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Determination of mechanical properties of finely-divided protective coating

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Abstract. The way of peristaltic pump unit capacity enhancement by the sputtering of protective coating at the most wear off subject parts is represented. Plasma sputtering of protective coating is an efficient way of part wear prevention and also restoration of damaged parts. For this hardening method a developed industrial unit of finely-divided protective coating plasmas sputtering (IU FDPCPS) is given in the article. The development of the unit is a topical issue due to the fact that the existing units either do not implement the function of plasma sputtering or implemented not to the fullest extent. The sequence of operations for the coating of required thickness is described. The objective of the thesis is the quality assessment. For this the mechanical properties of different types of coating were defined. To find a solution, the methods of defining of the mechanical properties of the coated surfaces are given. As a result, it was found out that among latten alloy, titanium and molybdenum, latten alloy is the most suitable material for sputtering to protect the pair from friction and tear off.

1 Introduction

Modern industry developing is quote dynamic and it introduces new technologies in all areas to obtain high-quality products at minimal cost. Development occurs using mathematical models in design and processes automation [1]. To improve durability and efficiency, special attention must be paid to resources efficient use.

One of the important developments in the field of pump units are peristaltic pumps. Their unique properties make them impossible to replace in such areas as medicine, pharmaceuticals, food and chemical industries, including treatment facilities, construction, aeration, vacuuming, crude oil refining, pumping aggressive and contaminated media from tanks and settling tanks [2, 3]. Peristaltic pumping units (PPU) consist of a drive that starts the rotation of the rotor; tube (hose) through which the medium moves; rotating rollers mounted on the rotor using a self-lubricating ring [4]. To ensure trouble-free operation of pumping systems, they are subject to strict requirements, including durability.

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At long-lasting operation, pump components such as the rotor, diaphragms, hoses and shoe can wear off, resulting in reduced efficiency and more frequent repairs [5, 6]. Failure to follow the operating instructions or use in unintended conditions can cause deformation or breakage of pump elements. Failure of peristaltic pumps can result in significant financial losses related to the shutdown of processes requiring constant or precise pressure.

One of the promising methods for increasing the performance of products is the protective coatings spurring [7, 8]. This process makes it possible to improve the properties of materials and protect against wear and corrosion [9, 10]. One type of coating is finely dispersed, which is a thin layer of material consisting of nanoparticle domains.

Interest in fine coating (FC) arouses due to its unique combination of properties. Nanoparticles used for coating improve adhesion and adhesion to the surface of the material. Additionally, finely dispersed coating can change the optical, electrical, magnetic and chemical properties of materials, depending on the composition and structure of nanoparticles [11].

Consequently, applying a finely dispersed coating to the most loaded and wear-prone parts of the PPU will increase its working life. In the thesis, coating spurring is carried out using a developed industrial unit for finely-divided protective coating plasms sputtering (IU FDPCPS).

The main difficulty when applying a coating is the selection of technological parameters depending on the parameters of the incoming raw materials and the required properties. After establishing the necessary spurring parameters and the spurring process itself, it is necessary to carry out tests to determine the mechanical properties of the resulting coatings [12].

In this thesis, molybdenum, titanium and latten alloy were coated. To study their mechanical properties, a series of tests were carried out: surface hardness, roughness, resistance to thermal cycling, tribological tests and abrasive wear tests.

2 Research methods

Currently, many methods are used to apply protective coatings to prepared substrates. These methods are classified according to the working medium through which sputtering is made.

When sputtering is formed using the interaction of ions, not only physical processes but also chemical reactions occur. In particular, reactions in plasma with charged noble gas particles. Such methods make it possible to give the surface numerous variations in properties depending on the demand. The disadvantage of ion methods is the difficulty of plasma stabilizing as well as the relative complexity of the equipment.

Gas-thermal methods have a wide range of variable sputtering properties which makes it possible to control the quality of coating. This allows you to vary the characteristics of reinforcing coatings. The main advantage of plasma sputtering over other methods is the ability to work with a large number of materials, both hardened and sputtered.

Both, gas-thermal sputtering method and the ion-plasma sputtering method satisfy the basic criteria of progressiveness. To great extent it refers to continuous hardening processes. However, the key advantages of the ion plasma method are high efficiency, wide application possibilities, the ability to optimize productivity and reduce human resources spent during production. This method is proper for designing an industrial unit of finely-divided protective coating plasms sputtering, fully satisfying all the necessary requirements for various tasks that makes it one of the most appropriate options.

