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## Nitriding of Long-Term Holes in the Cyclic-Committed Discharge

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**Abstract.** The effect of anhydrous nitriding in a glow discharge on microhardness, phase composition, and wear resistance of long holes in steels C45, 37Cr4, and 41CrAlMo7 with direct current supply and in cyclically switched discharge (CSD) was studied. Nitriding was carried out on a UATR-1 anhydrous nitriding unit with a discharge chamber diameter of 400 mm and a working height of 700 mm. Anhydrous nitriding in a glowing discharge was carried out at a temperature of 560 °C, a voltage of 730 V, a pressure in the chamber of 120 MPa, and the nitriding duration was 6 h. It was established that using holes with a relatively small diameter of glow discharge in a cyclically switched discharge for nitriding creates conditions for obtaining modified layers with higher physical, mechanical, and tribological characteristics. The results of microhardness measurement and their comparison with X-ray phase analysis data confirm the formation of  $\epsilon$ ,  $\gamma$ , and  $\alpha$  phases during nitriding along the entire height of the samples placed in the experimental model. The tests carried out in the dry friction mode showed an increase in the wear resistance of samples made of steel C45, 37Cr4, and 41CrAlMo7 during nitriding in a cyclically switched discharge. To achieve 100  $\mu\text{m}$  wear of 41CrAlMo7 steel during nitriding in CSD, 1400 m of friction path and 1000 m – during nitriding with direct current is required. It was established that using long holes of a glow discharge with different types of power for nitriding creates conditions for obtaining modified layers with variable characteristics. Nitriding of holes with a relatively small diameter of a glow discharge with a different power supply creates conditions for obtaining modified layers with different physicochemical and tribological characteristics.

**Keywords:** chemical-thermal treatment, glow discharge, strengthening, microhardness, wear resistance, dry friction.

## 1 Introduction

Various technologies are used to modify the internal surfaces of pairs (e.g., cementation, furnace nitriding, gas chrome plating). However, all of them have a number of disadvantages: the fragility of the surface layers, the long duration of the saturation process during furnace nitriding (96 h), the change in dimensions, and the need for further finishing during cementation. The specified disadvantages are absent when nitriding is used in a glow discharge. The process is less long than the furnace process, and when using anhydrous media, it becomes possible to meet the requirements of environmental safety and reduce the fragility indicators. However, nitriding in a glow discharge with direct current does not provide uniform surface treatment throughout the depth, and the inner surface of the hole away from the ends is practically not nitrided [2]. Therefore, the technology of anhydrous nitriding in a glow discharge with a cyclically switched power supply is

proposed. Practically all kinematic pairs of friction with translational motion, regardless of whether they are cylindrical in shape or arbitrary, structurally fall into the category of holes with a relatively small diameter. That is, the ratio of the length (depth) of the hole to its diametrical size exceeds the value of four. This indicator, accepted as a criterion of geometric ratios, is justified because, as is known, the nitriding process of similar structural elements is similar to a discharge with a hollow cathode. From the theory of this process, it is known that the actual field penetrates inside the holes to a depth of no more than two diameter sizes (if the holes are not round, then two smaller diameter sizes) [1]. Simultaneously, it should be considered that this indicator refers to the limit of the field, where the intensity is only 0.02 (about 2 %) of the nominal value at the end of the hole [2]. The numerical criterion for assigning nitriding objects to the category of holes with a relatively small diameter in the number of four diameters applies to structures in which the holes are through ones.

For blind recesses or holes, the value of the criterion can be reduced to two. Continuing to give examples of structures studied in this work, we can note plunger pairs of fuel pumps of diesel internal combustion engines and material cylinders of injection molding machines.

## 2 Literature Review

Currently, in surface engineering methods, preference is given to implementing methods of controlled modification of surfaces based on the action of concentrated energy flows. Vacuum, ion, and laser technologies, promising from the point of view of forming the structure and properties, have gained the most significant development. These surface modification methods went through several stages, which led to the creation of many technical solutions determined by the specifics of the processes and the design features of the equipment used.

One of the most developed is the method of anhydrous nitriding in a glow discharge. The paper [3] investigates the possibility of creating modified surface layers on austenitic stainless steels using low-pressure glow discharge nitrogen treatment, similar to sputtering, so that surface activation, heating, and nitrogen incorporation can occur in one step with a short duration.

In work [4], it was established that with the help of appropriate treatment parameters, glow discharge nitriding can significantly improve the corrosion resistance of austenitic stainless steels, such as AISI 316L and AISI 202, compared to untreated alloys.

