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Improvement of physical and mechanical characteristics of gearbox shafts using the oxycarbonitriding method

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Abstract

The article investigates the issue of promising methods for improving steels by modifying surface layers with the application of protective coatings. Priority areas of scientific research in the field of mechanical engineering have been identified for the development of new methods and technologies for increasing the wear resistance of steel surfaces by applying modified diffusion coatings. It has been shown that the key problem in optimizing the processes of saturation of metals and alloys with one element is cementation, nitriding, alitization, chromium plating, etc. Saturation with two or more elements is used very limitedly. The features of chemical-thermal treatment of steel are considered, with the help of which we have the opportunity to obtain a material characterized by increased characteristics and properties (surface hardness, corrosion resistance, wear resistance). Based on the identified features of coatings in terms of composition, structure, and properties, chemical-thermal treatment methods can be promising when operating them under wear conditions. However, the proposed innovative metallization method – oxycarbonitriding – can compete with these technologically complex processes. It was established that combined chemical-thermal treatment, including nitriding and oxidation, allows to significantly increase the corrosion resistance of the material, the longer the oxycarbonitriding process time, the thicker the oxycarbonitrided layer, but at the same time the absolute deformation value increases, which must be considered when processing precision surfaces. Promising directions for further scientific research in this field are identified.

Key words: modification, surface layers, chemical-thermal treatment, saturation of metals and alloys, diffusion coatings, corrosion resistance, wear resistance.

Introduction

There is no doubt about the prospects for the development of such defining branches of production as mechanical engineering, chemical, radio engineering, space, energy and nuclear engineering. Therefore, today the task of developing new methods and technologies for increasing the wear resistance of steel surfaces by applying modified diffusion coatings remains relevant.

The destruction of machine parts, tools and other products in the vast majority of cases begins with the surface, and it is to the surface layers that the above requirements apply. The reduction in wear resistance leads to billions of dollars in losses annually, and solving this problem is an important task. The main loss is not the loss of metal as such, but the enormous cost of products. That is why the annual losses of industrially developed countries are so large. Real losses cannot be determined by assessing only direct losses, which include the cost of the destroyed structure, the price of changing equipment, and the costs of measures to improve wear resistance. Indirect losses are even greater. This is simple equipment when replacing corroded parts and assemblies, disruption of technological processes.

Economic losses from metal corrosion are enormous. According to estimates of experts from different countries, these losses in industrially developed countries amount to from 2 to 4% of the gross national product. At the same time, metal losses, which include the mass of metal structures, products, and equipment that have failed, amount to from 10 to 20% of annual steel production [1]. In this regard, bulk alloying of alloys is usually uneconomical, and in many cases even impossible due to the almost complete loss of their plasticity and toughness

[1]. Therefore, in recent years, increasing attention of researchers and manufacturers has been paid to various methods of surface hardening. One of the main methods of surface hardening is chemical-thermal treatment.

Problem statement

Currently, the processes of saturation of metals and alloys with one element are quite widely used: cementation, nitriding, alitization, chromium plating, etc. Saturation with two or more elements is used very limitedly. It is quite obvious that multicomponent saturation allows to significantly change the properties of surface layers [1].

Analysis of recent research and publications

One of the most promising methods for improving steels is the modification of surface layers by applying protective coatings. The principle of combining high hardness, wear resistance, corrosion-oxidation resistance and chemical inertness to active environmental reagents with strength and wear resistance is most successfully implemented on 12X18N10T steel and is reflected in many works of modern scientists.

In work [2], a classification of coating methods is given according to the nature of the interaction between the base material being processed and the saturating element, as well as the possibilities of obtaining coatings. Each of the technological methods for coating has its own advantages and disadvantages.

There is a significant number of scientific works on the classification of chemical-thermal treatment. Thus, in work [3], an overview of existing methods of combined strengthening treatment of steel machine parts is given, the main methods of combined treatment of structural and tool steels are considered.

Chemical-thermal treatment is based on solid-phase, liquid-phase and gas-phase saturation of the surfaces of parts. Diffusing elements can saturate the surface of the part directly, without intermediate reactions, with a preliminary chemical reaction at the interface of the processed material and the coating or in the volume of the starting reagents. chemical-thermal treatment can be implemented by saturating nickel and nickel alloys with both metals and nonmetals. As a result, diffusion coatings are formed. The rate of formation, kinetics of coating growth, its structure and properties are largely determined by the process temperature, saturation time, diffusion parameters of the saturating components in the material and, finally, significantly depend on the chemical composition, structure and properties of the latter. During chemical-thermal treatment, the change in the chemical and phase composition, structure and properties of the surface layers is carried out at elevated temperatures.

