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REGULAR ARTICLE

Study of Antifriction Electrospark Coatings with MoS_2 for Face Seal Elements of Nuclear Power Plants

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The paper investigated the effect of electrospark alloying (ESA) on the formation of wear-resistant molybdenum disulfide (MoS_2) based coatings for use in face impulse seal (FIS) of pumping equipment operating under conditions of high temperatures, pressure, aggressive environments and radiation exposure. The coatings were obtained on samples of austenitic AISI 321 steel. Two sulfomolybdenation methods have been developed: the first method involves the preliminary application of a sulfur-containing paste before ESA processing with a molybdenum electrode; the second one involves applying the MoS_2 powder and subsequent ESA processing with a molybdenum electrode. The microstructure, microhardness, phase composition and tribological properties of the coatings have been investigated. It was established that the formed coating had a structure comprised of three layers. Those are a porous surface layer, a strengthened layer and a diffusion zone. The maximum microhardness reached 1127 HV at $W_p = 3.4$ J. The results of tribological tests according to the "ball-disk" scheme showed a decrease in the friction coefficient to 0.0078 on samples obtained by the first ESA method. The obtained results confirm the feasibility of using the ESA methods with the inclusion of molybdenum disulfide in order to increase the operational reliability and durability of FIS units under extreme operating conditions. The proposed technologies can be implemented in production of pumping equipment for nuclear, chemical and energy industries.

Keywords: Electrospark alloying, Molybdenum disulfide, Face impulse seal, Tribotechnical coatings, AISI 321 steel.

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1. INTRODUCTION

In modern technologies for pumping aggressive, toxic and radioactive liquids, in particular in nuclear power, chemical, oil refining and pharmaceutical industries, highly reliable seals for rotating units of pumping equipment, which are capable to operate effectively under extremely difficult conditions, are of key importance. Herein, weighty part is played by face impulse seal (FIS), the designs of which are based on a precise balance of the gaps between the sealing surfaces to ensure the droplet leakage or complete absence thereof [1, 2].

A critical factor limiting the FIS durability is a degradation of the sealing surfaces, which is caused by wear, corrosion, fretting, cavitation damage, and radiation-induced structural changes. In addition, the instability of the micro gaps between the end seal rings leads to a deterioration of the sealing-in and results potentially in a system fault rate. According to the statistics, more than 50 % of pump equipment failures at critical facilities are due to the destruction of seals [3, 4].

Given the limitations of traditional designs, non-contacting end seals, including those of the FIS type, are increasingly used in modern high-load pumps. Their action is based on the creation of pulse pressure in micro chambers, which provides self-regulation of the axial gap between the surfaces of the seal rings [5]. However, even in such systems, the wear of the end surfaces remains an unsolved problem that requires searching for new functional materials and coatings.

In this context, nanostructured coatings obtained by electrospark alloying (ESA), condensed ion bombarding (CIB), and coatings obtained by hybrid technologies are of particular interest [6, 7]. The prospect of methods for synthesizing coatings under conditions of concentrated energy flows also lies in creating non-equilibrium conditions with high pressure and temperature for the formation of a new phase composition [8]. Such coatings make it possible to form the strengthened surface layers, which demonstrate

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high hardness, wear resistance, corrosion and radiation resistance even under extreme operating conditions. In particular, the use of coatings based on nickel, chromium, molybdenum and their compounds (in particular MoS_2) is a promising direction, since these elements do not form long-lived radioactive isotopes, which fact is important for use in nuclear power plants [9-11].

From the perspective of materials science and condensed matter physics, the interaction mechanisms within friction zones along with the additional influence of pulsed thermal effects entail the formation of nanocomposite structures, relaxation processes, segregation of alloying elements, and microplastic deformation. These phenomena have been confirmed by experimental studies. [12-15]. With the help of super layered nano coatings, as described [13], it is possible to improve significantly the friction coefficient and thermal stability. Studies of the mechanisms for strengthening the surfaces with the use of participation of metal-matrix Nano phases [16] confirm the role of Cr, Ni and W in increasing wear resistance and hardness.

Moreover, the influence of electromagnetic and ionizing radiation on the functional properties of sealing materials is of great importance, especially as taking into account long-term exposure to radiation. This actualizes interdisciplinary research at the junction of nanomaterial science, electronic physics, tribology and nuclear engineering.