In work [5], stationary helicon wave plasma with a small diameter (10 mm) was used for nitriding the inner part of a thin austenitic stainless-steel tube. The results confirmed that the nitrided layer consists mainly of the austenite phase; iron nitride is not released. Given the successful control of the plasma discharge in a thin tube with a small diameter, this research paves the way for achieving high-performance nitride layers inside thin tubes.

In work [6], low-temperature plasma nitriding was proposed as a surface treatment to increase the technical durability of stainless-steel tubes and nozzles. Various analyses were performed to describe only the internal process of nitriding, from the pipes and nozzles' inner surface to their thickness depth.

In the study [7], the authors developed technological modes of pulsed ion-plasma nitriding of internal cylindrical surfaces using a hollow perforated anode. This leads to the formation of diffusion coatings consisting of different chemical and phase composition areas.

The purpose of the work [8] was to evaluate the possibility of nitriding deep holes of small diameter. The tests were carried out on cylindrical samples of unalloyed and low-alloyed steel with electro-drilled and mechanically hollow through and blind holes.

Attempts to nitride long holes in a glow discharge with a constant power supply only confirm the above theoretical conclusions regarding the discharge spread in relatively small diameter holes. Simultaneously, the inner surfaces of

the holes near the ends are nitrided with acceptable quality, while when the distance from the end of the hole increases, the nitriding results become less and less noticeable. Some improvement in the effects of the modification can be achieved by increasing the duration of the process. That is, the effect of ordinary furnace nitriding is manifested, but simultaneously, the main advantage of nitriding in the glow discharge is eliminated - a significant reduction (by more than an order of magnitude) in the duration of processing [2].

Thus, the task set in the work has practical significance since there are many options for its actual production application. Simultaneously, one should consider that such a process has not been sufficiently studied, except for purely technological aspects [9]. The possibility of nitriding the internal surfaces of holes with a relatively small diameter can be theoretically substantiated by the pumping of nitrogen ions into the inner cavity of the hole due to the effect of their movement by inertia. Since, in the absence of an electric field, the ions will continue to move tangentially to the trajectory that took place at the moment of termination of the discharge, it becomes possible for them to reach the region of the hole cavity, where the field is practically no longer active. This phenomenon is especially characteristic of ions that fly into the hole near its center.

Simultaneously, the trajectory of their movement is significantly straightened, the ions' collision probability with the walls of the hole decreases, and they fly a much longer path than they would in the case of continuous power [10]. In this way, an excess concentration of nitrogen ions is created, which further drifts into the depth of the hole, obeying the laws of diffusion. Since nitrogen ions are the main factor in the formation of nitrides, the nitriding process of the inner surface should theoretically occur at a speed that practically corresponds to the conditions of processing open surfaces [11].

The influence of the physical foundations of the nitriding process on the contact interaction of modified friction surfaces is considered in [12]. The conducted analysis confirmed the determining importance of the structure and composition of the gas discharge environment.

The research works on using pulse discharge in nitriding processes to improve surface layers' mechanical and tribological properties. In work [13], intending to optimize the nitriding process, experimental studies of pulsed plasma nitriding of carbon steel DIN C45 (AISI C1043) were carried out using a direct current pulse glow discharge. The influence of gas composition, temperature, processing time, and frequency on layer thickness and microhardness was studied. The obtained results are recommended for optimizing the nitriding process and computer control. To increase the tribological properties of austenitic stainless steels in [14], the authors use plasma nitriding of the surface in pulse mode. It has been found that the wear rate is reduced by up to 90 % compared to the base material when processed with a low-duty cycle. It is shown that wear and corrosion resistance

can be significantly increased by reducing the pulse duty cycle.

In work [15], samples of unalloyed steels were nitrided under fixed conditions using an alternating pulse current. It was established that the hardness and wear resistance increase significantly with increased pulsed current. This study comprehensively explains the contribution of pulsed current to nitriding efficiency and plasma reactivity. In the study [16], a pulsed power supply is used for plasma nitriding to overcome the problems of direct current plasma nitriding. Therefore, using a pulsed power supply ensured more accurate control of the nitriding process, post-adjustment of the pulse width, and avoidance of the phenomenon of arcing on the surface of the workpiece. Therefore, the advantages of using a pulsed discharge as a power source during nitriding are apparent, but its use for processing long holes has not been widely used.

