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RESULTS OF MEASUREMENTS OF SUBSTRATE DEFORMATION AND DETERMINATION BY BENDING OF INTERNAL STRESSES IN Ti-TiC-DLC COATING OBTAINED BY USING HiPIMS TECHNOLOGY

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Abstract. The paper presents the results of studies of internal stresses in ion-plasma coating Ti-TiC-DLC. A method based on measuring the deformation of the substrate was used to carry out this research. Plates of 08kp steel were used as a substrate, the geometry of which was chosen based on their further application for droplet erosion testing and determination of stresses arising in the coating under high-speed drop impact. A technique is presented for conducting research on substrates with a geometry that is changed in comparison with the classical configuration used to determine the internal stresses in the coating by the bending method. Bending values were obtained from the surface profiles obtained before and after coating, which were used to calculate stresses by using the Stoney formula. Application of the selected coating leads to the appearance of compressive stresses ranging from 3 to 9 GPa.
Keywords: stress measurement, diamond-like carbon coatings, high power impulse magnetron sputtering, droplet erosion

1. Introduction

Diamond-like carbon (DLC) coatings had a number of valuable properties that gave rise to interest in the application of such coatings in various fields of science and technology (automotive and mechanical engineering, medicine, electronics, etc.) [1]. They are quite easy to adapt to different substrates, have low resistance, high mechanical hardness, high wear resistance, chemical inertia and resistance to aggressive media, biological compatibility, good dielectric properties, and high heat conductivity [2-5]. However, in the manufacture of diamond-like coatings, it was practically impossible to avoid the appearance of residual internal mechanical stresses, which limited their use.

Internal stresses have a significant effect on the strength, adhesion, and other performance of the coatings. They arise in the coating at the forming stage, as well as in the operation of the articles due to various influencing factors, one of which is the water droplet impact, leading to erosion wear of the protected substrate material.

There are a number of methods used to investigate stresses in the material [6-9]. A widely used method for determining stresses in thin films is the bending method based on measuring the substrate deformation resulting from stresses in the coating [10-21].

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The study of changes in internal stresses in the coating after water droplet impact is possible when using substrates with geometry that meets the requirements for substrates for erosion tests at the set of research and development equipment of Unique Research Installation (URI) «Hydroshock rig «Erosion-M» NRU «MPEI».

The purpose of this work was to study the internal stresses exerted during the formation of the Ti-TiC-DLC coating using the HiPIMS technology (High Power Impulse Magnetron Sputtering) based on the developed method for measuring the deformation of a substrate with geometry changed in comparison with the classical configuration.

2. Materials and methods of research

To study internal stresses using the bending method, 08KP sheet steel substrates in the form of plates with a size of 10 × 20 × 1 mm were selected. The plates had holes for their attachment in the tooling during coating and in the holders for subsequent erosion tests on the URI «Hydroshock rig «Erosion-M» NRU «MPEI» (see Fig. 1).

The initial surface of the plates (hereinafter referred to as substrates) was subjected to pre-abrasive treatment, after which the primary surface profiles were measured. Profiles were measured on a Dektak 150 mechanical profilometer in two mutually perpendicular directions in accordance with the diagram (see Fig. 2). The scheme for measuring the curvature of the substrate is due to its geometry and the intended location of the erosion "trace", which is formed on the surface due to the high-speed water drop impact of the mono-dispersed flow.