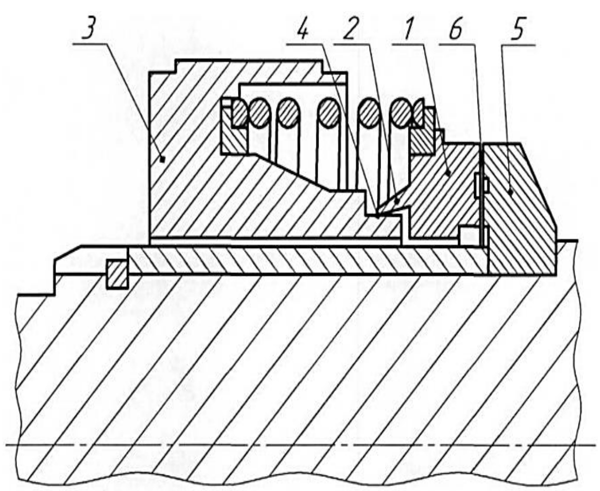


Fig. 1 – Schematic representation of the FIS: 1 – axially movable ring, 2 – ring shell, 3 – sealing sleeve, 4 – secondary seal, 5 – supporting ring, 6 – channels on the sealing surface of the ring (5)

In study [17], to increase the FIS reliability, it was proposed to install an intermediate sleeve made of F-4 fluoroplastic between sealing sleeve (pos. 3) and shell end surface (pos. 2), as shown in Fig. 1. However, despite its good antifriction properties and wide operating temperature range (from -269 to $+260$ °C), F-4 fluoroplastic material cannot be used in pump units of nuclear power plants. This is due to the fact that the temperature of the coolant in the primary circuit can reach 322 °C, and the coolant contact with radioactive water at such a temperature leads to the fluoroplastic degradation and, as a result, to the loss of sealing. In order to solve this problem and increase the efficiency of the secondary seal, an alternative design solution was proposed in [18]. It was suggested that the intermediate sleeve would be manufactured from the steel of AISI 321 grade, which is characterized by high corrosion resistance and stability under radiation exposure conditions. To reduce friction and prevent wear, it is advisable to apply a coating based on molybdenum disulfide (MoS_2) to the intermediate sleeve surface [19]. There was considered a technology of molybdenum disulfide synthesis during the ESA process using a special technological saturating medium (STSM) containing 33.3 % sulfur, with subsequent electrospark alloying by molybdenum. This method allowed the formation of a functional layer characterized by the specified antifriction and mechanical properties.

Therefore, an important scientific and applied problem is the creation of highly effective tribological coatings for elements of face impulse seal that would provide for reliable sealing, increased wear resistance, and stable operation under radiation exposure conditions.

2. MATERIALS AND METHODS

The critical parts of end seals operating under conditions of elevated temperatures, pressures, and aggressive environments of the primary circuit of a nuclear power plant are made of different grades of steel depending on the operational load. For example, the basic structural elements can be made of low and medium carbon steels, while the elements subject to intensive wear, such as seal rings, bushings, annular grooves, etc., are made of special corrosion-resistant austenitic steels, in particular AISI 321. This steel has a high thermal and corrosion resistance, and it is recommended for use in equipment operated in high-temperature and radioactive environments. In this regard, for the experimental studies, to develop technologies for modifying the surfaces of the sealing elements of the end seals, the samples made of AISI 321 steel measuring $10 \times 8 \times 8$ cm were used.

The process of ESA using a compact molybdenum electrode was carried out on an Elitron–52A type installation. The processing modes were set by a switch, while the pulse energy (W_p) being 0.13, 0.55 and 3.4 J. The process productivity was about 1 minute per 1 cm^2 of the processed area.

Two methods of sulfomolybdenation have been studied (Fig. 2). The first method consisted of preliminary application of a paste with sulfur content of 33.3 %, after which the ESA process was carried out using the same electrode. While carrying out the second method, onto the surface to be treated, molybdenum disulfide (MoS_2) powder has been applied, followed by the ESA process with a molybdenum electrode.