The following processes occur in this case [3]:

– formation of active atoms of saturating elements and their transfer to the processed surface;

– adsorption (chemisorption) of active atoms by the processed surface;

– diffusion movement of adsorbed atoms into the product and the resulting diffusion redistribution of elements of the base alloy.

Diffusion saturation of the surface zones of nickel and nickel alloys with metals and nonmetals leads to the formation of coatings consisting either of solid solutions of saturating elements in the original alloys, or of chemical compounds. Due to this, it becomes possible to obtain protective coatings on the surface of products with different composition and operational properties. The optimal choice of a saturating element for a particular base material provides a high level of adhesion, practically unattainable with other types of processing (plasma, gas-thermal). Diffusion coatings during HTO are applied, as a rule, under isothermal conditions in artificially created saturating environments.

As noted in many works [4-7], chemical-thermal treatment of steel is of certain scientific and practical interest. As a result of this treatment, we have the opportunity to obtain a material characterized by increased characteristics and properties (surface hardness, corrosion resistance, wear resistance). It has been established that during titanalization of samples from annealed steel 12X18H10T, multilayer coatings of Fe_2Ti, Ti_4Fe_2O, TiN, TiC are formed [5]. In addition, it has been shown that the TiN layer acts as a barrier, significantly reduces the content of iron, nickel and chromium in the coating, and also significantly inhibits the diffusion of titanium and aluminum into the base. The microhardness of TiN layers in complex compounds is 20.5...23.0, and the diffusion zone of the compounds is 5.5...12.5 GPa. The researchers concluded that, in terms of composition, structure, and properties, titanoaliding of 2X18H10T steel can be recommended for use as heat- and corrosion-resistant, antifriction [6].

Preliminary nitriding of 12X18H10T steel also has positive results. It was established [7] that as a result of titanoaliding of pre-nitrided steel, a multilayer coating with the participation of Fe_2Ti, Ti_4Fe_2O, TiN, CrN compounds is formed on the treated surface. The results of the work confirm the prospects of using titanoaliding and nitriding when operating under high temperatures, aggressive environments, and harsh friction conditions [7].

Complex saturation of 12X18H10T steel with chromium and titanium was implemented under reduced pressure [7]. The possibility of forming a chromium-alloyed coating with a barrier layer based on titanium nitride TiN on 12X18H10T steel is shown, the presence of which causes a decrease in the concentration of iron and titanium on the outer side of the coating, an increase in the concentration of aluminum, a decrease in the thickness of the zone of compounds and solid aluminum solution in the base.

Boration of 12Kh18N10T steel is considered in [8]. The introduction of copper or its alloys into the composition of the saturating powder media makes it possible to intensify the boridation of 12X18H10T steel without deteriorating the operational properties of boride coatings while simultaneously reducing their fragility.

Single-component chromium plating of 12X18H10T steel [8] is implemented for products operating under friction conditions. In order to increase the saturation rate and wear resistance, copper powder was added to the composition of the carburizer.

Presentation of the main material

Thus, known methods of applying multicomponent coatings in terms of composition, structure, and properties can be promising when used in wear conditions. However, the proposed innovative metallization method - oxycarbonitriding can be a worthy competitor to these technologically complex processes.

More details about the surface hardening method "oxycarbonitriding" and the processes that occur in the metal can be found in well-known scientific studies. First of all, this method can be compared with long-known surface treatment methods, such as: nitriding and carbonitriding. These methods and their combinations are increasingly gaining popularity in the areas of restoration and improvement of wear resistance, by changing surface layers with minimal changes in size, while providing the material with resistance to corrosion and wear with insignificant roughness values.

In work [9], the change in the size of parts during various types of chemical-thermal treatment is investigated, how the environment and temperature affect the process of surface saturation with carbon. Fig. 1 shows the difference in hardness.