Due to the mentioned above, the research aims to develop a method of anhydrous nitriding in a glow discharge of long holes to form the structure of the inner surfaces of the holes, which ensures an increase in their wear resistance. To realize the set goal, it was necessary to perform the following main tasks:

- to develop a methodology for studying the impact of nitriding on the structure of the nitriding layer depending on the depth of the hole;
- to develop a methodology for conducting accelerated tribological tests of nitrided surfaces;
- to create and implement the developed scheme of nitriding in a cyclically switched discharge (CSD) with the possibility of changing the frequency (ratio of the cycle period to the duration of the signal).

### 3 Research Methodology

Experimental studies were carried out on the model. The general view is presented in Figure 1 a.

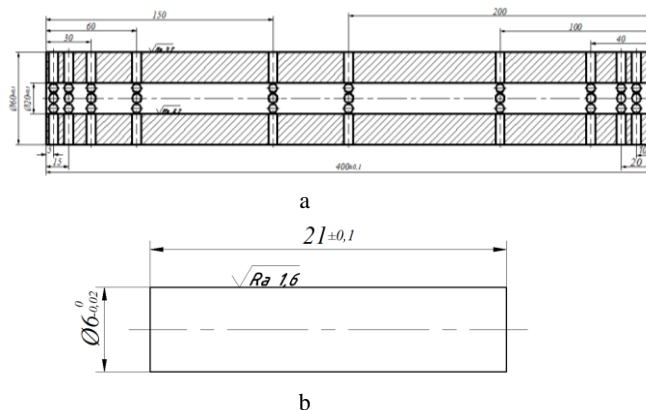


Figure 1 – Sketch of the cylinder model (a) and the sample sketch (b)

The model is a hollow cylinder in which radial holes are drilled at different distances from the end. Samples made of different steels are inserted into these holes (Figure 1 b).

Thus, each sample is nitrided from two ends, making it possible to nitride from the outside and the middle of the

model at almost the same temperature and compare the nitriding results of the two surfaces. Simultaneously, the difference in conditions is only in the location of these surfaces - external or internal. All other factors that could affect the modification results are practically identical.

The presence of a series of radial holes creates the possibility of simultaneous nitriding of samples made of different steels at the exact parameters of the technological process, which significantly speeds up experimental research.

The samples were installed in the radial holes and held there due to a certain tension. This achieved not only the retention of the samples in the holes but also the absence of burning near the ends of the samples, especially when fed with a direct current discharge. The appearance of this phenomenon is entirely accurate, as it is observed even with gaps of the order of 0.5 mm. Simultaneously, using a similar method of fixing samples greatly simplifies the model's design.

The total length of the models is 360 and 400 mm, and the diameter of the holes is 20 and 40 mm. Thus, the most significant coefficient of the ratio of the hole length to its diameter was 18 and 10, respectively.

Nitriding was carried out on a unit for anhydrous nitriding UATR-1 with a discharge chamber diameter of 400 mm and a working height of 700 mm. A nitrogen-argon mixture was used as a gas medium with a ratio of components by volume of 75 % nitrogen and 25 % argon. Anhydrous nitriding in a glowing discharge was carried out at a temperature  $T = 560$  °C, a voltage  $U = 730$  V, a pressure in the chamber of 120 MPa, and the nitriding duration was 6 h. The shape of the pulse at CSD is rectangular.

The structural steels of the following brands were selected for the study: C45 – high-quality carbon, 37Cr4 – chrome-plated, and 41CrAlMo7 – high-quality chrome-aluminum with molybdenum, as the most often used for nitriding in a glow discharge. Elemental composition studies were carried out using energy dispersive methods X-ray fluorescence spectrometer “Octopus” manufactured by “Ukrrentgen” with SDD detector X-123 (Amptek, USA). The steel grade is identified by determining the content of eight chemical elements. The chemical composition of the studied steels (ASTM A29) is given in Table 1.

Table 1 – Chemical composition of the structural steels, %

Steel	Content of alloying elements, %					
	C	Si	Mn	Cr	Ti	Al
C45	0.42–0.50	0.17–0.37	0.50–0.80	≤0.25	–	–
37Cr4	0.36–0.44	0.17–0.37	0.50–0.80	0.80–1.10	–	–
41CrAlMo7	0.35–0.42	0.20–0.45	0.30–0.60	1.35–1.65	0.15–0.25	0.70–1.10

The conducted studies of samples on the ability to absorb argon in the nitriding process showed the absence of argon in the surface layers of steels. Therefore, the solubility of argon in the studied steels is absent, and therefore, the obtained results are related exclusively to the interaction at the separation boundary of the activated nitrogen gas mixture with the surface of the steel.

