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## CHROME PLATING OF PRESS TOOLING USED IN THE PRODUCTION OF NEW ELASTOMERIC MATERIALS

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**Abstract.** This research has produced multicomponent titanium-doped chromium coatings to strengthen the working surfaces of press tooling dies operated under aggressive conditions during the vulcanization of products from new elastomeric materials. The pressing was carried out on a hydraulic vulcanization press 100–400 2E. with plate sizes 400×400 of new elastomeric materials based on 10 wt. % carbonized fiber from PAN (RC Helper) or (USA), which significantly increases the heat capacity of rubber based on a copolymer of vinyl develfluoride and hexopropylene by 15–30 % in the operating temperature range from 323 to 348 K. In the work, samples from mass-produced steels such as steel 20, steel 45, U8, 40H, and 40H16M were used for coating. Mixtures of powders with a dispersion of 60–250 microns of the following materials were used as reaction agents. In determining the required dispersion of reagents, we were guided by studies that found that the maximum completeness of the transformation is observed when using a reaction mixture with a fraction of 100–120 microns. The protective coatings on samples under the conditions of the SHS were obtained at the developed pilot plant DSTU12, consisting of the following main functional systems: reaction equipment; system for monitoring and controlling technological parameters; and gas utilization system. The organization of the SIS consists in creating a powder mixture and an environment that provides exothermic interaction at the local initiation of the process (ignition). Then, the reaction wave propagates spontaneously (combustion) and the synthesized product is cooled. According to the type of chemical reaction, three SWS processes can be distinguished: direct synthesis from elements; direct synthesis from compounds and exchange reactions; and metallothermal SHS (reactions with a reducing step). The surface microhardness of multicomponent chrome layers alloyed with titanium and boron is 20000–22000 MPa, which is due to the presence of the phase FeB. The microhardness of the alloyed Fe<sub>2</sub>B phase is 15000–16000 MPa. When chromium coatings are alloyed with titanium on the considered structural materials, the following phases are formed: (Fe, Cr)<sub>23</sub>C<sub>6</sub>, (Fe, Cr)<sub>7</sub>C<sub>3</sub>, Cr<sub>3</sub>C<sub>2</sub>, as well as phases: Fe<sub>2</sub>Ti, Cr<sub>2</sub>Ti. The surface microhardness of titanium-alloyed chromium coatings is: on steel 20–H<sub>100</sub>=16000 MPa, (phases: (Cr, Fe)<sub>23</sub>C<sub>6</sub>, (Cr, Fe)<sub>7</sub>C<sub>3</sub>, Fe<sub>2</sub>Ti, Cr<sub>2</sub>Ti, α-solid solution of Ti and Cr in α-iron), on steel 45–H<sub>100</sub>=18000 MPa (phases: (Cr, Fe)<sub>23</sub>C<sub>6</sub>, alloyed with titanium, α-solid solution of chromium in iron with Cr<sub>2</sub>Ti inclusions), on U8–H<sub>100</sub>=19500 MPa (phases: directly on the outer side of the coating there is a zone of carbides (Fe, Cr)<sub>23</sub>C<sub>6</sub>, (Fe, Cr)<sub>7</sub>C<sub>3</sub>, Cr<sub>3</sub>C<sub>2</sub>, (Ti, Cr)C).

**Key words:** self-propagating high-temperature synthesis; chromium; titanium; silicon; phase; microhardness; structure; elastomer; pressing.