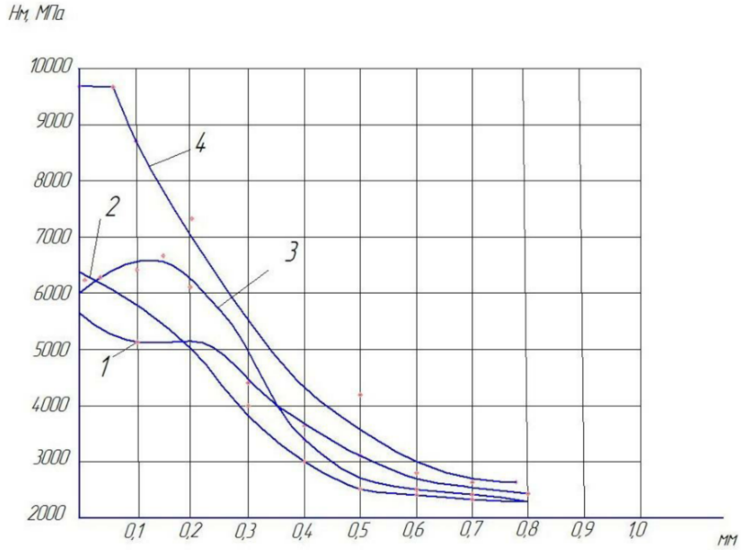


Fig. 1. Microhardness of restored parts by different methods of chemical-thermal treatment. 1) nitriding; 2) carbonitriding; 3) oxynitriding; 4) rotary chrome plating.

Processing of parts by oxycarbonitriding (Fig. 1, curve 3) was performed at temperature regimes of 590 – 610^{0}C and provides sufficient microhardness of surface layers at a depth of 0.1-0.2mm, which is 6370-6770MPa, and the total depth of the hardened layer varies within 0.25-0.35mm. The recorded changes in the dimensions of the parts of the plunger pairs of fuel pumps are 20-45µm, which is an advantage for high-precision parts that have worn out and lost their working dimensions.

More details about oxycarbonitriding are given in [10]. The author, using foreign literature and various methods of carrying out the selected process, highlights their positive aspects, which are combined into one process, and positively affects the corrosion resistance index and the friction coefficient, which under different loads is a constant value and is within the limits of $0.03<f<0.004$ after 40 hours of operation.

According to electrochemical studies conducted by Ebersbach and co-authors [11], nitriding in a gas environment leads to a 10-fold reduction in the corrosion rate in a sodium chloride solution (concentration 0.9M). Additional oxidation of the nitrided layer provides an even more significant effect, reducing the corrosion rate by two orders of magnitude, additionally reducing the rate of through corrosion by hundreds of times.

When nitriding iron in ammonia, the formation of a diffusion layer occurs in accordance with the phase diagram of the iron-nitrogen system (Fe-N). This diagram shows the dependence of the phase states of iron on the nitrogen content and temperature, which allows us to determine the conditions for the formation of iron nitrides (such as ε-Fe2-3N and γ'-Fe4N). Typically, the process occurs at elevated temperatures in an ammonia environment, where ammonia dissociates, releasing atomic nitrogen, which penetrates the metal. This process improves the hardness, wear resistance and corrosion resistance of the material due to the formation of a strengthened diffusion layer. The formation of oxynitride surface layers is carried out by oxidation of nitride layers. In the temperature range 450–700^{0}C, the affinity of metals for oxygen significantly exceeds their affinity for nitrogen.

This causes exchange reactions during the oxidation of nitrided layers, when oxygen partially replaces nitrogen in the surface nitride layer.

Thermodynamic calculations confirm this: iron nitrides interact with oxygen more actively than pure iron. For example, the isobaric-isothermal potential of the oxide formation reaction at 500°C is 209–293 kJ/mol for Fe, and 878–1463 kJ/mol for Fe_4N.

It is important that the high affinity of nitrides for oxygen ensures the formation of optimal oxide structures. In nitrides, the solubility of oxygen is three orders of magnitude higher than in pure iron (at 700°C – 3% versus 0.009% for α-Fe), which contributes to the formation of oxides of the first type, which are solid solutions of oxygen. Oxynitride zones have greater plasticity than Fe_2O_3 oxide films, and at the same time demonstrate similar anti-adhesive properties. As Mittemeyer and Collin [12] established, further oxidation of nitrided surfaces contributes to filling the pores of the α-phase with oxygen. Combined chemical-thermal treatment, which includes nitriding and oxidation, allows to significantly increase the corrosion resistance of the material.