After nitriding, the control samples are cylindrical in diameter of 5 mm and length of 20 mm, cut in half in the diametrical plane at cutting modes that ensured the invariance of the structure of the modified surface layer.

The samples were installed in the radial holes and held there due to a certain tension. This achieved not only retention of the samples in the holes but also the absence of burning in the vicinity of their ends when fed with a direct current discharge. The danger of this phenomenon is quite real, as it is observed even with gaps of the order of 0.5 mm. Simultaneously, using a similar method of fixing samples significantly simplifies the model's design, excluding devices such as screw clamps and collets.

The parameters of the technological mode are presented in Table 2.

Table 2 – Technological parameters of nitriding

Mode	$T$ , °C	$U$ , V	$P$ , MPa	Duration, h	Features
1	560	730	120	6	CSD; the model is open from 2 sides
2	560	730	120	6	The model is open from 2 sides
3	560	730	120	6	1 end is closed

In mode 1, CSD was used; in modes 2 and 3, constant power was used.

The processing of nitriding results primarily involved measuring the surface microhardness on a PMT-3 microhardness tester. The surface microhardness was studied not only on the samples' ends but also along the depth of the modified layer. Microhardness measurements were performed at a distance from the surface of 25, 50, 75, 100, 150, 200, 250, 300, and 600  $\mu\text{m}$ .

The X-ray phase analysis was carried out on an X-ray diffractometer general purpose of DRON-3 in the filtered radiation of an iron anode, in the range of  $2\theta$  angles from  $20^\circ$  to  $100^\circ$  with a scan step of  $0.1^\circ$  and an exposure time of 10 s. The X-ray imaging was performed from the flat ends of cylindrical samples subjected to nitriding to the depth of the modified layer.

Experimental studies of samples for wear resistance in the dry friction mode were carried out on a universal machine for testing materials for friction, model 2168UMT. The material of the counter body is steel 52100 with a base hardness of HRC61; pressure in the contact zone  $P = 16$  MPa; sliding speed  $v = 0.1$  m/s; the controlled parameter is linear wear  $h$ , which was determined as a

change in the linear size of the sample measured normal to the friction surface because of passing section length  $l$ .

As the friction path passed, the step of fixing the test results changed (Table 3).

Table 3 – Periodicity of measurement of test results

Friction path, m	0–50	50–200	200–400	400–1000
Measurement step, m	5	10	25	50

Tests were stopped when catastrophic wear occurred.

## 4 Results

Figures 2 a–b show the change in microhardness ratios along the depth of the modified layer, respectively, from the side of the outer ends to a similar indicator measured at the same depth but from the inner side for mode 1 (Figure 2 a), and Figure 2 b – for mode 3.

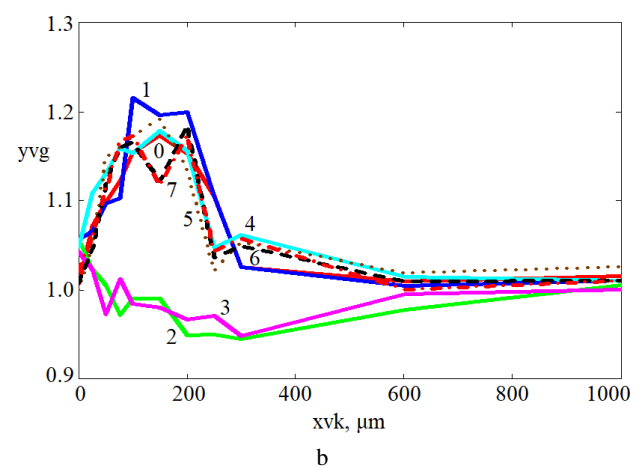
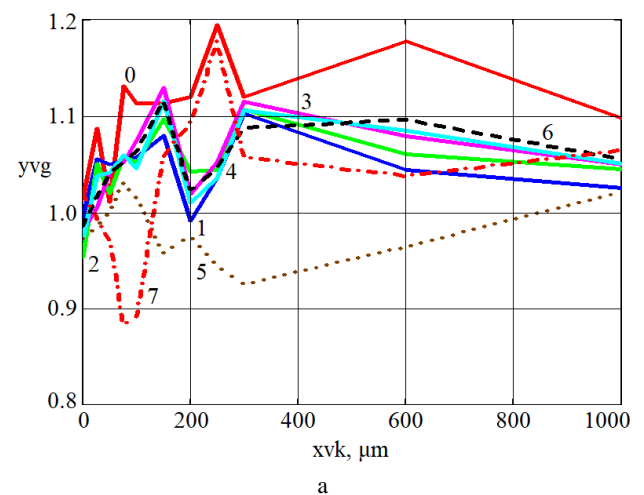


