# INVESTIGATION OF THE ABRASIVE DURABILITY OF THE MULTILAYER DIAMOND-LIKE COATINGS FOR THE RING-SHAPED DRY GASEOUS SEALS MADE OF SiC

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The abrasion resistance of multi-layer vacuum-arc diamond-like (DLC) coatings of various architectures: quasi-gradient, as well as coatings with alternating hard and soft layers, whose structure and mechanical characteristics are specified by the amplitude of the bias potential and the deposition time of each layer are investigated. Increasing the thickness of the DLC to 6…7 μm by using a multilayer architecture with alternating soft and hard layers allows to improve the abrasion resistance of the protective coating, however, the adhesion of such coatings on silicon carbide is somewhat lower than that of the quasigradient ones. The parameters of the process of synthesis of DLCs, providing a significant improvement in the mechanical characteristics of multilayer coatings, in comparison with the single-layer ones, are established.

PACS: 81.05.U-; 81.15.Jj; 52.77.Dq

## INTRODUCTION

Earlier [1], the NSC KIPT developed a process for depositing vacuum-arc diamond-like coatings (DLC) without the use of an intermediate metal sublayer on the end surfaces of annular silicon carbide elements, for dry gas seals (DGS) of high-pressure compressors. The counterbody (the second body in a pair of friction) for coating in the DGS is the reciprocal end ring of silicon nitride. The contact of their surfaces during start-up and braking leads to abrasive wear. In connection with this circumstance, an investigation of the abrasive durability of diamond-like coatings for rings made of silicon carbide presents an actual problem.

One of the main requirements for coating is adhesion to the substrate. The experiment shows that the provision of the required level of adhesion of DLC, which have a sufficiently high hardness and are capable of operating under the conditions of the DGS, is quite a challenge. The reason are the large internal residual stresses in such coatings, the greater, the higher their hardness. Several variants of the solution of this problem have been suggested in the literature. Some improvement in the adhesion of single-layer DLC can be achieved using a sublayer of metal (usually carbide-forming: titanium or, chromium) [2]. The use of a multilayer architecture allows the application of hard DLC with a thickness of several microns to different materials. One of the variants of the multilayer coating architecture is the combination of several functional monolayers of a thickness on the order of a micron each with different properties. The layer applied to the substrate is designed to provide a high level of adhesion to the substrate. Intermediate layers of plastic materials adjacent to the DLC should compensate for the residual internal mechanical stresses of hard DLC layers by means of plastic deformation, which, as a result, allows create a hard coating of sufficiently large thickness (several microns) [3].

Multilayer quasigradient coatings are a sequence of layers with monotonously varying characteristics. For example, the bottom layer is the softest, least stressed, each subsequent is more and more hard, but more stressed. The periodic structure of a large number of alternating very thin layers with different properties, when the period of the structure is much smaller than the thickness of the coating is a composite. In the special case of nanoscale layers (the period of the structure is 10…100 nm) such material is nanocomposite [4].

The purpose of this work is to study the abrasion resistance of multilayer DLC: quasigradient, as well as multilayer coatings consisting of periodically repeated hard and soft DLC layers.

## 1. EXPERIMENTAL TECHNICS

The coatings were deposited by a vacuum arc method from a plasma source with a rectilinear particulate filter [1] on substrates of two types: 12Cr18N10T stainless steel plates of size 20x17 mm with a thickness of 1.5 mm and coupons of silicon carbide with a diameter of 15 mm with a thickness of 6 mm.

Abrasive tests were carried out according to the scheme of a rotating abrasive disk – a plane. The DLC deposited on the substrates of stainless steel were tested. As an abrasive, corundum powder (grain size 30…125 μm) was used on the vulcanite binder. According to the literature data, the hardness of corundum is from 18 to 24 GPa, that is, slightly higher than that of silicon nitride – 14…16 GPa.

During the test, the rotational speed of the disk with a diameter of 27.6 mm was 2790 rpm, which corresponds to a linear velocity of abrasive grains V = 400 cm/s, the pressing force of the disk to the plane of the sample was 2.2 N. The duration of the test was 300 s. The abrasive wear of the samples was evaluated by weighing, as well as visually by optical microscopy.

To stabilize the operation of the plasma source, argon was introduced into the vacuum chamber to a pressure of $1 \cdot 10^{-2}$ Pa. Samples were fixed on the water-cooled substrate holder. A bias potential was applied to the substrate holder in the form of high-voltage pulses of negative polarity. The pulse generator provides the following pulse parameters: amplitude 0.5…2 kV, duration 6…20 μs, repetition rate 1.2…12 kHz.