Previously, to carry out the process of improving the working surfaces of gearbox shafts, samples from steel 35ХГТ DSTU 7806:2015 identical to the part were used. To understand how the microstructure of the metal changed after the heat treatment process, it was decided to first consider the structure before the chemical-thermal treatment. To begin with, microsections were made from the prepared samples, an example of which is shown in Fig. 2.

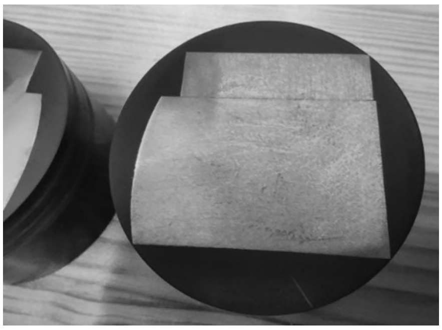
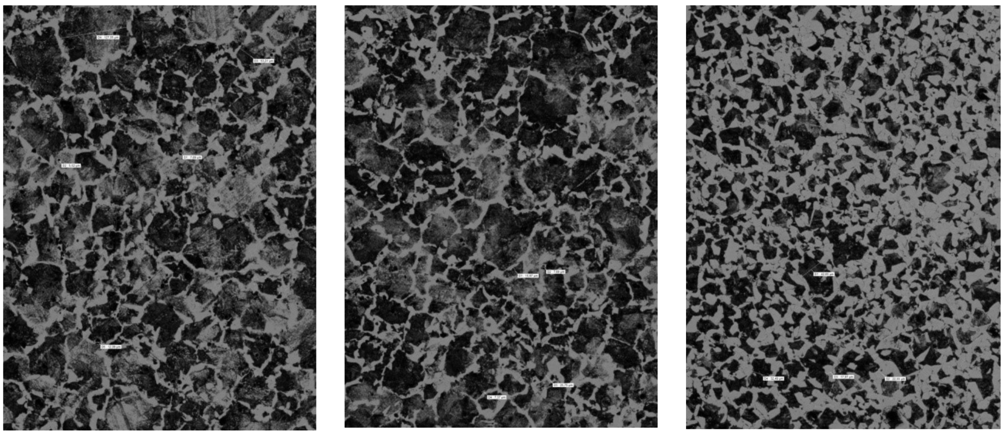


Fig. 2. Photo of a manufactured and prepared microsection of 35ХГТ steel

The placement of samples in the microsection is chosen in such a way as to be able to study the microstructure of the longitudinal or cross-section. Where the larger piece is the cross-section, and the smaller and narrow piece is the longitudinal. The section of samples for microsections is necessarily performed by cold methods without exceeding the temperature of critical points to preserve the structure, for example, water jet or saw cutting with cooling. Fig. 3 shows the microstructure of steel before the oxycarbonitriding process at a magnification of ×100 in the longitudinal section. The microstructure of steels before heat treatment ideally consists of ferrite and pearlite. But in practice, the ideal structure cannot be achieved due to foundry production technologies and equipment. In 35ХГТ steels, ferrite has the form of a mesh, the thickness of which in different batches may not differ significantly. Based on practice, the finer the mesh, the worse the processing and roughness. Also, inclusions are observed in the steel, previously it is residual unformed ferrite, in the form of clusters that have a hardness of 332 HV, and in general the hardness of this steel does not exceed 202 HV. The samples show a ferrite-pearlitic structure, a uniform distribution of the ferrite mesh with occasional enlargements of pearlite cells. The measured pearlite to ferrite ratio is 65:35 and there is a reduction in ferrite in the ratio of 75:25 according to DSTU 8233-56. The size of pearlite grains is on average 60-80 μm, and with uniform enlarged grains 150-200 μm.

