

Properties of protective carbon films applied on stainless steels via magnetron sputtering

A. E. Litvinov, Dr. Eng., Associate Prof., Head of the Dept. of Technical Mechanics and Special Machines¹,
e-mail: artstyleone@mail.ru;

V. Yu. Buzko, Cand. Chem., Associate Prof.^{1,3}, Dept. of Radiophysics and Nanomaterials²;

S. N. Ivanin, Cand. Chem., Researcher, Center for Advanced Technologies and Nanomaterials¹⁻³;

M. V. Kolodin, Mag. Eng., Dept. of Radiophysics and Nanomaterials²

¹ Kuban State Technological University (Krasnodar, Russia)

² Kuban State University (Krasnodar, Russia)

³ Kuban State Agrarian University (Krasnodar, Russia)

Thin films of hard diamond-like carbon (DLC), containing amorphous carbon with disordered graphite ordering, which is striving to a tetrahedral diamond-like coordination, are interesting for material scientists due to their excellent mechanical and tribological properties. Dense nanostructured carbon films DLS containing tetrahedral 46.7 % of sp³-links with nanoparticles having average size 28 ± 8 nm, were applied on stainless steel SS 304 samples via the method of high-frequency magnetron sputtering without adhesive substrate and with it; their microstructural parameters were examined. The results displayed that assessed dimensions of individual carbon nanoparticles were varied within the range 25–27 nm. Obtained films of diamond-like carbon are characterized by rather low surface roughness at the level 3.6 nm. It is an important practical property, because low roughness promotes decrease of friction and wear, what can be required in various technological applications. DLC films displayed hydrophilic behaviour with measured value of the contact angle 62 ± 2° and 55 ± 2° (for a film with adhesive titanium layer) in comparison with the value 52 ± 1° for stainless steel SS 304. Additionally, the surface structure of Ti-DLC film was analyzed after wear resistance testing via friction. It was revealed as a result of analysis that wear degree of film surface was rather low. Absence of defects, cracks and saving of definite degree of homogeneity of disordered film structure testify on its good wear resistance.

Key words: high-hard protective coating, nanostructured films, diamond-like carbon, stainless steel, magnetron sputtering, thermomechanical wear.

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Introduction

The family of amorphous carbon films with mainly tetrahedral coordination of links is called as diamond-like carbon (DLC) [1, 2]. This material demonstrates very high hardness in thin films [1–4] with rather elastic properties, as well as excellent heat conductivity [4], high electric resistance [1, 4], chemical inactivity [2, 4], very low sliding friction coefficient [5, 6], high transparence in infrared range [5], high coefficient of thermal emission capacity in near infrared spectral region [6].

Low coating adhesion with metallic substrates can often be a feature of diamond-like carbon films [7–9]. It is caused by high internal stresses in a DLC film. Such problem can be solved either by use of carbide-forming materials for a substrate (such as titanium and iron), or by use of multi-layer coatings with adhesive substrate [9–11].

Deposition of protective DLC coatings was conducted at the industrial cathode-arc equipment via their application of chromium binding layer on substrates (made of nitrided stainless steel) [10]. It was followed by consequent carbon bombardment with various shift stresses, cathode currents and temperature, as well as direct pulverization of diamond-

like carbon functional layer with thickness 20 μm. The stage of carbon bombardment was included for creation on inter-phase structure, which contain of the mixed Cr and C layer and carbon transition layer (rich for sp²-links). Adhesion of DLC layer was assessed with assistance of scratching tests, while structure of separation surface was examined via transmission electron microscopy. The value of adhesive strength for all obtained structures was examined in the conditions of destruction, and the obtained picture was characterized by shearing distortions and intensive coating lamination and was similar for each experiment [10]. According to the obtained results, violation of adhesion occurs between chromium intermediate layer and DLC layer. It can be suggested that carbon transition layer with higher content of sp³ links is less sensitive to thickness variation, because sp³ links provide higher shear strength [10].

According to the research [11], the temperature gradient can provide serious effect on carbon transition layer, which was grown during a bombardment, because the temperature has influence on content of sp³-links. Thus, it can be assumed that the lower is the temperature, the higher is content of sp³-links in the formed layer. Then adhesion increase of DLC

films is connected with variation of carbides and increase of content of sp^3 -links in carbon transition layer, which was formed directly above the mixing layer [10]. Therefore, it can be concluded that use of lower temperature during carbon bombardment provides less part of sp^2 -links in a sample indeed [10]. Improvement of adhesion of a sample DLC layer can be explained by higher shear strength, which was caused by higher part of sp^3 -links.