The metallographic study of the coatings was performed using optical and scanning electron microscopy according to the standard methods. To analyze the distribution of the chemical elements over the thickness of the coating, a local X-ray spectral microanalysis was performed with the use of a SEO-SEM Inspect S50-B scanning electron microscope equipped with an AZtecOne energy-dispersive spectrometer with an X-MaxN20 detector (Oxford Instruments, Great Britain). The distribution of microhardness in the near-surface zone was studied by the Vickers scale using a NOVA 330/360 hardness tester at the load of 20 g according to the standard method. X-ray phase analysis was carried out applying CoKα radiation with the use of a D8 Advance diffractometer (Bruker AXS, Germany). The diffractograms were obtained by step-by-step scanning with a step of 0.05° and an exposure of 3 s for each point. The phase composition was determined both on the surface and at the depth of 15 μm after preliminarily polishing.

The tribological characteristics of the formed coating were studied using a friction machine of the T-21 type in compliance with the requirements of the DIN 50324:1992-07 standard according to the "ball-disk" scheme (Fig. 3).

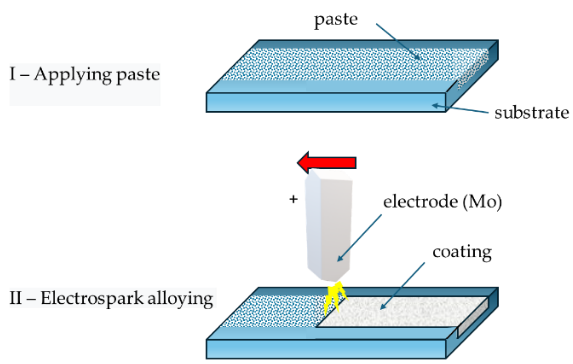


Fig. 2 – Scheme of the ESA sulfomolybdenation method

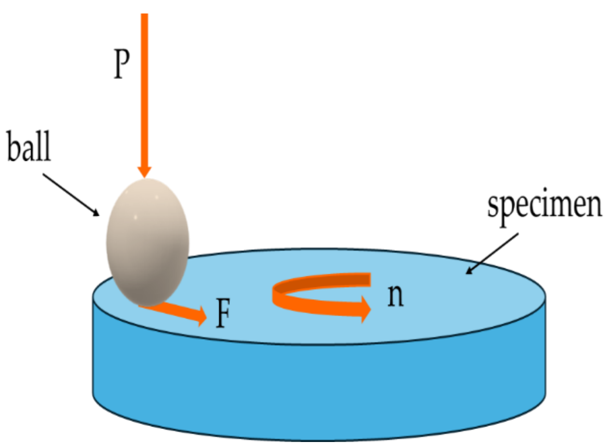


Fig. 3 – Schematic representation of tribological tests using the T-01M device

The specimens having a diameter of Ø25 × 6 mm were used for testing. The tests had been being carried out while the ball travelling a distance of $L = 4000$ m at the load of $P = 20$ N and $L = 1000$ m at $P = 40$ N, $\omega = 360.0$ rpm. During the experiment, the friction force F and the friction coefficient μ have been being recorded.

The series of the specimens for the wear resistance tests included:

– The specimens treated with a molybdenum electrode without prior application of the paste;

– The specimens treated using each of the two above described sulfomolybdenation methods.

3. RESULTS AND DISCUSSION THEIROF

3.1 Studying the Structure and the Phase Composition of the Coatings

There were carried out the metallographic studies of the coatings, which had been obtained on the AISI 321 steel using the first ESA method, namely, applying a molybdenum electrode and a sulfur-contained paste. The obtained coatings were characterized by a three-layer structure, which included as follows: a surface porous layer, a strengthened layer, a diffusion zone, and the base metal. The surface layer is uneven and has a reduced hardness (within HV 111-167), which is due to the presence of residues of the interelectrode medium (paste) and to the incomplete compaction. Under it, there is a strengthened layer formed because of melting the steel surface and interaction thereof with the alloying electrode (molybdenum) and the paste. With increasing the discharge energy, an increase in the hardness of this layer is observed: namely, from HV 515 at $W_p = 0.13$ J to HV 1060 at $W_p = 3.4$ J. This regularity indicates the activation of the processes for forming the coating and for strengthening the diffusion interaction under conditions of increasing electric spark treatment energy. This is evidenced by the increase in the thickness of both the transition and strengthened layers with increasing W_p (Fig. 4, c, d).