Figure 2– Distribution of the ratio of microhardness (external side to the internal) by the depth of the modified layer on steel 37Cr4 for modes 1 (a) and 3 (b)

Simultaneously, the location of the graphs on the vertical scale corresponds to the distance of the samples from the end of the model.

The designations used in the work drawings are as follows: y-axis – I.1.194 (ends of the model); yvk0 – 5 mm

(the center of the hole for the sample is 5 mm away from the end of the model); yvk1 – 10 mm, yvk2 – 15 mm, yvk3 – 30 mm, yvk4 – 40 mm, yvk5 – 50 mm, yvk6 – 60 mm, yvk7 – 100 mm, yvk8 – 150 mm, yvk10 – 200 mm; y-axis is the depth of the nitrided layer,  $\mu\text{m}$ .

Analysis of the ratio of microhardness from the outside to a similar indicator from the inside, depending on the distance to the open face of the model, showed that the excess of the ratio indicator for mode 2 increases with increasing distance from the face, while for mode 3 it remains unchanged around unity.

Comparison of Figure 2 a and Figure 2 b testify to fundamentally different results of nitriding since in mode 3, the coefficient of the microhardness ratio is significantly larger and, in some cases, exceeds 1.2. Additionally, the nature of the ratio distribution along the depth of the nitrided layer is different since in mode 3, at a relatively small depth, the microhardness values are practically equalized, while in mode 1, the unevenness persists to great depths.

Analysis of the ratio of microhardness from the outside to a similar indicator from the inside, depending on the distance of the sample under study to the open end of the model, respectively, for modes 2 and 1. showed that the excess of the ratio indicator for mode 2 increases with increasing distance from the end, and for mode 1 (as an average the value of all values) remains unchanged around unity.

Figure 3 presents the change in the average value of the ratio of microhardness at a certain depth of the nitrided layer for steel C45 to a similar indicator on the end of the sample ( $m$ ) for mode 1 is shown (dashed line (a) – outer side, solid line (b) - inner side), similarly - in Figure 3 b – for mode 2, in Figure 3 c – for mode 3, and in Figure 4 – a comparison of the average values of the outer and inner sides for all three modes.

It follows from Figure 3 that the nature of the dependence of the ratios is significantly different for certain modes. In all versions of the technology, there is a difference in the values for the inner and outer sides of the samples. The nature of the dependence of the ratios is significantly different for certain modes.

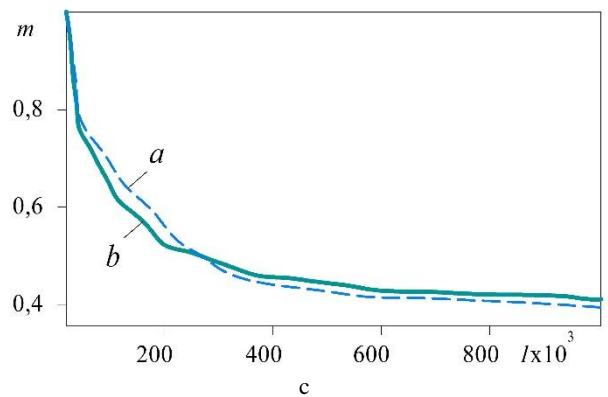
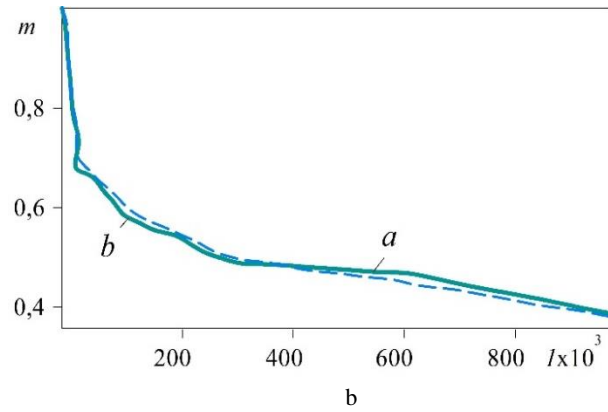
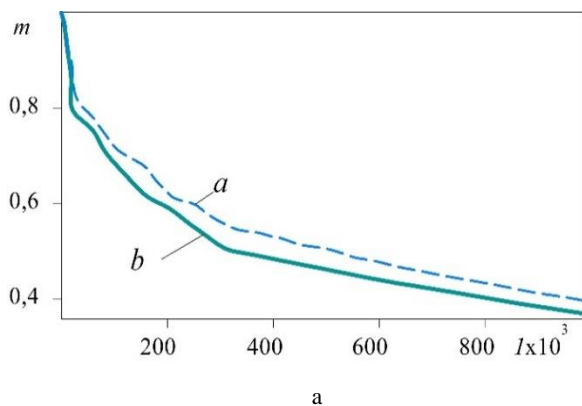