The aim of this research was manufacture of DLC films with high protective capacity via the method of magnetron sputtering on substrates made of stainless steel SS304 (without adhesive layer or with it) within the medium infrared range. The research also was devoted to examination of microstructure and properties of manufactured DLC nanomaterials and determination of the relationship between DLC film strength and microstructure of samples.

Used sensors and equipment

The samples of German stainless steel SS 304 with thickness 0.5 mm were used in this research. Non-magnetic austenitic stainless steel SS 304 is an analogue to the domestic stainless steel 08Kh18N10 and is widely used for manufacture of equipment for food and processing industries, for different kinds of electric conductors and cable connectors in micro- and radio electronics.

High-clean graphite foil with thickness 0.5 mm (carbon part 99.999 %, made in China) was used as a source of carbon to conduct the processes of magnetron and plasma sputtering of DLC film; targets for laboratorial unit for magnetron and plasma sputtering were cut from this film.

Microstructure images of DLC film samples on metallic substrates were obtained in the scanning electron microscope «JSM–7500F» (JEOL Ltd, Japan). Elementary composition of stainless steel samples was examined via the method of energy-dispersive electron micro-analysis (EDA) using «INCA X-Max» (Oxford Instruments) adapter to the scanning electron microscope «JSM–7500F». Stainless steel samples with size 5×5 mm for measurements were cut by fiber-optic ytterbium laser from a sheet with 0.5 mm thickness and were located at special brass holders using carbon scotch.

The measurements of elementary composition for a stainless steel sample were carried out three times in different areas and the results were statistically averaged. To assess the parts of sp^3 - and sp^2 -links in the formed layer of DLC film, a laser Raman spectrometer HO-SP-LRS218 (Holmarc Opto-Mechatronics Ltd, India) was used. Parameters of DLC film wetting by water were determined with assistance of a the contact angle meter HO-IAD-CAM-01BC (Holmarc Opto-Mechatronics Ltd, India).

Study of the elementary composition of the stainless steel SS 304 via EDA method

The results of determination of elementary composition via EDA method for examined samples of the stainless steel SS 304 are presented in the **Table 1**.

Table 1. Elementary composition of the samples of the stainless steel SS 304 examined via EDA method		
Element, % (mass.)	Average	Average deviation
Fe	69.51	0.39
Cr	19.83	0.53
Ni	7.71	0.51
Mn	2.55	0.27
Si	0.40	0.09

The data of the Table 1 correlate well with known compositions of the samples of the stainless steel SS 304 from different technical sources. The features of surface microstructure of the sample of the stainless steel SS 304, which were obtained via the method of scanning electron microscopy (SEM) during EDA measurements, are shown in the **Fig. 1**.

Based on the data of the Fig. 1, the back non-polished part of the examined sample of stainless steel SS 304 is characterized by rather higher roughness, exceeding roughness of the face polished part by 4–5 times. Taking into account the data on surface microstructure of the examined sample of stainless steel SS 304, consequent elementary analysis via EDA method was conducted only for face polished parts of the samples, because minimal micro-roughness of metallic surface provided maximal accuracy of determination of elementary composition of these samples.