The metallographic studies of the coatings on the AISI 321 steel specimens by the second ESA method, namely, the one using a molybdenum electrode and a paste containing MoS_2 powder, have shown (Fig. 5) that the surface layer consists of 4 sections. Those are as follows: the dark layer is on the top, the strengthened layer is located below the above and has a thickness of up to 30 and 60 μm and a hardness of 534 and 1127 HV, respectively, after alloying thereof at $W_p = 0.55$ and 3.4 J (Fig. 6), then there is a transition zone and a base.

The electron microscope studies allowed to characterize the structure and determine the qualitative distribution of the elements in the coating (Fig. 7). The following zones are clearly visible: the dark layer, the "white" strengthened layer, the diffusion zone and the base. The local X-ray spectral analysis allowed determining that molybdenum was concentrated on the surface. With increasing energy, the intensity of the energy impact (W_p), the diffusion zone of molybdenum increases. It should also be noted that with increasing W_p, there increase the number and size of the pores, which are evenly spaced and have spherical shapes. Such a morphology of the pores and their locations can positively affect the tribotechnical performance of the coatings.

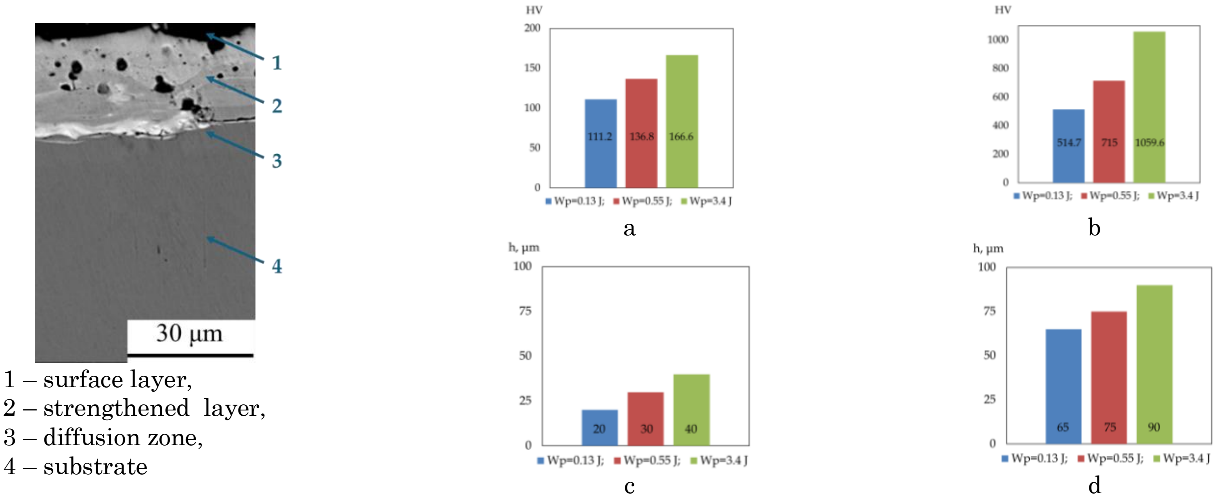


Fig. 4 – Structure and parameters of coatings on AISI 321 steel obtained by the first ESA method – using a molybdenum electrode and a paste containing sulfur: a and c – Vickers hardness and thickness of the near-surface layer (1), respectively; b and d – Vickers hardness and thickness of the strengthened layer (2), respectively

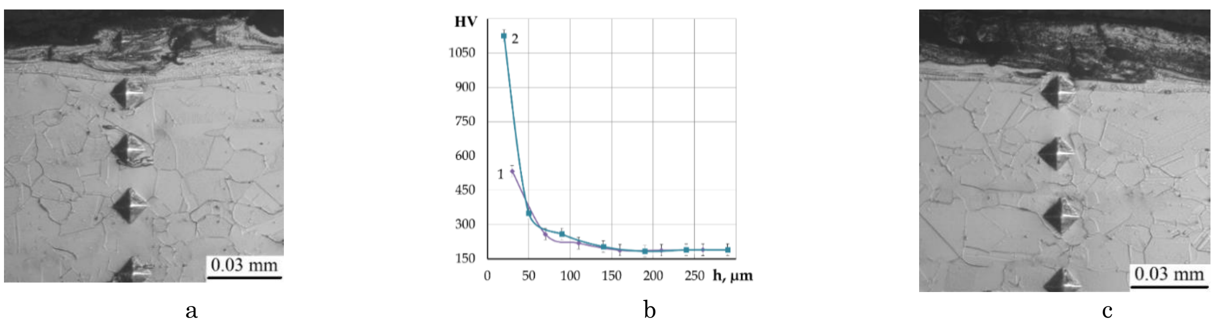


Fig. 5 – Microstructures (a, c) and microhardness distribution (b) in the coating on AISI 321 steel using the second ESA method applying a molybdenum electrode and a paste containing MoS2 powder. ESA at Wp: a – 0.55 J, b – 3.40 J. On the graph: 1 – 0.55 J, 2 –3.4 J