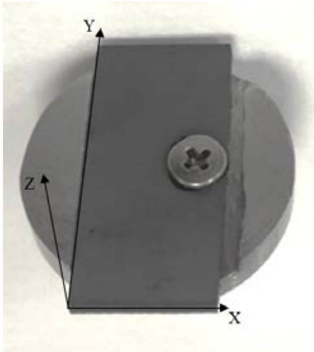

Fig. 1. Attachment of substrate in erosion test holder

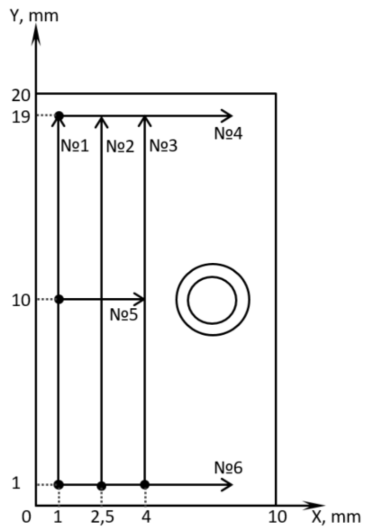

Fig. 2. Diagram of substrate surface profile measurement

The length of the profiles, when measured along the long side of the substrates (along the Y-axis) was 18 mm, when measured along the short side (along the X-axis) – from 8 to 4 mm (the length of the profiles is reduced due to the presence of a fastening hole). Note here that said cross-profiles are auxiliary to assess substrate outlier unevenness (Z-axis) due to its fixation in tooling at the subsequent application of cover and at fixation in the holder at erosion test with due account being taken of a relatively large area of fasteners relative to the width. Longitudinal profiles are intended for subsequent assessment of the effect of water drop impact action on substrate bending. Their location was determined based on the fact that the expected width of the erosion "trace" arising from the water droplet impact, the composition is 2÷3 mm, and in the future, the profiles will pass in the area of the water drops impact and near it.

After obtaining the primary (preliminary) surface profiles, a coating of the type Ti-TiC-DLC was formed on the substrates by a magnetron method on the specialized ion-plasma equipment «Gefest-HiPIMS». The coating mode was based on the study results of the main mode parameters influence the coating properties. The selected mode provides a good combination of hardness, adhesion, and tribological characteristics. For the synthesis of coatings, target-cathodes from titanium VT1.0 were used. The substrates were degreased and wiped prior to installation in the vacuum chamber. Then they were installed in a vacuum chamber, providing planetary rotation inside the plant. After that, the vacuum chamber was closed and pumped out to a high vacuum to a pressure of 10^4 Pa. Simultaneously with the pumping out of the vacuum chamber, it was heated to 200 °C to intensify degassing processes. After reaching the pressure in the vacuum chamber 10^{-4} Pa, the heating was turned off, the vacuum gate was throttled for half and plasma-forming gas was supplied to a pressure of 0.3 Pa. High purity argon was used as plasma-forming gas. On the substrates moving inside the vacuum chamber planetary, supply negative voltage (bias voltage) of the order of 1000 V and anomalous discharge was burned, ion cleaning (IC) was carried out. To intensify the IC process, 2 magnetrons for low power (up to 1 kW) were included on the surfaces of the substrates. After IC, an adhesive layer of pure carbide-forming metal – titanium – was applied to the surface of the substrates. At the same time, magnetrons worked in dual-mode at a power of up to 5 kW. The negative voltage applied to the substrates was 110-120 V. An intermediate layer of titanium carbide was applied after the adhesive layer was formed. For this purpose, reaction gas of high purity methane with a flow rate of up to 1.8 l/h was additionally supplied to the chamber through the gas inlet system. After the intermediate layer was formed, the final coating layer, DLC, was applied. For this, the flow rate of the reaction gas – methane smoothly increased in the range from 1.8 l/h to 9 l/h, while the negative bias voltage was up to 180 V.

After the coating formation, the surface profiles were re-measured in the same areas and the same length as the preliminary profiles. The determination of stresses in the coating was carried out by using the well-known Stoney formula [17-21]:

$$\sigma = \frac{1}{6}\left(\frac{1}{R_{post}} - \frac{1}{R_{pre}}\right)\frac{E}{(1-\nu)}\frac{t_s^2}{t_f}, \quad (1)$$

where σ – stress in the film, after deposition, R_{pre} – substrate radius of curvature, before deposition, R_{post} – substrate radius of curvature, after deposition; E – Young's modulus substrate material, ν – Poisson's ratio substrate material, t_s– substrate thickness, t_f – film thickness. For 08KP steel, Young's modulus was assumed to be 203 GPa, the Poisson coefficient was 0.28.