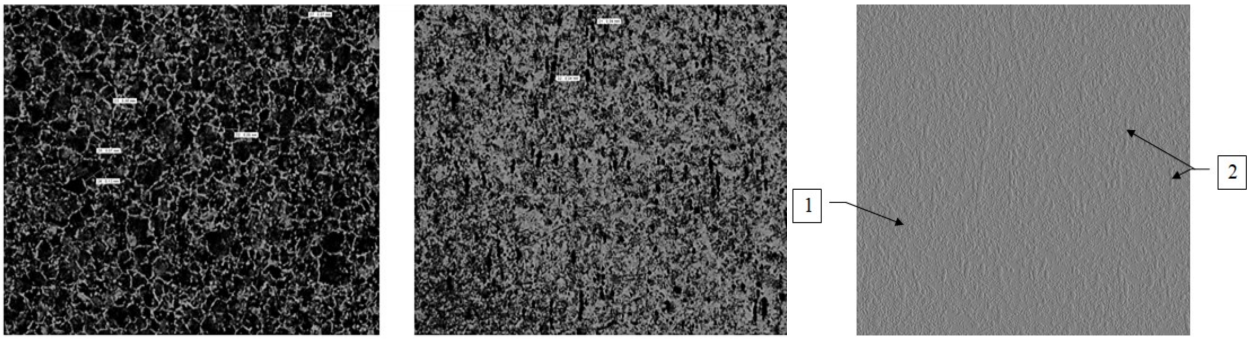


a) b) c)

Fig. 3. View of the microstructure of the samples in cross section

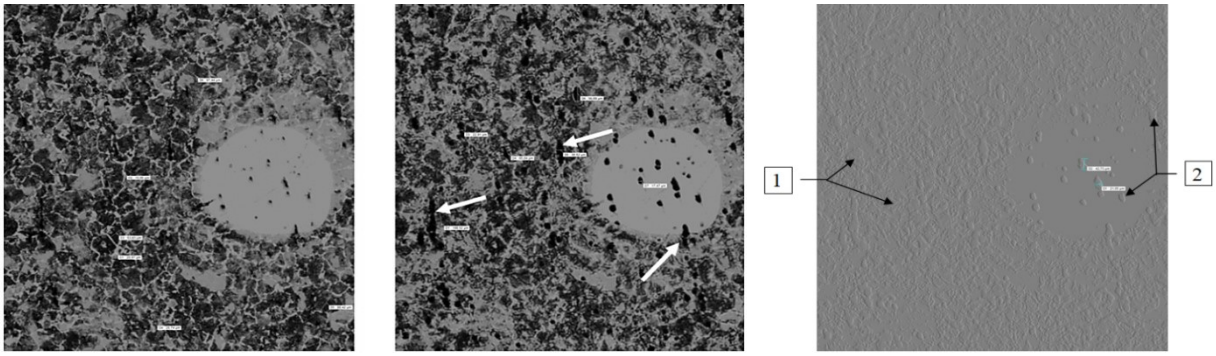
When examining the microstructure, a certain regularity of the appearance of the microstructure was noticed, which consists in the fact that an increased amount of ferrite (light mesh) is located along the edge of the rolling roller along the entire length. So, in Fig. 3a and 3b, the structure of the central region is shown, and in Fig.

3c - closer to the edges. When studying the microstructure of longitudinal section samples, sulfide inclusions were found, which accumulate in the form of stripes along the length of the rolled product. In a vertical section, the inclusions have a round shape. These inclusions are poorly visible, so it is necessary to focus the microscope lens below the pearlite grains and ferrite mesh, because they are etched deeper. Therefore, for objective vision, it is necessary to adjust the focus specifically for these inclusions, as shown in Fig.s 3a; 3b. It was also found that sulfide inclusions are etched faster than the base metal. To check for inclusions in a particular area of the microsection, there is a special mode of the microscope camera that shows the surface relief (Fig. 4 c). The etched pearlite zones are shown by the arrow number 1, and sulfide inclusions are shown by the arrow number 2. They are also visible in unetched areas (Fig. 5 c).



a) b) c)

Fig. 4. Example of microstructure in different modes with magnification ×25: a) focus on ferrite network; b) focus on sulfide inclusions; c) view of the structure in relief.



a) b) c)

Fig. 5. Example of microstructure in different modes with magnification ×50: a) focus on ferrite network; b) focus on sulfide inclusions; c) view of the structure in relief.

Analyzing longitudinal cross-section samples from different batches, an accumulation of residual ferrite is observed, which certainly has a negative impact on mechanical and heat treatment in the future. This defect is shown by arrows in Fig. 6, which has the form of large accumulations ranging in size from 50 to 600 μm.