## **Introduction**

Today, composite materials (rubbers, rubber) based on natural and artificial rubber have become an integral part of the metallurgical, textile, and chemical industries. The use of rubbers makes it possible to produce structural and tribotechnical products characterized by enhanced damping properties, high elasticity, and corrosion resistance. In addition, the use of rubbers instead of metals reduces the material consumption of structures and machines, shortens the production time of parts (even of complex configurations), and increases corrosion resistance [1]. The pressing was carried out on a hydraulic vulcanization press 100–400 2E with plate sizes of 400×400 of new elastomeric materials based on a copolymer of vinylidene fluoride and hexopropylene, ethylene-propylene rubber, containing carbonized fiber from PAN (polyacrylonitrile), stone (granite) flour, and aluminosilicate microspheres as Np [2, 3]. It is designed to apply a force on the sample with simultaneous temperature exposure. It is used for vulcanization of rubber compounds in molds, the maximum force is 1000 kN. The organization of the SHS is to create a powder mixture and an environment that provide exothermic interaction at the local initiation of the process (ignition). Then, the reaction wave propagates spontaneously (combustion) and the synthesized product cools down. According to the type of chemical reaction, three SHS processes can be distinguished: direct synthesis from elements; direct synthesis from compounds and exchange reactions; metallothermic SHS (reactions with a reducing step) [4]. Various chemical compounds (carbides, borides, silicides, nitrides, intermetallics, chalcogenides, etc.) can be products of SHS processes. Depending on the aggregate state of the reagents and reaction products, SHS processes are divided into gas-free, low-gas, filtration, and metallothermic [5]. The selection of SHS systems is based on the principles of process combustion. The main feature is the formation of a useful product as a result of an exothermic reaction, while the size of the thermal effect is of interest only to the extent that the combustion process itself can provide.

## **Problem statement**

Accelerated development of technology has made it extremely important to increase the reliability and durability of machine parts, devices, installations, and structural materials used in aggressive environments on equipment for vulcanizing products made of elastomeric materials, improve their quality and efficiency, and thus, to save metals, combat corrosion and wear of machine parts and critical components. The solution to these problems is associated with strengthening the surface layers of products. The role of surface hardening processes in the durability of machines and mechanisms has increased especially today, as the development of machine-building industries is associated with increased loads, temperatures, and aggressive environments in which critical parts operate.

Production of multicomponent chrome coatings alloyed with titanium to strengthen the working surfaces of parts operating in aggressive environments on equipment for vulcanizing products made of new elastomeric materials.

The surface microhardness of titanium-alloyed chromium coatings is: on steel 20– $H_{100}=16000$  MPa, (phases:  $(Cr, Fe)_{23}C_6$ ,  $(Cr, Fe)_7C_3$ , titanium-alloyed,  $Fe_2Ti$ ,  $Cr_2Ti$ ,  $\alpha$ -solid solution of Ti and Cr in  $\alpha$ -iron), on steel 45– $H_{100}=18000$  MPa (phases:  $(Cr, Fe)_{23}C_6$ , titanium-alloyed  $\alpha$ -solid solution of chromium in iron with  $Cr_2Ti$ ), on U8 steel –  $H_{100}=19500$  MPa (phases: directly on the outer side of the coating there is a zone of carbides  $(Fe, Cr)_{23}C_6$ ,  $(Fe, Cr)_7C_3$ ,  $Cr_3C_2$ ,  $(Ti, Cr)C$ ).

## **Objectives and problems of research**

The purpose of our study is to obtain multicomponent chrome coatings alloyed with titanium and aluminum to strengthen the working surfaces of parts operated under aggressive conditions on equipment for vulcanizing products from new elastomeric materials using modern methods of surface hardening of materials, including the SHS technology, with the main goal of developing new approaches to improve the mechanical properties and extend the service life of materials. As part of this study, we plan to conduct a broad review of existing methods, thoroughly study the features of the SHS processes, conduct a series of

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experimental studies to determine the optimal process parameters, and analyze the data obtained to determine the effectiveness of the SHS method for improving the mechanical properties of materials and extending the service life of machine parts and equipment for vulcanizing products from new elastomeric materials.

### **Main material presentation**

Modern operating conditions for machine parts, assemblies, equipment, tools and mechanisms place increased demands on their physical and mechanical characteristics and service life. The role of surface hardening processes in the durability of machines and mechanisms has increased especially nowadays, since the development of machine-building industries is associated with increased loads and temperatures at which critical parts operate in aggressive conditions on products made of new elastomeric materials equipped for vulcanization.

The development of many areas of modern industry is impossible without the development and use of new resource-efficient technologies. In this case, an important role is played by technologies that can impart special properties to surface layers. To the greatest extent, these requirements are met by the SHS technology, which allows to production of materials and coatings with controlled composition, structure, and performance characteristics.