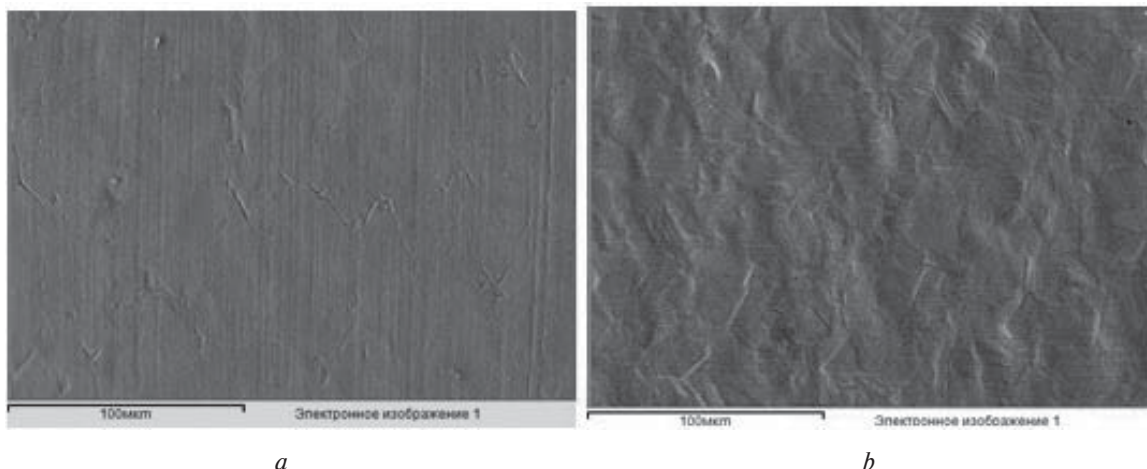


Fig. 1. Surface microstructure of the samples of the stainless steel SS 304, which was examined via the method of scanning electron microscopy: *a* – face polished part of the steel SS 304; *b* – back rough part of the steel SS 304

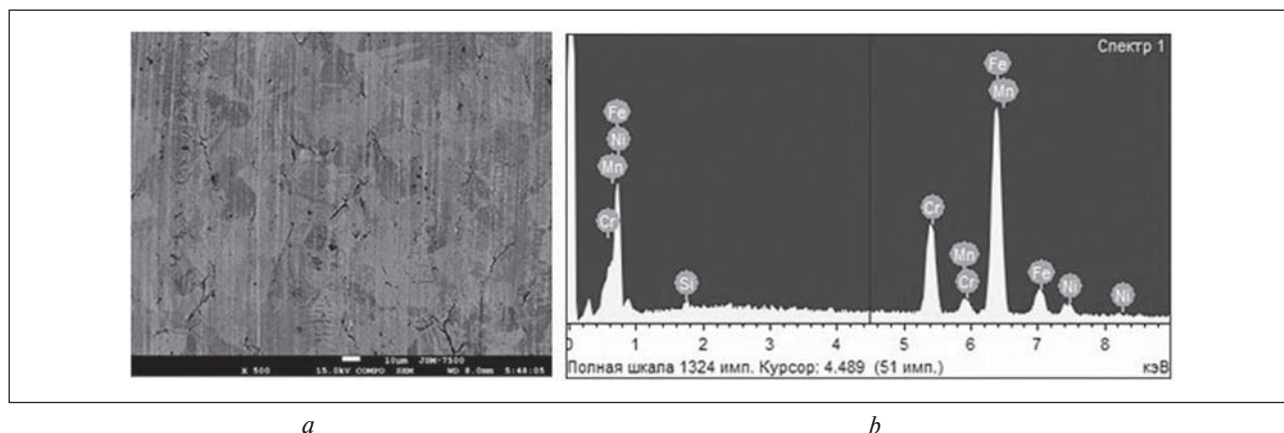


Fig. 2. Surface of the sample (a) and EDA spectrum (b) of the examined sample of stainless steel SS 304

The surface of the sample of stainless steel, which was snapshot in the conditions of reflected electrons, is presented in the Fig. 2, a. The varied EDA spectrum of the examined sample of stainless steel SS 304 is shown in the Fig. 2, b. This spectrum of EDA microanalysis of the examined sample of stainless steel SS 304 displays the peaks of electronic absorption and clearly reflects its elementary composition in significant concentrations of chemical elements.

Study of DLC films and Ti-DLC composite films on the surface of stainless steel SS 304

The samples of DLC film were applied on the surface of prepared samples of stainless steel SS 304 with size 5×5 mm with observation of the following optimal parameters of magnetron sputtering: magnetron capacity 300 Wt, sputtering current 0.43 A, argon feed $25 \text{ cm}^3/\text{min}$, heating temperature of the rotating table with the samples 270°C .

Calculated speed of magnetron sputtering of DLC film constituted at present time $3.2 \pm 0.2 \text{ nm}/\text{min}$ (according to experimental data), though the measured speed of DLC film sputtering was $7.5 \pm 0.2 \text{ nm}/\text{min}$ during brief initial time period 10 min. This initial speed of sputtering decreased systematically by the managing program during sputtering process. The samples of stainless steel SS 304 were purified two times using cotton wool and water-free isopropyl alcohol, with removal of fat deposits (the brand “For microelectronics”, China). These samples were placed on a working table of the laboratorial unit for magnetron sputtering CY-MSP300S-DC (China) using a tweezers made of surgery stainless steel.