The thickness of the deposited DLC was measured using an interferometric microscope MII-4. Adhesion to the substrate was evaluated based on the results of


*ISSN 1562-6016. ВАНТ. 2019. №1(119)*
*PROBLEMS OF ATOMIC SCIENCE AND TECHNOLOGY. 2019, № 1. Series: Plasma Physics (25), p. 216-218.*

scratch testing. The diamond indentor (with a spherical shape with a radius of curvature of 0.5 mm) moved along the surface at a rate of 0.57 mm/s at a constant load in the range 5…40 N. The hardness of the coatings was measured by a nanoindentor G-200, continuous stiffness measurement (CSM).

## 2. RESULTS OF THE RESEARCH

The experiments showed that the wear of the samples was uneven over the contact surface with the abrasive disc. It was maximal in multiple parallel tracks formed by moving grains of abrasive upon contact with the coating (Fig. 1).

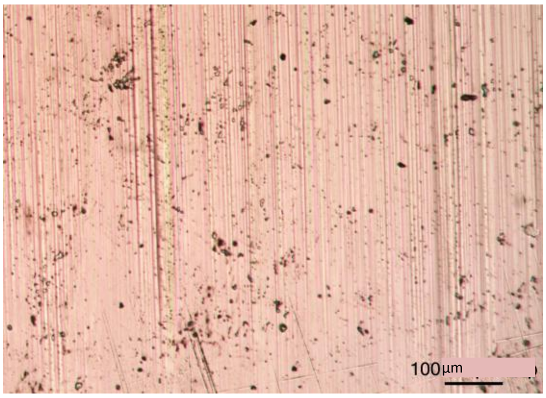


*Fig. 1. Traces of abrasive wear on the surface of an eight-layer APP 6 µm thick*

The length of each single track was about 1 mm, the width of 1…2 µm. The depth of the track during the test period of 300 s reached 2…2.5 µm. The adjacent tracks, overlapping each other, overlap partially. The resulting profile, as can be seen from the observation in the interference microscope (Fig. 2), is quite smooth, which is typical for cases when the hardness of the abrasive and the processed material are close in magnitude and wear of both one and the other material occurs.

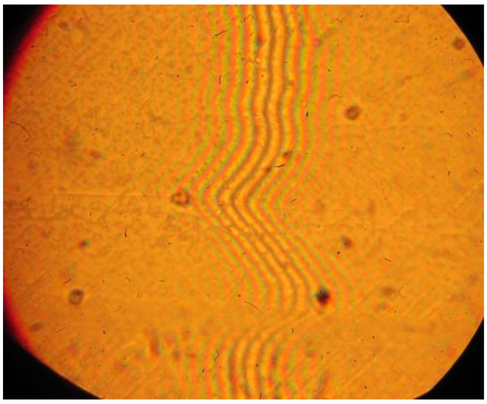

*Fig. 2. Interferogram of the profile of abrasive wear tracks*

The loss of mass of samples was determined by weighing on analytical scales. For all the investigated coatings, it was $(1.5…2)\cdot10^{-4}$ N for 300 s, that is, at the sensitivity limit of the scales. In this regard, the method used did not allow comparing the abrasive wear rates of multilayer DLC coatings of various architectures.

Analysis of the microphotographs of the surface of the samples after the tests showed that the tracks of wear of the thinnest coating (2.4 µm thick) reached the substrate during the test, which led to catastrophic destruction of the coating (Fig. 3).


*Fig. 3. Traces of abrasive wear on the surface of a three-layer DLC with a thickness of 2.4 µm*

On coatings 3…4 µm thick only single tracks reached the substrate. At a thickness of 6 µm and above under the same conditions of testing, damage to the coatings was not observed (see Fig. 1).

Adhesion of multilayer DLC deposited on coupons from silicon carbide was evaluated by the results of a scratch test (the load on a diamond indenter with a spherical tip of 500 µm radius was from 5 to 40 N).

According to the results of this test, the adhesion level to silicon carbide of quasigradient DLC with a total thickness of 2.1 µm is 40 N [1]. Among examined DLC with alternating soft and hard layers the best adhesion to silicon carbide showed a five-layer DLC with soft upper layer, a total thickness of 3.2 µm n, which withstood without destruction test with load 30 N. It should be noted that the authors of [4] also reported an improvement in the tribological characteristics of nanocomposite DLC in the presence of a soft top layer. Coatings on silicon carbide consisted of four, eight and twelve DLC layers with an upper hard layer, total thickness of 2.3, 4.1 and 6 µm respectively, were scratched to the base under loads 10…15 N.

Indirectly, the adhesion of the coating to the substrate can be evaluated from the results of observation of the coating surface in the vicinity of imprints of diamond indenters (Vickers, Rockwell, Berkovich) obtained under different loads [5]. This test makes it possible to assess the crack resistance of the coating, a very important characteristic of hard coatings that work at high loads. We used a PMT-3 hardness tester with a Vickers pyramid loaded in the range 0.5...2 N.