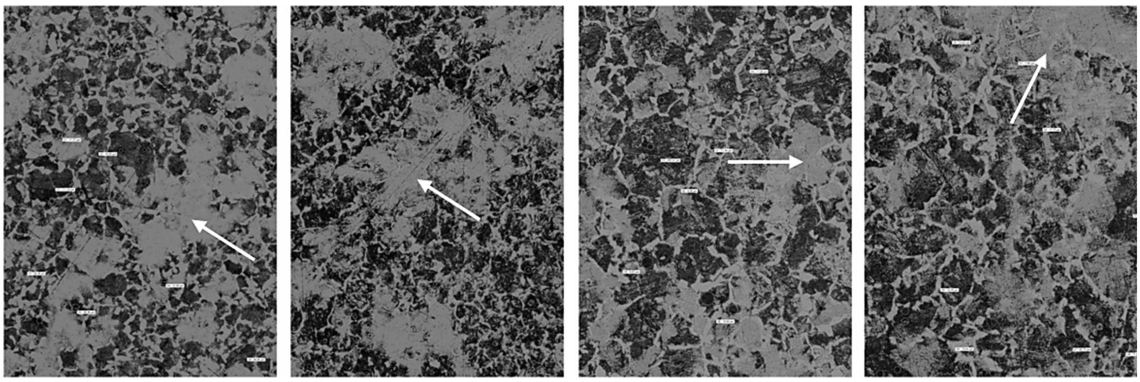
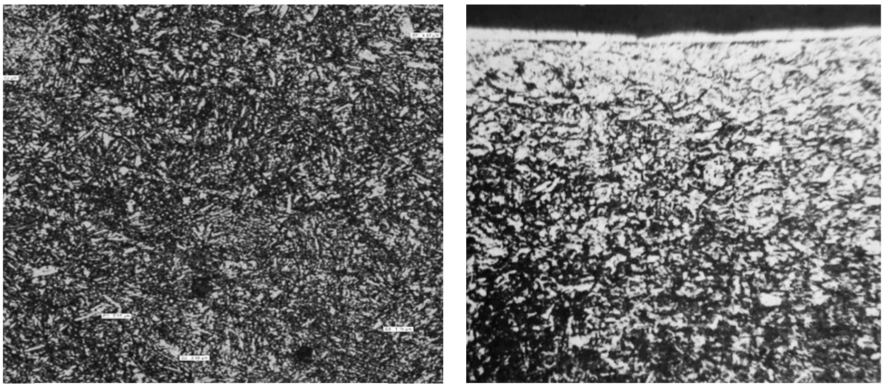


Fig. 6. Appearance of the microstructure of 35ХГТ steel with defects, ×100

After applying the oxycarbonitriding method, grinding of the metal structure, burnout and a decrease in the number of sulfide inclusions are observed. As expected, a white oxycarbonitride layer is observed on the surface of the part, which is indicated by an arrow (Fig. 7 b).



a) b)

Fig. 7. Image of the microstructure after heat treatment. a) microstructure in depth; b) surface microstructure with oxycarbonitride layer

The hardness of the samples was measured by the Rockwell method. The hardness of the upper layer was set within 50-58 HRC, and the hardness of the inner layers was on average 30 HRC. The thickness of the formed oxycarbonitride layer was 0.12-0.20 mm.

When studying the deformation effect on the cylindrical part, a PMT-3 microhardness tester was used. Absolute deformation (δD) was determined according to formula 1. With a duration of chemical-thermal treatment of about 8 hours, the absolute deformation value is 0.024 mm. at Ø49 mm.

$$\delta D = \frac{D_2 - D_1}{2} \tag{1}$$

where: D_1 is the diametrical size before oxycarbonitriding;

D_2 is the diametrical size after oxycarbonitriding.

The absolute deformation value was calculated at least in three places of one diametrical size. It was experimentally established that the absolute deformation value increases by 0.003 mm for each subsequent hour of the surface hardening process. Therefore, the longer the oxycarbonitriding process time, the thicker the oxycarbonitriding layer, but the absolute deformation value increases, which must be taken into account when processing precision surfaces. After the chemical-thermal treatment processes, local chemical analysis of the samples was carried out in a special laboratory. Measurements of chemical inclusion values were carried out at seven points on each sample. An example of one of the samples is shown in Fig. 8.

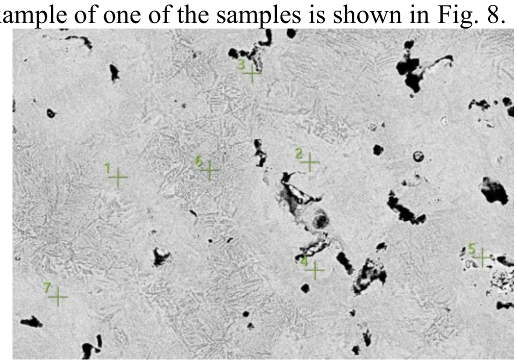


Fig. 8. Steel microstructure with measurement points

After the measurement, the obtained values were averaged and grouped into Table 1.