2.1 Design of IU FDPCPS

IU FDPCPS is intended for applying protective fine coating.

The composition of IU FDPCPS is presented in Figure 1.

The service unit serves to supply power to the FDPCPS control unit and control the sputtering process. Thermal stabilizer connects the service unit using water hoses. Thermal stabilizer serves to cool the microplasmatron. To organize the gas system, an argon cylinder is connected to the service unit using a gas hose. The hydraulic and gas systems are connected to the service unit using gas and water hoses. Pedal designed to turn on the powder supply.

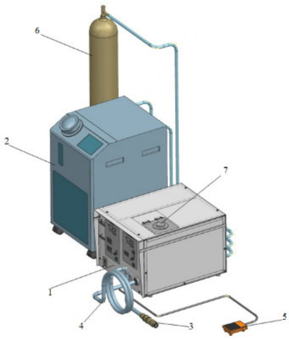

Fig. 1. Design of an industrial unit: 1 – service unit, 2 – thermal stabilizer, 3 –microplasmatron, 4 – hose, 5 – pedal,6 – gas cylinder, 7 – hatch box with dispenser.

2.2 FC Application Technology

The technological process of applying fine coatings using IU FDPCPS consists of the following technological operations:

1. Determination of the parameters of the technological regime for applying FC depending on the parameters of the incoming raw materials and the required properties of the coating. These parameters are determined based on Eq. 1, obtained by the empirical method.

$$H = 50.63 + 4.88I - 1.88G - 1.38L - 0.07R, \quad (1)$$

where H is the required coating thickness, µm;

I – discharge current, A;

G – consumption of plasma-forming gas l/min;

L – distance to the sprayed part, mm;

R – powder consumption, g/h.

2. Preliminary preparation of FC raw materials is carried out by calcining powder materials in a dryer at a temperature of 120 - 130 °C for 1.5 - 2 hours.
3. Preparation of the base surface is carried out by cleaning, degreasing and abrasive blasting of the spurred surface of the part.
4. Coating application consists of the following operations:

- Ionization of spurred particles of coating raw materials that occurs during plasma start-up.
- Spurring coating particles onto the prepared base.
- Solidification of FC at room temperature.
- Technological control of the finished coating is carried out by visual inspection of the coating.

The resulting coating must be continuous, uniform in color, without cracks, peeling, swelling or other defects.

This way, experimental samples of molybdenum, titanium and latten alloy coatings deposited on metal plates made of C22 steel were obtained.

To study their mechanical properties, a series of tests were carried out. The samples were pre-conditioned for 24 hours at ambient temperature (20±2) °C and relative humidity (50±5) %.

2.3 Carrying out tests to determine the mechanical properties of prototypes

The study of the surface hardness of the experimental sample (ES) of FC is carried out in accordance with ASTM E92-17 Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials to select the best properties of coatings of the structural elements of the PPU on the PMT device.

The sample is fixed in the device. It is required that during testing it remains stationary and at the same time located perpendicular to the axis of the diamond tip. An indenter in the shape of a tetrahedral pyramid is pressed into the sample with a given load.

The sample is removed from the stage and the absence of traces of material deformation on the back side of the sample, visible to the naked eye, is checked.

Next, using an electron microscope, the size of the prints is measured in a bright field with a magnification lens of 200–400x (numerical aperture A = 0.65). Measurements are made in 5-10 places on the surface of the test object. The centers of the prints should be located in the designated areas.

Then, using Eq. 2, the microhardness number is calculated, which is equal to the ratio of the normal load F (N) applied to the diamond tip to the conditional area of the lateral surface of the mark made S(mm^2).

$$HV = \frac{F}{S} = \frac{0.102 \cdot 2F \cdot \sin\left(\frac{d}{2}\right)}{d^2}, \quad (2)$$

where d is the arithmetic mean of the lengths of both diagonals of the square print, mm.

The microhardness of the material under study is calculated as the ratio of the sum of microhardnesses at different points of the sample to the number of measurements.

ES FC tests to determine roughness are carried out in accordance with ASTM D4417-21 Standard Test Methods for Field Measurement of Surface Profile of Blast Cleaned Steel1 using a profilometer of the 1st degree of accuracy.

The surface roughness parameters of the ES FC are selected according to ASTM D4417-21:

- R_a – arithmetic mean deviation of the profile.
- R_{max} is the distance between the line of protrusions and the line of depressions of the profile within the base length.