Microstructure of fabricated DLC film with approximate thickness 300 nm on SS 34 substrate is presented in the Fig. 3 with various magnification levels. Microphotographs reflecting presence of DLC film on stainless steel and allowing to assess its thickness are displayed as well.

It can be seen (Fig. 3, a), that applied thin DLC film shows relief of metallic microcrystallines of the initial sample of stainless steel SS 304, which is used as substrate. It is confirmed by the fact, that the boundaries between metallic microcrystallines (which are laying under DLC film) and their orientation direction are seen definitely in the Fig. 3, a. It can be seen also (Fig. 3, b), that the average size of aggregated carbon nanoparticles makes about 85–95 nm for the sample of stainless steel SS 304 of fabricated DLC film, while assessed average size of individual nanoparticles is about 28 nm (see Fig. 4). It characterized small sputtering speed for DLC film and displays significant part of more large carbon nanoparticles, which were repeatedly recrystallized during heating.

Microstructure of fabricated DLC film is characterized by apparently larger size of carbon nanoparticles in comparison with the results of the previous research [6], probably owing to high initial speed of magnetron sputtering $97.5 \text{ nm}/\text{min}$ instead of $3.6 \text{ nm}/\text{min}$, with higher magnetron capacity (350 Wt instead of 50 Wt) and larger distance from carbon target to substrate. It can be also supposed that used heating of substrate at the temperature 270°C provides the effect on larger size of carbon nanoparticles in DLC film in our research in comparison with the previous research [6].