To define the radius of curvature of the substrate surface before and after coating formation (Pre-, Post-deposition) using profilometer software for the obtained primary profiles (Raw curves), the roughness component was excluded (see Fig. 3). After eliminating the roughness of the profile (Pre-, Post-deposition Data Curves), the bend of the substrate was evaluated, then it determined the radius of curvature of the surface and the amount of internal stresses.

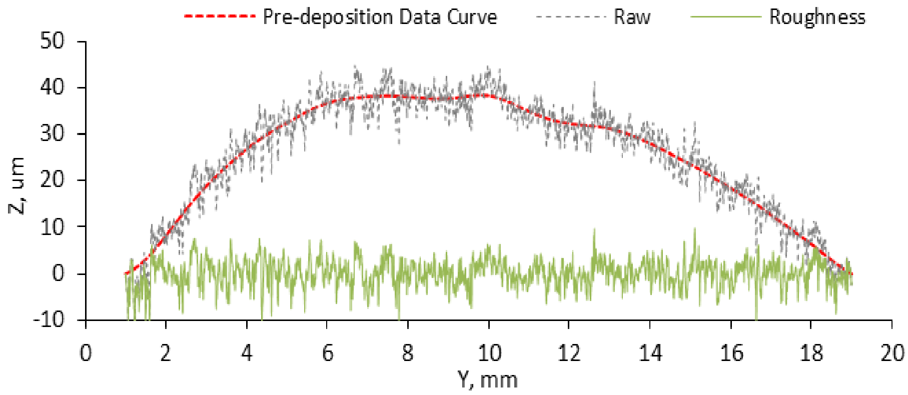

Fig. 3. Characteristic view of the division of the primary surface profile into a roughness profile and a waviness profile with macro-deviation

The surface curvature radius was estimated according to the formula:

$$R = \frac{l^2/4 + d^2}{2d}, \quad (2)$$

where R is the radius of curvature of a substrate, l is profile length (or the analyzed site of a profile), d is a vertical (in the Z-axis direction) bend of a substrate on l length. The thickness of a coating demanded calculation of internal stresses for formula (1) was defined on the metallographic cross-section slips made after measurement of profiles of a surface.

At the production of cross-sections slips the cutting of substrates on the abrasive detachable machine with a linearly mobile system of a cut PowerMet of 3000 (Buehler GmbH) was carried out. Then substrates were pressed with the use of an automatic press for a hot press-fitting of Simplimet of 1000 (Buehler GmbH) in an electroconductive compound with the high content of graphite for providing further researches of a substrate on the scanning electron microscope. Grinding and polishing of the pressed substrates were carried out on the grinding and polishing BETA/1 (Buehler GmbH) machine with an automatic nozzle of VECTOR.

The made metallographic cross-section slips were investigated on the scanning electron microscope of TESCAN MIRA 3 LMU with the cathode Schottky with field emission issue in the mode of the return reflected electrons (BSE).

3. Results and discussion

The characteristic appearance of coated and uncoated substrates is shown in Fig. 1. The initial substrates are characterized by the presence of a curvature prior to coating due to fabrication and pre-abrasion of the surface. The characteristic view of the longitudinal and transverse profiles (after eliminating the roughness profile) before and after coating formation is represented by Figs. 4-5.

The amount of vertical bending in the Z-axis direction of the substrates on cross profiles having a length of 4 to 8 mm taken in the X direction (see Fig. 2) is 3.5 to 10 μm. Defined on longitudinal, taken in the Y direction (see Fig. 2), profiles having a long length (18 mm), the total vertical bend of the images in the Z-axis direction is from 9 to 60 microns. If there are sections with a reverse bend (in-bent parts), the sign of the vertical bend and the radius of curvature determined by it was considered negative. The radius of curvature of the initial substrates varies in the range of 0.29÷0.40 m when determining the central transverse profiles No. 5 from the shortest and closest to the fastening hole. from 0.80 to 1.86 m – when determined by edge cross profiles No. 4 and 6, from 0.66 to 1.26 m – along with the profile No. 3 closest to the fastening hole, from 0.77 to 0.88 m – according to longitudinal profiles No. 1 and No. 2.