The analysis of imprints when observed in an optical microscope showed that when the indentor is 1…2 N loaded, in the coatings with a hard upper DLC peripheral cracks are formed on the outside of the imprint (Fig. 4). The initiation of cracks and their further development during the penetration of grains of abrasive in the sample is, apparently, the main cause of the destruction of the coating during grain movement along the surface at abrasion tests.

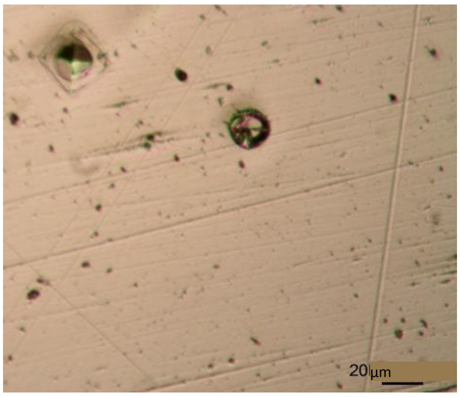


*Fig. 4. Imprints of the Vickers pyramid at loads 1 and 2 N. There are four layers in the DLC, the upper layer is hard*

## CONCLUSIONS

The multilayered DLC coatings are synthesized: quasigradient, as well as coatings with alternating hard and soft layers, whose structure and mechanical properties are determined by the amplitude of the substrate bias potential and the deposition time of each layer, and their abrasion resistance is investigated. The parameters of the synthesis process are established, which ensure a significant improvement in the characteristics of multilayer coatings, as compared to single-layer coatings.

The increase in the thickness of the DLC to 6…7 μm due to the use of a multilayer architecture with alternating soft and hard layers allows to improve the abrasion resistance of the protective coating, however, the adhesion of such coatings to silicon carbide is slightly lower than that of the quasigradient ones.

From the point of view of the complex of characteristics, along with the quasigradient one, as an advanced protective coating for the end seals of the DGS from silicon carbide, one can also consider DLC with alternating soft and hard layers and a soft top layer.

The work was carried out within the framework of the program of scientific research of the NAS of Ukraine: “Reliability and durability of materials, structures, equipment and facilities” (Resource-2), contract № 88 P4.8/2018.

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*Article received 28.09.2018*

### ИССЛЕДОВАНИЕ АБРАЗИВНОЙ СТОЙКОСТИ МНОГОСЛОЙНЫХ АЛМАЗОПОДОБНЫХ ПОКРЫТИЙ ДЛЯ УПЛОТНИТЕЛЬНЫХ КОЛЕЦ ИЗ КАРБИДА КРЕМНИЯ СУХИХ ГАЗОВЫХ УПЛОТНЕНИЙ

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Исследована абразивная стойкость многослойных вакуумно-дуговых алмазоподобных покрытий (АПП) различной архитектуры: квазиградиентных, а также покрытий с чередующимися твердыми и мягкими слоями, структура и механические характеристики которых задаются амплитудой потенциала смещения и временем осаждения каждого слоя. Увеличение толщины АПП до 6…7 мкм за счет использования многослойной архитектуры с чередующимися мягкими и твердыми слоями позволяет улучшить абразивную стойкость защитного покрытия, однако адгезия таких покрытий на карбиде кремния несколько ниже, чем у квазиградиентных. Установлены параметры процесса синтеза АПП, обеспечивающие существенное улучшение механических характеристик многослойных покрытий по сравнению с однослойными.

### ДОСЛІДЖЕННЯ АБРАЗИВНОЇ СТІЙКОСТІ БАГАТОШАРОВИХ АЛМАЗОПОДІБНИХ ПОКРИТТІВ ДЛЯ УЩІЛЬНЮВАЛЬНИХ КІЛЕЦЬ З КАРБІДУ КРЕМНІЮ СУХИХ ГАЗОВИХ УЩІЛЬНЕНЬ

***В.В. Васильєв, О.А. Лучанінов, В.Є. Стрельницький***

Досліджено абразивну стійкість багатошарових вакуумно-дугових алмазоподібних покриттів (АПП) різної архітектури: квазіградієнтних, а також покриттів з періодично повторюваними твердими та м’якими шарами, структура та механічні характеристики яких задаються амплітудою потенціалу зміщення і тривалістю осадження кожного шару. Збільшення товщини АПП до 6…7 мкм за рахунок використання багатошарової архітектури з періодично повторюваними твердими та м’якими шарами дозволяє покращити абразивну стійкість захисного покриття, однак адгезія таких покриттів на карбіді кремнию дещо нижча, ніж у квазіградієнтних. Встановлено параметри процесу синтезу АПП, які забезпечують суттєве поліпшення механічних характеристик багатошарових покриттів порівняно з одношаровими.