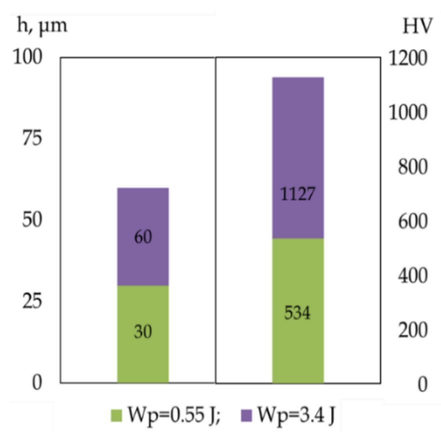
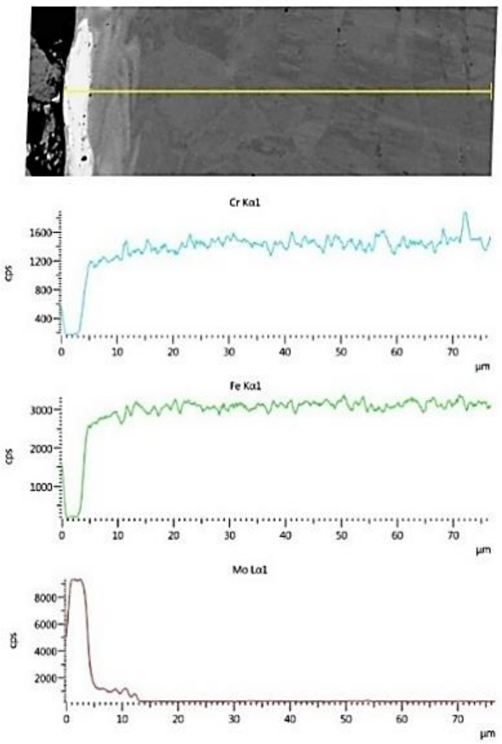


Fig. 6 – Parameters of coatings obtained using the second ESA method: maximum Vickers hardness (HV) and thickness of the strengthened layer (h) under different ESA modes

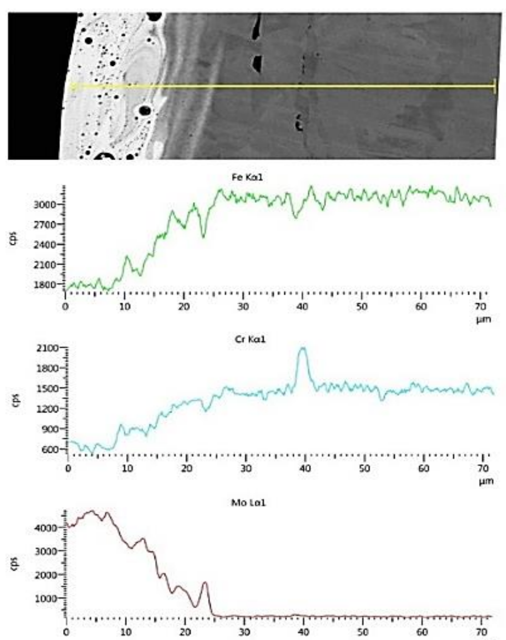
The phase composition of the coatings on AISI 321 steel after processing by the second method is represented by the bcc and FCC solid solutions, as well as molybdenum disulfide (Fig. 8). Up to 44% of molybdenum disulfide has been found on the surface, which fact can have a positive effect on the tribological properties of the coatings. With increasing Wp, the amount of molybdenum disulfide decreases. Therefore, while the ESA process at the discharge energy of 0.55 J, molybdenum disulfide is about 44%, and at that of 3.4 J, it is about 26%.