The essence of the SHS method is to carry out exothermic reactions in the mode of combustion wave propagation with the formation of combustion products in the form of material compounds that have practical value and valuable characteristics [6, 7].

SHS processes and products are widely used in various industries: mechanical engineering (abrasives, tool and structural materials, ceramics), metallurgy (ferroalloys and ligatures), chemical industry (refractory compounds, fumigants, pigments, catalysts), inorganic materials technology (for producing powders, compact materials and products, coating and welding of parts), creation of functional gradient materials, production of single crystals, phosphors, high-temperature superconductors and nanomaterials [8–9].

We used samples from mass-production steels such as steel 20, steel 45, U8, 40H, 40H16M for coating. As reaction agents, we used mixtures of powders with a dispersion of 60–250 microns of the following materials. In determining the required dispersion of the reagents, we were guided by studies that found that the maximum completeness of the transformation is observed when using a reaction mixture with a fraction of 100–120 microns. The protective coatings on samples under the conditions of the CWS were obtained using the developed pilot plant DSTU12, consisting of the following main functional systems: reaction equipment; system for monitoring and controlling technological parameters; and gas utilization system.

When chrome coatings are alloyed with titanium on the steels under consideration, phases are formed  $(\text{Fe, Cr})_{23}\text{C}_6$ ,  $(\text{Fe, Cr})_7\text{C}_3$ ,  $\text{Cr}_3\text{C}_2$ , and phases:  $\text{Fe}_2\text{Ti}$ ,  $\text{Cr}_2\text{Ti}$ . It was found that the alloying elements diffuse deep into the steel to a depth of 210 microns. Al and Ti are evenly distributed over the thickness of the protective chromium coating. Unsteady-state temperature conditions lead to an increase in the concentration of alloying elements in chromium coatings, unlike isothermal conditions.

Although research in the field of SHS processes is widely covered in scientific papers, the following scientific issues and areas have not yet been sufficiently studied:

- creation of structural and microkinetic models;
- mathematical modeling of processes;
- development of non-isothermal kinetics methods;
- development of continuous and combined SHS processes;
- development of technologies for direct production of materials and products.

The existence of limiting temperatures, up to which the propagation of the combustion front is possible, introduces certain restrictions on the use of the combustion mode as a technological one. On the contrary, the thermal autoignition mode is free from these restrictions. by diluting the initial powder mixture

with an inert substance up to 85–90 % wt. %, it is possible to reduce the maximum temperature of the process to the technologically necessary temperatures. The main compounds in the gas phase in the temperature range of 400–1600 K are as follows: SiCl, SiCl<sub>2</sub>, SiCl<sub>3</sub>, SiCl<sub>4</sub>, SiJ<sub>2</sub>, SiJ<sub>3</sub>, AlJ, AlJ<sub>2</sub>, AlCl, AlCl<sub>2</sub>, CrCl<sub>2</sub>, CrF, CrF<sub>2</sub>, CrF<sub>4</sub>, BF<sub>3</sub>, TiCl<sub>2</sub>, TiCl<sub>3</sub>, TiCl<sub>4</sub>, WCl<sub>2</sub>, WCl<sub>3</sub>, WCl<sub>4</sub>, etc., as well as iodine in atomic and molecular form.

The current stage of development of science and technology involves the intensive creation of materials with a qualitatively new level of performance properties, which is achieved by using both new physical and chemical processes and new combinations of known methods to obtain materials. Therefore, one of the promising areas where the method of self-propagating high-temperature synthesis is applied is the technology of applying functional coatings to structural materials [10].

The thermodynamic modeling of thermochemical processes, which consists in the thermodynamic analysis of the equilibrium state of systems as a whole (full thermodynamic analysis), was carried out to study the mechanism of protective coating formation. Thermodynamic systems are conditionally distinguished material areas whose interaction with the environment is reduced to the exchange of heat and work. The use of thermodynamic modeling makes it possible to quantitatively simulate and predict the composition and properties of complex heterogeneous, multi-element, multiphase systems over a wide range of temperatures and pressures, taking into account chemical and phase transformations. This makes it possible to effectively study thermochemical processes in existing high-temperature installations and optimize their conditions, predict the results of high-temperature interactions, dramatically reduce the time and cost of research, and successfully systematize theoretical and experimental information.