Figure 3– The ratio of microhardness at the depth of the modified layer to microhardness for steel C45 at the end for modes 1 (a), 2 (b), and 3 (c)

In all versions of the technology, there is a difference in the values for the inner and outer sides of the samples. The nature of the dependence of the ratios is significantly different for specific modes. In all versions of the technology, there is a difference in the values for the inner and outer sides of the samples.

In mode 1 (Figure 4), the microhardness distribution is better (upper curve) because the microhardness decreases more gradually and smoothly over the depth of the layer compared to other modes.

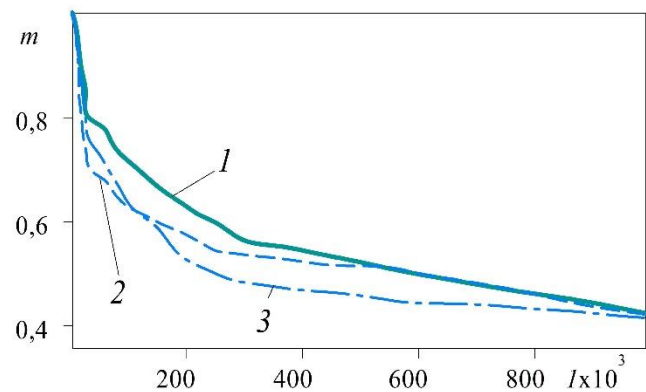


Figure 4– Relative microhardness along the depth of the nitrided layer of samples made of steel C45 strengthened by modes



Figure 5 shows the results of measuring the microhardness of the inner surface of C45 steel along the height of the pipe for different processing modes.

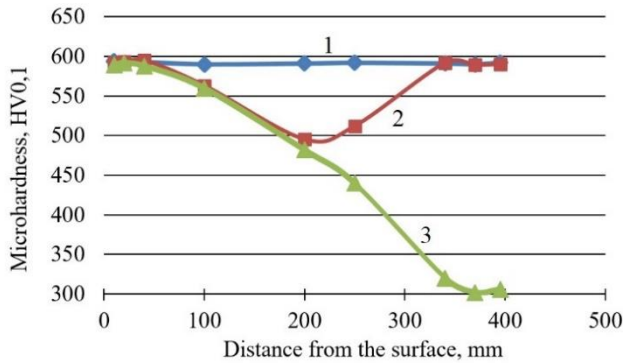
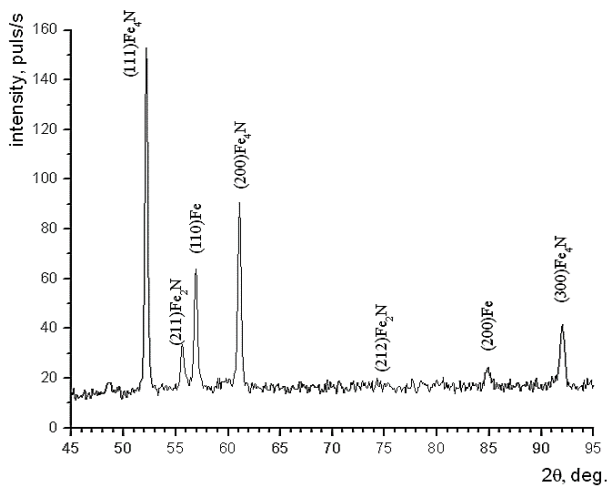


Figure 5– Distribution of microhardness along the height of the pipe for steel C45