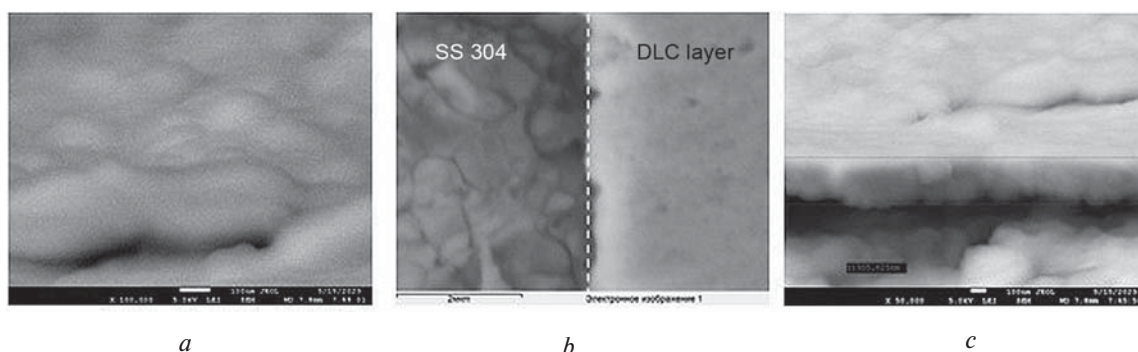


Fig. 3. SEM images of DLC film on the examined sample of stainless steel SS 304

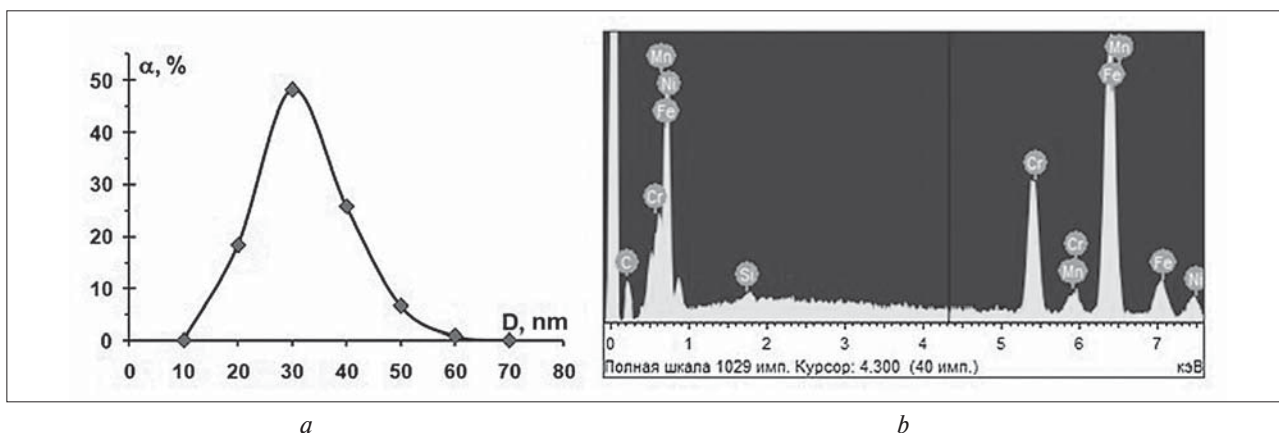


Fig. 4. Distribution for size of nanoparticles (a) and EDA spectrum (b) of the examined sample with applied DLC film

According to the data in the Fig. 3, c, we can see that applied thin DLC film, which was applied via magnetron and plasma sputtering, has the observed thickness 305.6 nm, what is close to the calculated value 300 nm. These data are based on previously known values of DLC film sputtering speed, which were obtained on the laboratorial unit for magnetron sputtering at the preset chosen level of magnetron capacity. Small lamination of DLC film from metal surface was observed in several cases on the edge of SS 304 substrate owing to the effect of oxide crystallines due to laser cutting; it allowed, in its turn, to determine thickness of applied DLC layer.

The measured EDA spectrum of the examined sample of stainless steel SS 304 with applied DLC layer (Fig. 4) displayed that this layer apparently increased the part of observed carbon via the method of X-ray spectral energy-dispersive microanalysis.

The results of determination of elementary composition via EDA method for the examined samples of the stainless steel SS 304 with DLC film are presented in the **Table 2**.

Table 2. Elementary composition of the sample of the stainless steel SS 304 with DLC film examined via EDA method		
Element, % (mass.)	Average	Average deviation
C	13.68	0.99
Fe	60.58	1.11
Cr	16.74	0.66
Ni	7.17	0.92
Mn	1.55	0.42
Si	0.38	0.19

To assess the parts of sp^3 - and sp^2 -links in the formed layer of DLC film, the method of Raman spectroscopy (laser Raman spectrometer HO-SP-LRS218, Holmarc Opto-Mechatronics Ltd, India) was used. Parameters of DLC film wetting by water were determined with assistance of a the contact angle meter HO-IAD-CAM-01BC (Holmarc Opto-Mechatronics Ltd, India). KP-spectrum of the examined film showed presence of typical oscillating bands if C-C groups for the films of diamond-like carbon [12–14] in the areas $1300\text{--}1350\text{ cm}^{-1}$ (ID) and $1600\text{--}1650\text{ cm}^{-1}$ (IG). Squares of oscillating bands for examined DLC film were calculated according to the data of analysis of KP spectrum lines form. It allowed to determine the relationship if

bands $ID/IG = 1.14$ and to calculate the part of tetrahedral sp^3 -links for the examined DLC film equal to 46.7 %.

Fabrication and examination of DLC film of stainless steel 304 with adhesive titanium layer

In order to examine adhesive characteristics of diamond-like carbon films on cleaned substrates of stainless steel SS 304, composite coatings of titanium and diamond-like carbon (Ti-DLC) were applied via the method of magnetron sputtering, by pulverization of titanium target before DLC application.

The coating process was started from preliminary deposition of DLC substrate with thickness 5 nm on SS 304 substrate. Then titanium layer with thickness 25 nm was applied, it served for increase of DLC coating adhesion to stainless steel substrate. Capacity used during titanium layer deposition made 150 Wt. Finally DLC layer of graphite target was applied with magnetron capacity 300 Wt. All processes of magnetron and plasma sputtering were carried out at the room temperature with permanent operating pressure 0.22 Pa. Thickness of obtained Ti-DLC coatings was kept at the level 330 ± 5 nm for all samples.

The proposed mechanism of forming of a composite wear-resistant Ti-DLC coating on SS 304 substrate is presented in the **Fig. 5**.