3.2 Studying the coating tribological properties

The study of the tribological properties of the coatings formed using the two different ESA methods has been conducted. Fig. 8 represents the dynamics of the changes in friction forces F, in particular the maximum (F_{max}) and average ($F_{average}$) values obtained during testing. The three series of the specimens were used as the object of testing, namely: the coatings obtained using the ESA method by a molybdenum electrode without prior application of paste; the coatings formed by the first method using the paste containing sulfur, as well as the coatings formed by the second method using the paste based on the molybdenum disulfide powder (MoS_2). The coatings being studied were obtained at the discharge energy of W_p = 3.40 J. In addition to the friction force, the friction coefficients (μ) were determined as the integral indicators of the coating efficiency values under friction conditions. The results obtained allow establishing the regularities of the paste impact and the ESA energy parameters on the tribotechnical behavior of the applied coatings.

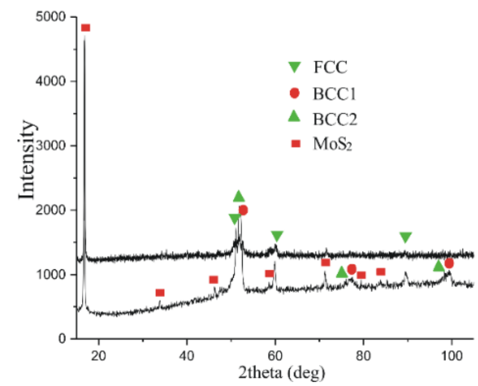


a

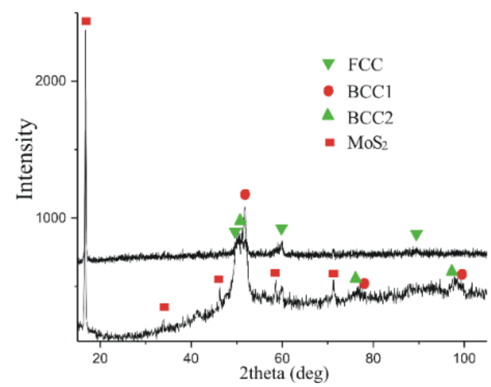


b

Fig. 7 – Distribution of elements in AISI 321 steel coatings obtained using the second ESA method, Wp: a – 0.55J; b –3.4 J



a

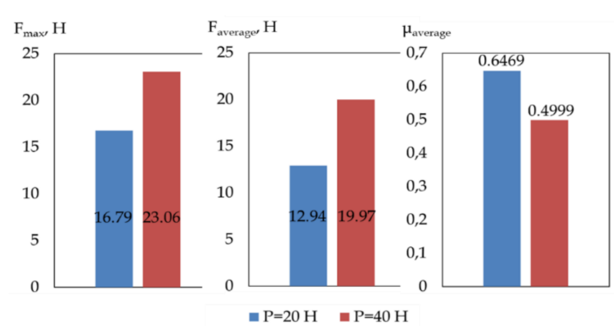


b

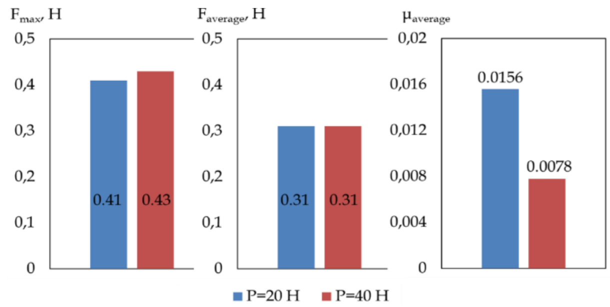
Fig. 8 – Diffractograms of 12X18H10T (12Kh18N10T) steel coatings with MoS2 powder at Wp: a – 0.55 J; b –3.4 J

For the specimens having the molybdenum-based coatings, there has been recorded an increase in the friction force with an increase in the applied load. Thus, the friction coefficient at the load of $P = 20$ N is 0.6469, while at $P = 40$ N it decreases to 0.4999 (Fig. 9). Studying the tribological behavior of the coatings formed by the first ESA method has shown that these specimens are characterized by the lowest values of both the friction force and the friction coefficient among all those having been studied.