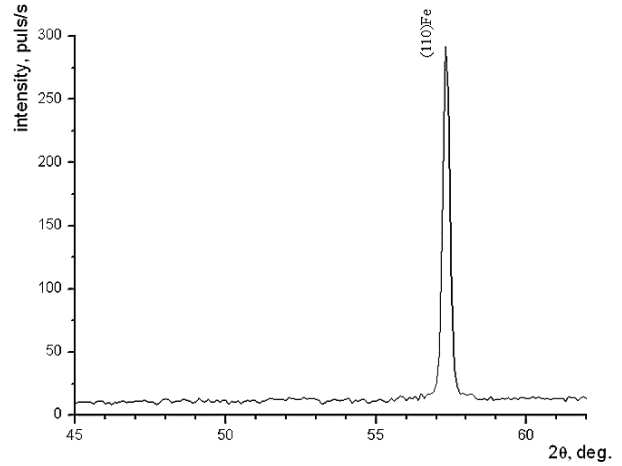
As can be seen from Figure 5, during nitriding in the CSD (mode 1), the surface microhardness along the height of the pipe of the modified layer of C45 steel, respectively, from the side of the inner ends, remains constant, while during nitriding with a direct current, it decreases and reaches a minimum for samples placed in the center of the pipe (mode 2). For mode 3 (dead hole), the surface microhardness constantly decreases and reaches the initial value for the upper sample.

The results of microhardness measurement and their comparison with the data of X-ray phase analysis confirm the formation during nitriding in a cyclically switched discharge (mode 1) for all steels of  $\epsilon$ ,  $\gamma$ , and  $\alpha$  phases studied in the work along the entire height of the samples placed in the experimental model (Figure 6 a).

Simultaneously, for samples placed at a distance of more than 150 mm for all steels nitrided in mode 3 with a constant supply current, only the  $\alpha$  phase is formed (Fig 6 b).

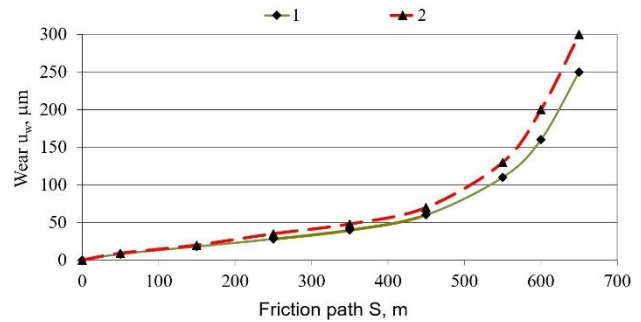


a

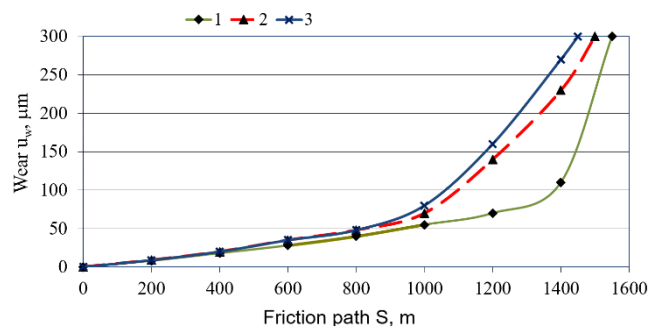


b

Figure 6– Section of the diffractogram of the steel samples at a distance of 200 mm from the end: a – C45 (mode 1); b–37Cr4 (mode 3)



a



b

Figure 7 – The dependence of wear on the friction path at different nitriding modes: a – steel C45 (modes 1, 2); b – steel 41CrAlMo7 (modes 1–3)

With a friction path of up to 30 m, wear for all samples made of 41CrAlMo7 steel after nitriding with a cyclically switched glow discharge (mode 1) is practically absent, and further up to 1100-1200 m wear proceeds at a constant rate along the entire length of the pipe. After 1300 m of the friction path, catastrophic wear of the sample surfaces occurs (Figure 7 b, mode 1).

When nitriding in a glow discharge with a DC power supply, the results of hardening of the surfaces of samples made of 41CrAlMo7 steel are significantly worse (Figure 7 c), especially for mode 3 (“close” hole). For these samples, approaching the center of the pipe, catastrophic wear occurs after 1000 m of the friction path. Wear occurs at a significant rate, and the rate of wear increases as the sample moves away from the open end of the pipe.

As a result of almost identical physicochemical and strength characteristics of nitrided layers [17] on steels C45 and 37Cr4, their tribological characteristics are almost the same [18].