EDA spectrum for a composite Ti-DLC layer and corresponding data with elementary analysis are presented below

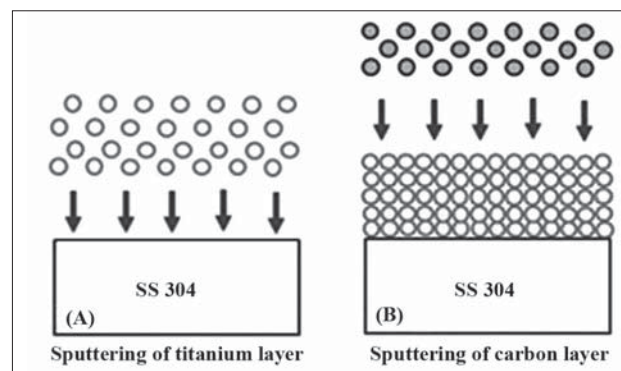


Fig. 5. The scheme of magnetron sputtering of a composite Ti-DLC coating

Table 3. Elementary composition of the sample of the stainless steel SS 304 + Ti with DLC film examined via EDA method

Element, % (mass.)	Average	Average deviation
C	13.63	0.96
Fe	60.19	1.02
Cr	16.22	0.67
Ni	7.05	0.82
Mn	1.49	0.54
Si	0.36	0.18
Ti	1.06	0.32

(Fig. 6 and Table 3). Microstructure of titanium adhesive layer on SS 304 substrate for various magnification layers is also shown in the Fig. 6.

The data of Fig. 6 display that adhesive titanium layer on SS 304 substrate is mainly characterized by island structure for large magnification level and contains approximately of nanoparticles with average size about 22–25 nm.

Microstructure of fabricated DLC film with approximate thickness 330 nm on SS 304 substrate, applied through ad-

hesive titanium layer, is presented in the Fig. 7 for relatively large magnification.

According to the data from the Fig. 7, thin DLC film, which was applied on stainless steel SS 304 substrate through an adhesive titanium layer via magnetron-plasma method, is characterized by principally other type of microstructure in comparison with DLC film without titanium layer. Also it can be seen, that average size of aggregated carbon nanoparticles in a stainless steel SS 304 sample of fabricated DLC film constitutes about 75–80 nm, what testifies on low sputtering speed of DLC film. Analysis of the results displayed that surface roughness of applied composite wear-resistant Ti-DLC coating was approximately 3.6 nm for Ti target (with capacity 150 Wt) and the observed texture of Ti-DLC coating was stipulated by material of used stainless steel.

DLC films displayed hydrophilic behaviour with measured value of the contact angle $62 \pm 2^\circ$ and $55 \pm 2^\circ$ (for a film with adhesive titanium substrate) in comparison with the value $52 \pm 1^\circ$ for stainless steel SS 304 (based on the data of three measurements). Additionally, the surface structure

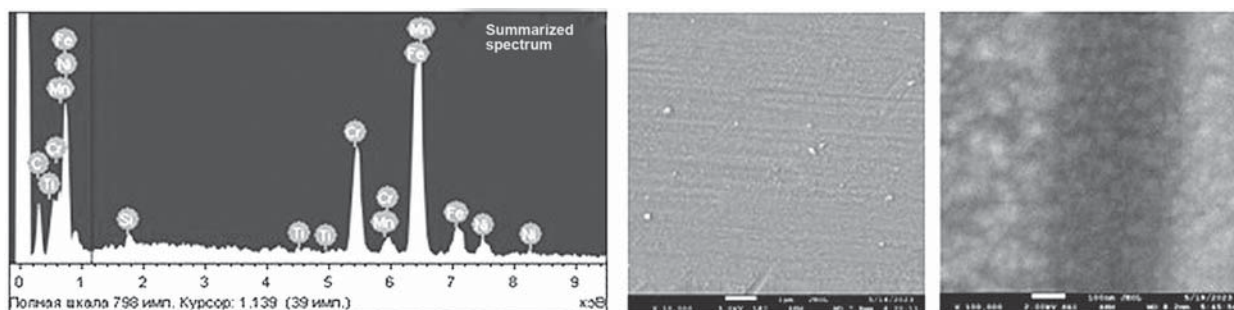


Fig. 6. EDA spectrum and microstructure of the sample of stainless steel SS 304 + Ti with applied DLC film