The test sample is installed on the profilometer. The sensor is brought to the sample and the profilometer is adjusted.

The tracing parameters are set and the trace is started. The main specified tracing parameters are: trace scanning speed: 0.5 mm/s; filter for eliminating interference when measuring a profile: λ_b = 0.8 mm; accurate R_t scale: 50 (for measuring small roughness: less than 2 μm); tracing length: 4 mm. The distance between measurement sections must ensure practical uncorrelatedness of the roughness parameters determined on adjacent traces, and must correspond to a length of at least 8 mm.

The arithmetic mean deviation R_{amid} of the ES FC profile is calculated as the arithmetic mean of the obtained R_a values of all traces of the ES FC under study. The highest height R_{max} of the ES FC profile is determined as the largest value of the obtained heights R_{max} of all traces for each ES FC.

A study of the resistance of ES FC to thermal cycling is carried out to select the best characteristics of coatings for PPU structural elements. ASTM D6944-15(2020) is used for testing.

Tests are carried out under the following conditions in a climate chamber of heat and cold: temperature varies from 10 °C to 40 °C with a constant heating rate of 5 °C/min; atmospheric pressure from 760 to 800 mm Hg, relative humidity 75%.

In the process of preparing the climatic chamber of heat and cold, a test run of the installation is carried out, checking the temperature sensors, and debugging the temperature loading cycle of the ES FC as follows:

1. Heating of samples to the maximum temperature T_{max} = +40 °C at a speed v = (2 ± 0.5) °C/min.
2. Cooling of samples to a minimum temperature T_{max} x = +10 °C at a rate v = (2 ± 0.5) °C/min.
3. Heating of samples to the maximum temperature T_{max} = +40 °C at a speed v = (2 ± 0.5) °C/min.
4. Cooling of samples to a minimum temperature T_{max} = +10 °C at a rate v = (2 ± 0.5) °C/min.
5. Heating of samples to the maximum temperature T_{max} = +20 °C at a speed v = (2 ± 0.5) °C/min.

The test objects are installed in the climatic chamber in such a way that the distance from the walls of the climatic chamber to the surface of the sample is at least 10 mm.

After thermal cycling, the experimental FC samples were removed from the climate chamber and examined using a LIP-3-10x magnifying glass to identify possible defects in the form of delamination or destruction of the FC.

The coefficient of friction was measured with a CSM INSTRUMENTS tribometer (TRB), according to ASTM G99-17 Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus. The counter samples required to create a friction pair with ES FC are a ball with a diameter of 6 mm, made of a harder material than FC. Standard counter samples supplied with the CSM INSTRUMENTS (TRB) tribometer are used, namely HP silicon nitride. Before testing, a lubricant is applied to the pre-cleaned surface of the counter sample.

The material under study, together with the counter sample, is fixed in the tribometer and fixed using fasteners (Figure 2). It is important to avoid rubbing off the lubricant from the counter sample. The following condition must also be met: the minimum distance between the side end of the sample being tested and the middle of the counter-sample zone bordering the sample must be at least 10 mm.

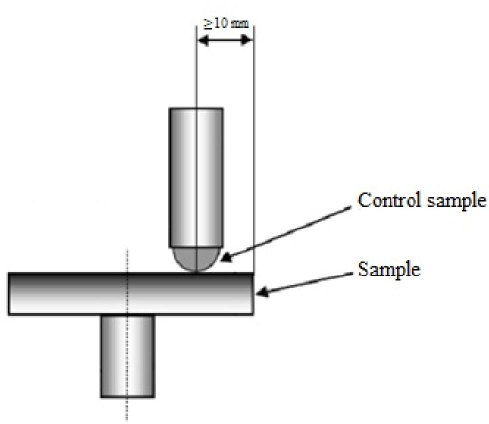

Fig. 2. Installation of the sample (ES FC) and counter sample.

The sample and counter-sample are brought together until a common contact is formed. An axial force equal to 1 N is applied to the material under study, while the counter sample rotates at a frequency of 2 r/min. After turning on the electric drive of the tribometer, the idle friction force was measured, then the samples were loaded with a force equal to 10 N. Using a tribometer in automatic mode, the average value of the friction co efficient in the area of contact between the samples was measured.

Next, the rotation of the counter sample stops, the applied load is removed, and it moves to the middle of the sample. In this case, the following condition must be met: in subsequent measurements, the gaps between different traces must correspond to a minimum length of 5 mm.

The friction coefficient of the ES FC is calculated as the arithmetic mean of all values of the friction coefficient of the corresponding pair of ES FC and the counter specimen.