The calculation of the thermodynamic equilibrium of arbitrary systems (determination of all equilibrium parameters, thermodynamic properties, chemical and phase composition) is carried out by minimizing the isobaric-isothermal potential or maximizing the entropy of the system, taking into account all potentially possible individual substances  $q$  in equilibrium. Computational methods developed based on the variational principles of thermodynamics suggest that:

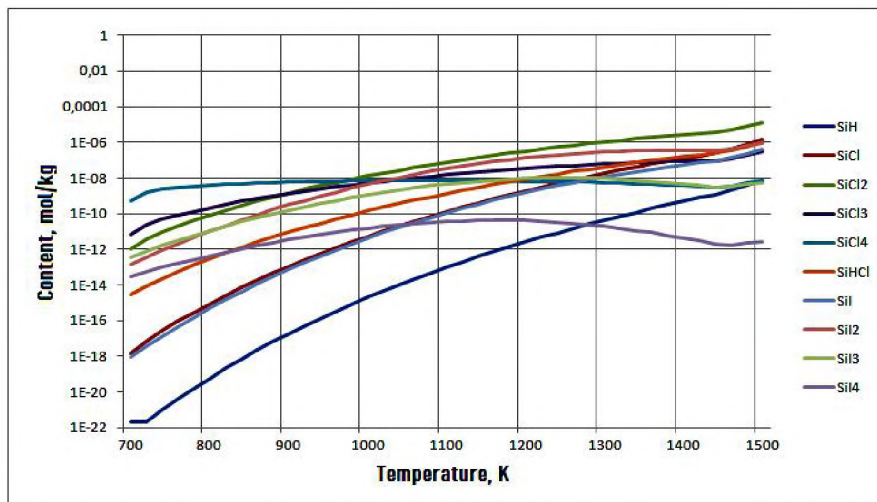
1. Of all the permissible values of moles  $Mq$  of individual substances in a thermodynamic system, those that minimize the thermodynamic potential of the system correspond to equilibrium values;
2. Of all the permissible values of energy  $Ui$  contributed by each independent component (atom), those that maximize the total energy contribution of individual atoms to the system correspond to equilibrium values.

The constituent parts of a system are all possible and existing substances in different aggregate states formed from the elements included in the system under study. The components of a thermodynamic system are the substances that are minimally necessary for the composition of this system. The number of components is equal to the number of substances present in the system minus the number of independent reactions that bind these substances. In thermodynamic modeling, condensed individual substances are compounds with a multiple of the number of atoms that form them.

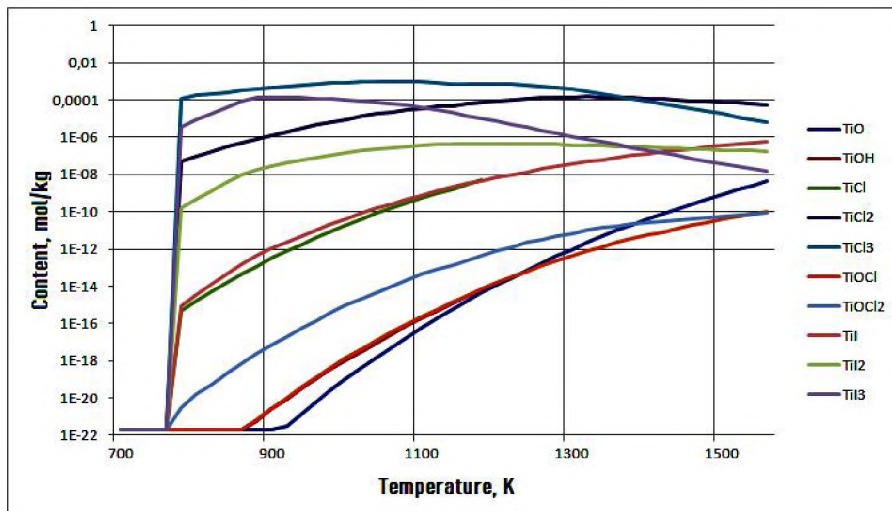
Substances with fractional stoichiometric coefficients are considered solutions. condensed phases include compounds in solid (crystalline or amorphous) and liquid states. Individual substances that have the same chemical formula but are part of different phases are considered to have different constituents. The constituents of the gas phase are molecules, radicals, atoms, ions, and electron gas.