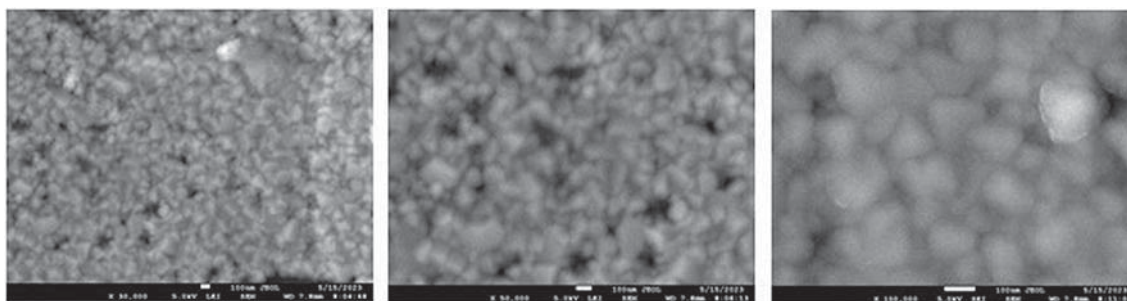
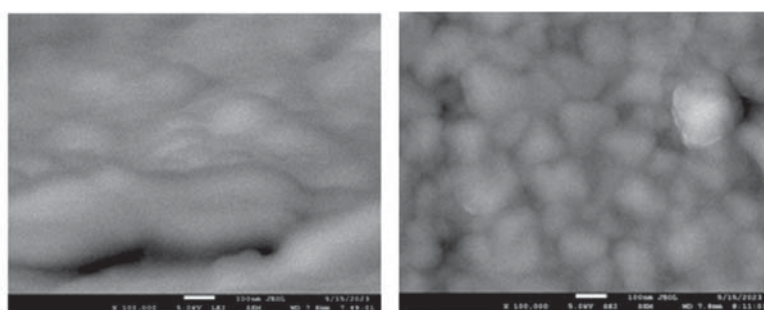


Fig. 7. SEM images of DLC film on the examined sample of stainless steel SS 304 with titanium layer



without titanium layer

with titanium layer

Fig. 8. Comparison of DLC film microstructure in the examined sample of stainless steel SS 304 with titanium layer and without it

of Ti-DLC film was analyzed after wear resistance testing via friction. Most probably it was connected with form variation of carbon nanoparticles in applied carbon film (**Fig. 8**) both in the cases of using adhesive titanium layer and without it. It can be seen from the Fig. 8, that DLC film, which was grown on titanium layer, is characterized by form of particles and by smaller size of nanoparticles.

Mechanical properties of composite Ti-DLC coatings were examined using micro-hardness meter CSM UNHT. To conduct indentation testing, a standard diamond Berkovich tip with maximal applied load about 0.3 mN was used. Indentation depth was less than 25 % of total coating thickness for excluding substrate effect on testing results. The values of hardness and Young module for examined composite Ti-DLC coating made of stainless steel were ~ 21.4 GPa and ~ 171 GPa respectively, what shows good correlation with the results of researches [5–10]. Ti-DLC coating made of stainless steel demonstrated higher hardness level in comparison with a simple DLC film (about 16.8 ± 0.9 GPa) for this steel. Supposedly it is connected with the fact, that the conditions for forming of ultra-hard TiC nanocrystallines, which increase hardness of a composite Ti-DLC coating, are created in the fabricated composite film after magnetronic sputtering combined with annealing at the same time.

Microstructure of the composite Ti-DLC coating after double scratch test via scratching by tungsten carbide needle under load 1 N is presented in the **Fig. 9** for two photo shooting procedures (detecting of low-energy and scattered electrons).

According to the data of the Fig. 9, thin DLC film, which was applied on stainless steel SS 304 substrate through a titanium layer via magnetronic-plasma method, is characterized by high resistance on the base of a scratch test. Complete

destruction of the DLC film in the area of scratch-test is not observed, what is confirmed by obtained microphotograph in the conditions of reflected electrons (COMPO). DLC film is destroyed not through the whole depth, because carbon nanoparticles, which are typical in their size and shape, are apparently observed in the area of removal of destructed layer [8–12]. The right microphotograph in the Fig. 9 displays the area of violations of protective DLC film with large magnification.

