estimated by the angle of the torsion dynamometer twisting. Test results are presented in the form of dependences of the friction coefficient on the test duration. The graphs show the average values of the friction coefficient obtained from the results of three repeated tests. After the end of each test, wear scars were photographed on an optical microscope to assess the degree of damage of the sample surface.

3. Results and discussion

The results of tribological tests of samples without coating and with TiN and AlTiN coatings are shown in figure 2. The microphotographs of wear scars obtained after the end of tests are shown in figure 3.

When tested without lubricant (figure 2 (a)), the antifriction properties of all samples have similar dependences and sufficiently close values of the friction coefficient, taking into account the high scatter of values obtained during dry friction. The friction coefficient begins to increase strongly at the beginning of tests and stabilizes at 0.8…0.9 (steel-to-steel friction coefficient), which is probably a consequence of destruction of coatings (figure 3 (b) and (c)). The TiN coating has undergone destruction to a greater extent and the wear scar exceeds the size of wear of the uncoated specimen, which indicates that the destroyed coating particles act as an abrasive and intensify surface wear.

Under test conditions with the PAO-4 lubricant medium (figure 2 (b)), the antifriction properties of samples without and with coatings differ significantly, while the difference in antifriction properties between TiN and AlTiN coatings is insignificant and is within the measurement error. When testing samples with coatings, the coefficient of friction gradually increases throughout the test from 0.16 to 0.28, which is probably associated with gradual deterioration of coatings. As it can be seen from the photographs of wear scars (figure 3 (e) and (f)), the coatings are damaged and traces of plastic deformation of the surface are visible, however, during the test, the destruction of coatings under these conditions does not occur.
The results of tests of TiN-DLC and AlTiN-DLC coatings without lubricating medium show (see figure 4), that the friction coefficient with TiN-DLC coating is higher, which is a consequence of the coating destruction. The results of obtained wear scars are presented in figure 5. The increase in the friction coefficient during TiN-DLC test is most likely associated with stages of coating destruction (figure 5 (a)) and friction coefficient tends to the values obtained during testing of uncoated specimens. The AlTiN-DLC coating didn’t destruct during the test, only small traces of wear are visible on the surface (figure 5 (b)). Also it has a stable friction coefficient of 0.1 after the running-in stage.
Results of tribological tests of TiN-DLC and AlTiN-DLC coatings in PAO-4 lubricating media are shown in figure 6. The resulting wear scars are shown in figure 7. Antifriction properties of these two coatings in PAO-4 are close in values at a steady friction mode. At the stage of running-in the friction coefficient decreases and then stabilizes in the range of 0.09…0.11. The running-in process for TiN-DLC coating test is faster, and the resulting wear scar is larger according to the test results (figure 7).

![Figure 6. Dependences of the friction coefficient on the test duration of TiN-DLC (1) and AlTiN-DLC (2) in PAO-4.](image)

![Figure 7. Photographs of wear scars after testing TiN-DLC (a) and AlTiN-DLC (b) in PAO-4.](image)

The antifriction properties of the AlTiN/TiN-DLC coating do not differ significantly depending on the presence of the lubricating medium. From this result, it can be concluded that the presence of DLC is the determining factor for the antifriction properties of the tested friction pair. In [3,4], more detailed results of tribological tests of DLC with intermediate TiN and AlTiN layers in various lubricating media are given.

4. Conclusion

One of the most important characteristics of coatings is adhesion, which allows to maintain the required antifriction properties throughout the required life cycle of the friction unit. The destruction of coatings leads to a growth in the friction coefficient and increases the wear of surfaces in the coupling due to the formed abrasive particles from the destroyed hard coating.

Evaluation of adhesion showed that the AlTiN coating is more preferable than TiN for use as an intermediate layer for diamond-like coatings due to providing better adhesion to the sample surface. A similar result was obtained based on the tribological tests: the AlTiN coating is less susceptible to wear and destruction during friction, which ensures the preservation of the antifriction properties of the diamond-like coating.

The lubricating medium is not the main determining factor of the antifriction properties in the friction pair “steel - DLC”; the most significant in this case is the diamond-like coating itself, and the greatest attention should be paid to the ability of coating to resist destruction.

References

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