Table 1

Values of chemical inclusions of steel 35ХГТ DSTU 7805:2015.

	Value in %						
Element	Point 1	Point 2	Point 3	Point 4	Point 5	Point 6	Point 7
C	4.8	4.2	4.8	4.9	5.8	3.5	4.9
O	0.0	0.0	0.0	0.0	0.6	0.0	0.0
Si	0.4	0.5	0.5	0.3	0.3	0.5	0.5
Cr	0.9	0.8	0.9	0.7	0.7	1.0	0.9
Mn	0.6	0.6	0.6	0.6	0.4	0.5	0.7
Fe	92.7	91.8	92.6	85.0	74.4	93.7	92.5
Ni	0.3	0.3	0.3	0.3	0.3	0.4	0.3
Cu	0.3	1.8	0.3	8.0	17.3	0.3	0.3
Mo	0.1	0.0	0.0	0.1	0.1	0.0	0.0

As can be seen from Table 1, the chemical elements are not evenly distributed in the samples. Thus, at point 5, the maximum value of carbon is observed - 5.8% and copper - 17.3%, the smallest amount of iron is 74.4%, manganese - 0.4%, chromium - 0.7%, silicon - 0.3%. The smallest amount of carbon is observed at point 6 - 3.5%, but the largest amount of iron is present - 93.7% and nickel - 0.4%.

Conclusions

In this work, the material of the part modified by the oxycarbonitriding method was studied. A study was conducted to change the structure and metal, what defects are observed. The parameters of the oxycarbonitriding process were studied, and experimentally established which ones are necessary for the selected part. A wear-resistant oxycarbonitriding layer with a thickness of 0.12-0.2 mm was obtained, which has a hardness of about 60 HRC on the surface. A chemical analysis of the samples was also carried out and non-uniformity of the location was revealed. After conducting the chemical-thermal treatment, dimensions were obtained that will correspond to the tolerances on the drawing.

Prospects for further research.

One of the priority areas of using such technologies is to improve the operating characteristics of cutting tools, friction pairs of machine parts, gearbox shafts operating under extreme mechanical and temperature loads.

CCHT technology is environmentally friendly and can in most cases replace galvanic coatings (oxidation, zinc plating, chrome plating, cadmium plating, etc.), which can provide a cost reduction of 35 - 40%.

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Мєшков Ю. Є., Дмитрієв М. С. Покращення фізико-механічних характеристик валів коробок передач за допомогою методу оксикарбонітрування

У статті досліджується питання перспективних методів удосконалення сталей модифікуванням поверхневих шарів з нанесенням захисних покриттів. Визначені пріоритетні напрями наукових досліджень в галузі машинобудування з розробки нових методів та технологій з підвищення зносостійкості поверхонь сталей за рахунок нанесення модифікованих дифузійних покриттів. Показано, що ключовою проблемою в оптимізації процесів насичення металів і сплавів одним елементом являється цементація, азотування, алітування, хромування і т.д. Насичення двома чи декількома елементами застосовують дуже обмежено. Розглянуто особливості хіміко-термічної обробки сталі, за допомогою якої маємо можливість отримати матеріал, який характеризується підвищеними характеристиками та властивостями (поверхневою твердістю, стійкістю до корозії, зносостійкістю). Виходячи з визначених особливостей покриттів за складом, будовою, властивостями методи хіміко-термічної обробки можуть бути перспективними при їх експлуатації в умовах зношування. Однак, гідну конкуренцію цим технологічно складним процесам може скласти запропонований інноваційний метод металізації – оксикарбонітрування. Встановлено що комбінована хіміко-термічна обробка, що включає азотування і окиснення, дозволяє значно підвищити корозійну стійкість матеріалу, чим більший час процесу оксикарбонітрування тим товстіший оксикарбонітридний шар, але при цьому збільшується значення абсолютної деформації, що потрібно враховувати при обробці точних поверхонь. Визначені перспективні напрямки подальших наукових досліджень в зазначеній галузі.

Ключові слова: модифікування, поверхневі шари, хіміко-термічна обробка, насичення металів і сплавів, дифузійні покриття, стійкість до корозії, зносостійкість