The CSM INSTRUMENTS (TRB) installation was used as an installation for determining resistance to abrasive wear; counter samples were used similar to those used in tribological tests.

The mass of the sample was measured on a laboratory scale, and the thickness of the sample at three points using a micrometer. The sample is coated with lubricant and then steel wool is used as an abrasive. The material, the coating of which will be tested, together with the counter sample is fixed with fasteners in the device. As in the above test, it is necessary to avoid reducing the amount of lubricant on the sample.

The CSM INSTRUMENTS (TRB) device allows you to adjust certain values. When rotating the sample, the axial loading force must correspond to 20 N and last for at least 10 s. at a sample rotation speed of 2 r/min. After measurements are completed, the samples must be removed from the unit and cleaned with acetone. The mass of the sample is measured on a laboratory scale and the thickness at the point of contact with the counter sample at three points using a micrometer.

These tests are carried out three times. When measuring the initial thickness of a sample, the thickness is measured only in places where there was no previously contact between the sample and the counter sample. When creating contact between the sample and the counter-sample, it must be taken into account that the points at which measurements are taken must be located more than 5 mm from each other.

Next, the calculation was performed as follows:

a) The average thickness of ES before testing $h_{b.mid}$ is calculated as the arithmetic mean of the thickness of ES before testing, measured at three points.

b) The average thickness after testing $h_{a.mid}$ is calculated as the arithmetic mean of the thickness of the ES after testing at the points of contact between the ES FC and the counter-specimen, measured at three points.

c) Linear wear Δh of ES FC is calculated using formula (3):

$$\Delta h = h_{b.mid} - h_{a.mid} \tag{3}$$

d) The average linear wear Δh of each ES FS is calculated as the arithmetic average of linear wear for all measurements of the corresponding ES FC, performed according to Eq.3.

e) Determination of wear by mass Δm ES FC is calculated using Eq. 4:

$$\Delta m = mb - ma \tag{4}$$

where mb, ma – mass of the sample before and after testing (respectively), mg.

f) Average wear by weight $\Delta m$is calculated as the arithmetic average of wear by mass, based on all measurements of the corresponding ES FC, performed according to formula (4).

3 Test results

Tests were carried out using the above methods to determine the characteristics of the FC. The obtained values are shown in Table 1.

Table 1. Microhardness of FC.

Name of the test sample	**Arithmetic mean value of microhardness, HV, [MPa]**	**Arithmetic mean deviation of the profile R_{amid}, [μm]**	**Maximum profile height R_{max}, [μm]**	**Average value of the friction coefficient of the test object, $k_{fr.mid}$**	**Average linear wear $\overline{\Delta h}$, [mm]**	**Average wear by mass $\overline{\Delta m}$, [g]**
ES FC with molybdenum coating	450	0.31	1.60	0.191	0.016	0.003
ES FC with titanium coating	322	0.28	1.60	0.175	0.015	0.001
ES FC with brass coating	172	0.27	1.52	0.118	0.009	0.001
ES without coating	237	0.32	1.73	0.163	0.012	0.001

Thermal cycling tests were carried out, after which no defects or changes were found on any of the samples, including the sample without coating.

It was found that within the framework of the technology used, finely dispersed coating made of latten alloy has the lowest hardness, the lowest surface roughness, the lowest friction coefficient, the lowest wear value, which will ensure the least wear of the pair of which this coating is one element.

Molybdenum coating has the highest hardness, the highest coefficient of friction, and average linear and mass wear. This coating is not recommended for use on rubbing elements.

Uncoated steel plate has average mechanical properties.

Thus, Latten coating is most suitable for spraying to protect the friction pair from wear.

4 Conclusion

Application of a protective coating by gas plasma sputtering is an actively developing technology for surface hardening, which requires equipment that allows the necessary technological operations to be performed. The work carried out a comparative analysis of various types of sputtering, and it was concluded that sputtering by the gas-plasma method will give the desired properties to the surface due to the wide range of materials used. The technology of plasma application of finely dispersed coating was described. Using this technology, three types of FC were applied: molybdenum, titanium and latten alloy coating. A comparison was made of the mechanical properties of the resulting coatings, on the basis of which it can be concluded that latten alloy coating is optimal when it is necessary to protect the friction pair element, which is the strength of this work.

The limitations of the work are the selection and study of only three types of fine coatings. In the future, it is possible to study not only the above coatings, but also other materials.

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