Fig. 10 presents variation of a surface layer of DLC coating structure after conduction of wear resistance test using friction method with tribometer MMD1. This tribometer of “pin-on-disc” type operates with metallic ball pin made of stainless steel AISI 430, with diameter 5 mm and normal load 2 N. Analysis of this figure allows to formulate several observations about surface state after testing.

Based on the data of the Fig. 10 it can be noted that wear resistance of the surface of protecting Ti-DLC film, which is caused by friction, is rather small. This conclusion can be made on the base of absence of visible defects, expressed micro-cracks and essential violations. General surface view (**Fig. 10**) displays definite degree of homogeneity of disordered structure in protecting Ti-DLC film [12–15].

Conclusions

Thin films of diamond-like carbon on stainless steel SS 304 were fabricated in this research via the method of magnetronic sputtering with use of adhesive titanium layer and without it. The films were subjected to consequent examination to assess their characteristics. The obtained results displayed that assessed size of individual carbon nanoparticles was varied in films within the range 25–27 nm. Fabricated films of diamond-like carbon are characterized by rather

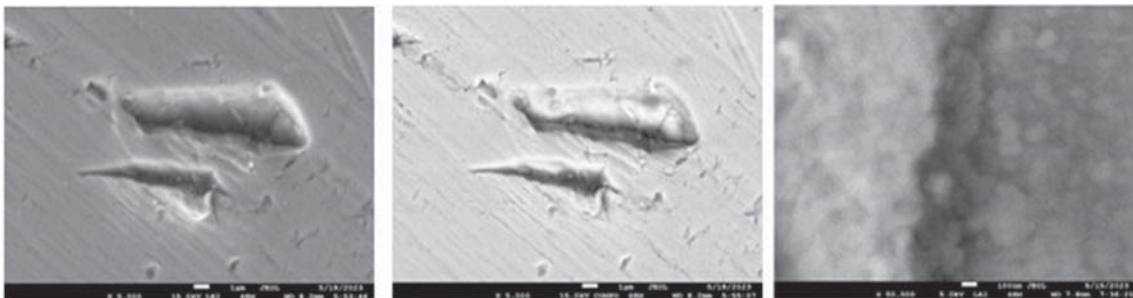


Fig. 9. SEM images of Ti-DLC film on the examined sample of stainless steel SS 304 after double scratch-test (pictures made at different procedures)

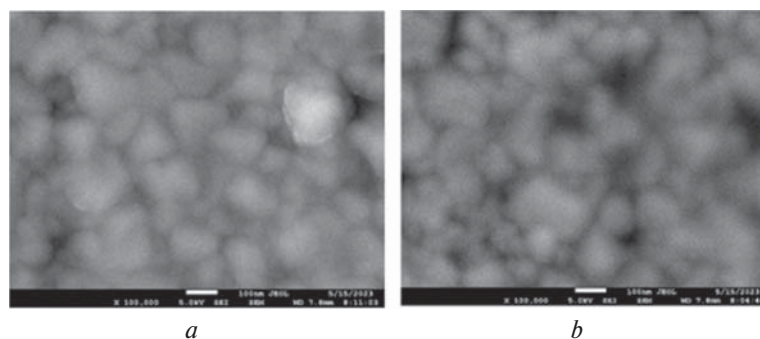



Fig. 10. SEM images of DLC film on surface of the examined sample of stainless steel SS 304 before (a) and after (b) wear resistance testing

low surface roughness at the level 3.6 nm. It is considered as important practical property, because low roughness promotes decrease of friction and wear, what can be required in various technological applications.

Use of adhesive titanium layer in sputtering of DLC film allowed to increase surface hydrophilic behaviour with measured value of the contact angle $62 \pm 2^\circ$ and (for a DLC+Ti film) in comparison with the value $55 \pm 2^\circ$ (for DLC film on stainless steel SS 304) and $52 \pm 1^\circ$ for stainless steel SS 304. Additionally, the surface structure of Ti-DLC film was analyzed after wear resistance testing via friction.

Additionally, surface structure of Ti-DLC film was analyzed after conduction of a wear resistance test via friction method. As a result, it was revealed that degree of surface wear of DLC protective films was rather low. No defects or cracks, together with save of definite homogeneity degree of carbon film disordered structure on protected stainless steel surface were revealed; it testifies on its good wear resistance. 

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