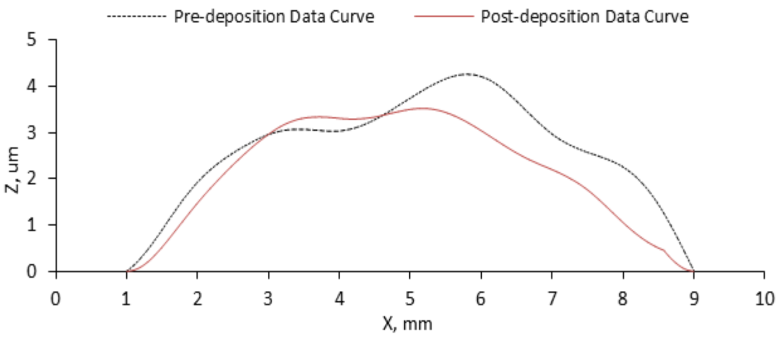

Fig. 4. Characteristic view of transverse profiles (after exclusion of roughness profile) before and after coating formation

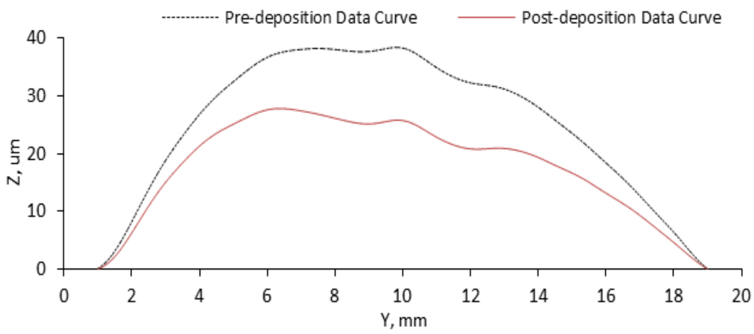

Fig. 5. Characteristic view of longitudinal profiles (after elimination of roughness profile) before and after coating formation

The ion-plasma diamond-like coating of type Ti-TiC-DLC, formed using the HiPIMS technology, according to the results of a micro-microscopic study, has a thickness that varies from 1.2 to 1.9 μm for the test batch of substrates. A characteristic view of the coating structure is given in Fig. 6.

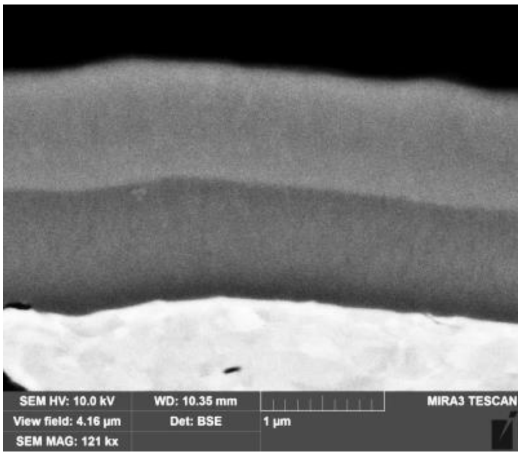

Fig. 6. A characteristic view of the Ti-TiC-DLC coating

The coating reduces vertical bending and increases the radius of curvature of the substrate. Vertical bending on transverse profiles having a length from 4 to 8 mm after application of the coating on different substrates is from 1.5 to 8 mcm. The bend defined on the longitudinal profiles having a length of 18 mm is 7 to 57 μm. The radius of curvature of coated specimens Ti-TiC-DLC ranges from 0.31÷0.44 m when deter-mining the central transverse profiles No. 5 from the shortest and closest to the fastening hole, from 1.0 to 3.0 m – at determination by edge cross profiles No.4 and No. 6, from 0.7 to 2.47 m – along with the closest of the longitudinal profile No. 3 to the fastening hole, from 0.79 to 1.1 m – according to longitudinal profiles No. 1 and No. 2.

The surfaces of the value of average internal tension calculated by a formula Stoney (1) for various substrates on various profiles in a covering of Ti-TiC-DLC fluctuate in the range from 1 to 10 GPa. The average (for the entire batch of samples examined) stress values obtained from longitudinal and transverse surface profiles in different substrate regions for erosion testing are shown in Figs. 7-8. At the same time the dispersion of values of internal tension if to compare various substrates in similar areas, is from 1.5÷3 of GPa for longitudinal profiles and up to 3÷5 of GPa for the cross.