In the case of the coatings obtained by the second method, at the load of $P = 20$ N, over time, it is observed a moderate increase in the friction force, which is probably



a



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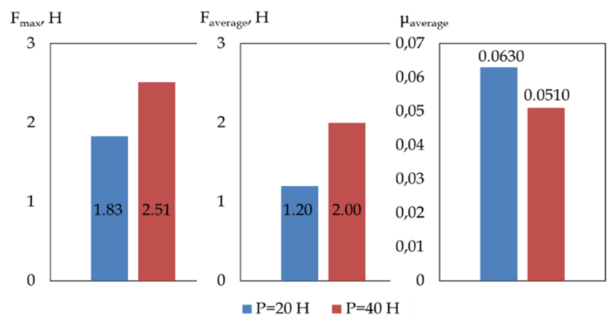

c

Fig. 9 – Changes in tribotechnical characteristics under different testing conditions: a – ESA by molybdenum; b – ESA according to the first method using a molybdenum electrode and a paste containing sulfur; c – ESA according to the second method using a molybdenum electrode and a paste containing MoS_2 powder. F_{max} – maximum friction force, $F_{average}$ – average values of friction force, $\mu_{average}$ – average values of friction coefficients

due to a longer process of the surface running-in under reduced load conditions. Thus, the coatings containing molybdenum disulfide (MoS_2) demonstrated the best results in reducing the friction force and wear intensity as compared to the coatings based on pure molybdenum.

4. CONCLUSION

1. There has been improved the ESA technology for increasing the reliability of the FIS operating under radiation exposure conditions.

2. Two methods of the ESA sulfomolybdenation have been developed and experimentally tested: those using a paste based on sulfur as well as on the MoS_2 powder. It has been found that both methods provide the formation of a strengthened layer having high tribological characteristics, however, the highest hardness (up to 1127 HV) is demonstrated by the coatings obtained using the second method (with the paste based on MoS_2) at the discharge energy of 3.4 J.

3. The metallographic studies have shown the presence of a diffusion zone, which ensures a strong connection of the coating with the base material and revealed a regular increase in the thickness of the strengthened layer with increasing the pulse energy.

4. The phase analysis confirmed the presence of up to 44% molybdenum disulfide in the surface layer. With increasing the energy, its content decreases. This fact should be taken into account when choosing the ESA modes to preserve the surface layer antifriction properties.

5. The tribological tests have shown that the coatings obtained using a paste containing sulfur are characterized by the lowest friction forces and friction coefficients (0.0078 at the load of 40 N), and also provide the lowest level of wear as compared to the coatings made of pure molybdenum.

6. The results of the study confirm the feasibility of using antifriction coatings based on molybdenum disulfide, synthesized by the ESA method, to increase the wear resistance and ensure reliable sealing in the face impulse seal of the pump equipment operating under extreme conditions, particularly in nuclear power plants.

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Дослідження антифрикційних електроіскрових покриттів на основі MoS_2 для елементів торцевих ущільнень АЕС

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У роботі досліджено вплив електроіскрового легування (ЕІЛ) на формування зносостійких покриттів на основі дисульфіду молібдену (MoS_2) для застосування в торцевих імпульсних ущільненнях (ТІУ) насосного обладнання, що працює в умовах високих температур, тиску, агресивного середовища та радіаційного опромінення. Покриття отримані на зразках з аустенітної сталі AISI 321. Розроблено дві методики сульфомолібденування: перша передбачає попереднє нанесення сірковмісної пасти перед ЕІЛ молібденовим електродом; друга – нанесення порошку MoS_2 з подальшим ЕІЛ молібденовим електродом. Досліджено мікроструктуру, мікротвердість, фазовий склад і трибологічні властивості покриттів. Встановлено, що сформовані покриття мають тришарову структуру: поруватий поверхневий шар, зміцнений шар і дифузійну зону. Максимальна мікротвердість досягала 1127 HV при W_p = 3,4 Дж. Результати трибологічних випробувань за схемою «кулька-диск» показали зниження коефіцієнта тертя до 0,0078 на зразках, отриманих першою методикою ЕІЛ. Отримані результати підтверджують доцільність використання методів ЕІЛ із включенням дисульфіду молібдену для підвищення експлуатаційної надійності та довговічності вузлів ТІУ в екстремальних умовах експлуатації. Запропоновані технології можуть бути впроваджені у виробництво насосного обладнання для ядерної, хімічної та енергетичної промисловості.

Ключові слова: Електроіскрове легування, Дисульфід молібдену, Торцеві імпульсні ущільнення, Триботехнічні покриття, Сталь AISI 321.