## 5 Discussion

Comparison of absolute surface microhardness values is not entirely correct, especially if we consider the staged nature of the experiments. This conclusion is because the surface microhardness data largely depend on the properties of the surface point where these measurements were made, for example, the presence of carbides, grains, and their boundaries. For this reason, the principle of comparing the relative values of surface microhardness was adopted as the starting point for the analysis (Figures 2 a–b).

Tests for wear resistance were initially carried out under conditions of extreme friction [19], but it turned out that in the mode of dry friction, it is possible to achieve results at the same pressure values for almost all steels. The latter excludes the issue of comparability when analyzing research results. In addition, the study of wear resistance in the dry mode of friction ensures significantly higher productivity of experiments. In contrast to experiments with extreme friction, dry friction can be applied to different steels at the same pressure value, which eliminates the problem of comparability of results and contributes to the objectivity of conclusions regarding the effectiveness of various modification processes. According to the results of previous experiments, such a compromise value of pressure can be 16 MPa [20].

Even more illustrative from the point of view of choosing the nitriding mode is a comparison of the microhardness values along the depth of the modified layer to a similar indicator at the end of the sample (it is evident that only in terms of principle decisions, and not specific values of the parameters of the technological mode, if only because the indicators to a certain extent it is difficult to compare the ratios for the modes with constant power supply and cyclic-switched power, because the duration of the process was the same, for cyclic-switched power, the discharge flowed at a ratio of 2 for only half of this time). This indicator more objectively reflects reality since the accuracy and location of the measurement have a lesser effect on the reliability of the analysis (Figures 3 a–c).

An obvious conclusion from Figures 3–5 is that changing the technology makes it possible to ensure the required nitriding quality of holes with a relatively small diameter.

Simultaneously, using a cyclically switched discharge should be considered the most promising.

However, surface modification in a cyclically switched discharge generally opens new possibilities related to CSD variants, which are characterized by frequency, period, and pulse shape. Implementing the process of adjusting the switching frequency, pitch - the ratio of the cycle period to the duration of the signal, and the shape of the signal itself opens wide opportunities to significantly influence the surface treatment results.

The influence of the shape of the discharge power signal on the kinetics of the nitriding process and its results opens vast opportunities for studying the process itself. The presence of surges at the beginning and at the end of the cycles can, in principle, significantly affect both the nature of the nitriding process itself and the structure and phase composition of the modified surface layer since short-term and sufficiently powerful voltage surges should lead to intensive surface sputtering. The destruction of the monolayer of nitrides, which has just formed on the surface, will contribute to the increase of the depth of the nitrided layer due to the diffusion of nitrogen particles, as well as, to a certain extent, leveling off the blocking effect of the surface nitride layers.

The use of cyclically switched discharge nitriding in glow discharge technology allows for obtaining adjustable and even predictable processes of surface modification of metals and alloys and the formation of surface layers with specified properties, especially for parts of complex configurations.

This work studied only the nitriding of long holes in a cyclically switched discharge. Other structural elements in detail (e.g., groove keys, slots of various configurations) were not studied in the work but are in the plans for further research.

## 6 Conclusions

It was established that using holes with a relatively small diameter of a glow discharge for nitriding with a different type of power supply creates conditions for obtaining modified layers with different physical, mechanical, and tribological characteristics.

The results of microhardness measurement and their comparison with the data of X-ray phase analysis confirm the formation of  $\epsilon$ ,  $\gamma$ , and  $\alpha$  phases during nitriding in the CSD along the entire height of the samples placed in the experimental model. The microhardness of the surface nitrided layer with the use of CSD was the same for all samples and was 592 MPa for steel C45, 797 MPa for steel 37Cr4, and 1098 MPa for steel 41CrAlMo7.

For other samples with a constant nitriding current, significant fluctuations in the microhardness values of the samples placed at different heights from the open end of the experimental model were observed. For through holes (mode 2), the deviation of microhardness was 592/495, 794/688, and 1100/960 MPa, respectively, for steels C45, 37Cr4, and 41CrAlMo7 (the numerator for the sample at the distance of 10 mm, the denominator at the distance of 200 mm from the end of the model).

The tests carried out in the dry friction mode showed an increase in the wear resistance of samples made of steel C45, 37Cr4, and 41CrAlMo7 during nitriding in a cyclically switched discharge.

It was established that using long holes of a glow discharge with different types of power for nitriding creates conditions for obtaining modified layers with variable characteristics.

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