When using the considered geometry of the samples to further follow the change in stresses during erosion tests, it was proposed to exclude from the assessment the pros closest to the fastening hole (longitudinal profile No. 3 and transverse profile No. 5) due to the greatest dispersion of the values obtained. In case of exclusion from the calculation of these profiles, the average value of stresses in the considered coating Ti-TiC-DLC in the longitudinal direction will be 3.0 GPa, in the transverse direction will be 9.0 GPa.

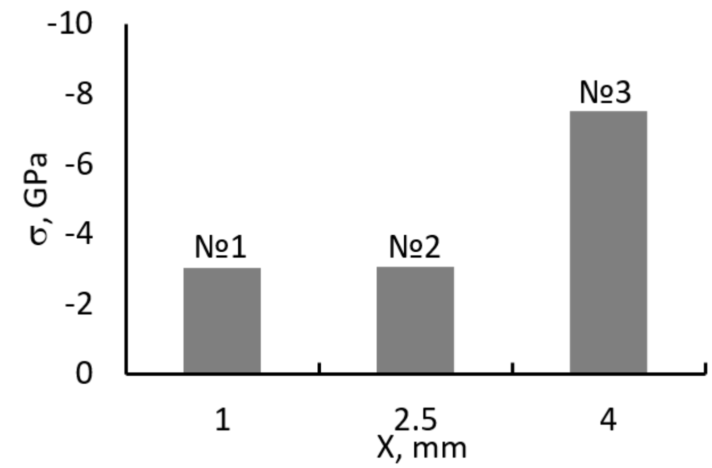

Fig. 7. Mean stress values obtained for different substrate regions for erosion testing on longitudinal surface profiles

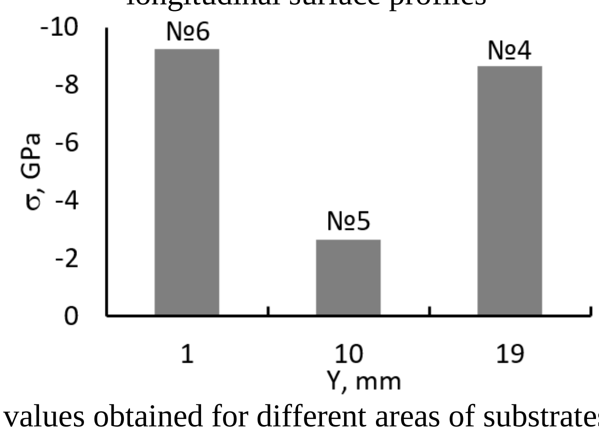

Fig. 8. Mean stress values obtained for different areas of substrates for erosion tests on transverse surface profiles

4. Conclusion

In the work, studies of internal stresses in the Ti-TiC-DLC coating obtained by using the HiPIMS technology were carried out. Pressure studies were carried out by using a developed technique for substrates with geometrics other than the classical configuration used in determining internal stresses in the coating by bending. According to the profiles on the top of the substrates obtained before and after the coating in the longitudinal and transverse directions, bending values were obtained, which were used to calculate curvature and stresses according to the Stoney formula. As a result of conducting studies of a batch of samples, it was revealed that the application using the HiPIMS technology of the selected ion-plasma diamond-like carbon coating of the Ti-TiC-DLC type leads to compressive stresses from 3 (in the longitudinal direction) to 9 GPa (in the transverse direction).

The obtained high values of stresses in the coating can be associated with a non-excluded contribution to the determined bending of temperature deformations in the material of the substrates themselves, which occur during heating due to the process of coating formation. To take this into account, it is further planned to evaluate the bending of the substrates after the technological process, carried out with a protective shield installed for a number of samples, preventing the application of the coating, but preserving the remaining influencing factors. Nevertheless, to assess the effect of the water drop impact on the deformation of coated samples, only the surface condition preceding it is important, so overstatement of the stress value in the coating will not affect it.

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