With increasing temperature, the amount of products in the gaseous phase increases, and condensed products are released (Fig. 1, 2). It is characteristic that in the temperature range of 400–1600 K the share of condensed phase decreases, which is associated with the evaporation of the used carriers. simultaneously, starting from the temperature of 800 K, the decomposition of reaction products occurs, essentially chemical transport of elements takes place.

Extensive thermodynamic parameters of the system, i. e. proportional to the amount or mass of matter in the system, are volume  $V$ , entropy  $S$ , internal energy  $U$ , enthalpy  $H$ , Helmholtz energy  $F$  ( $F=U-T-S$ ), Gibbs energy  $G$  ( $G = H-T-S$ ); intensive thermodynamic parameters, i. e. independent of the amount or mass of the system, are pressure  $P$ , thermodynamic temperature  $T$ , concentration, molar and specific thermodynamic values.



**Fig. 1.** Content of gaseous silicon compounds in the reactor in the mode of thermal spontaneous combustion of the SHS-charge



**Fig. 2.** Content of gaseous titanium compounds in the reactor in the mode of thermal spontaneous combustion of the SHS charge for the system

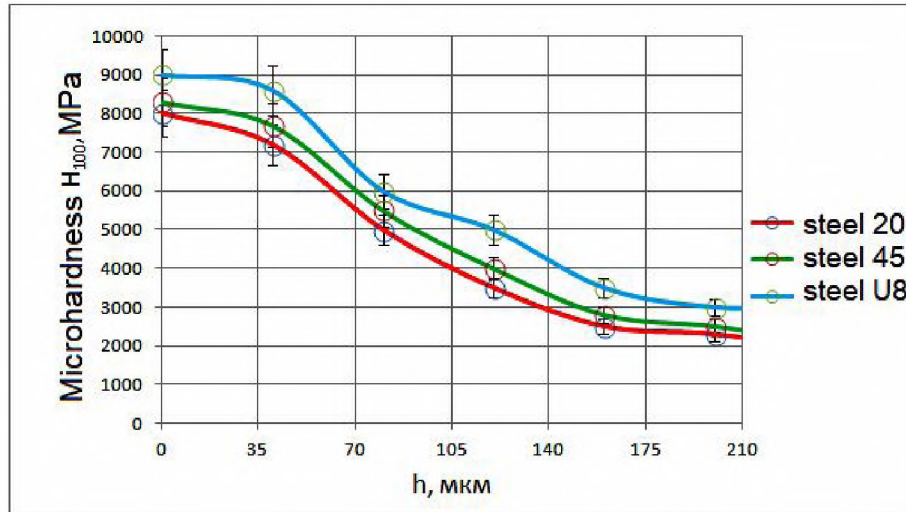
The microhardness of the surface layers of metal products after chemical and thermal treatment, laser and plasma surfacing, metallization, plating, etc. is related to their wear resistance, fatigue strength, as well as the reliability and durability of the finished products. Microhardness is an important characteristic of the physical and mechanical properties of coatings; it determines the resistance of coatings to aggressive environments. This is especially true for the upper layers of the coating, which are primarily in contact with corrosive and erosive media.

Microhardness tests are carried out either with the help of benchtop devices that use the scheme of a vertical portable microscope with a revolving head and direct loading with weights (PMT-2 and PMT-3 devices), or as an attachment to horizontal metal microscopes with a spring load (Hahnemann et al. device). Microhardness tests have found important applications where other methods are not available: 1) determination of the hardness of individual microstructural components; microhardness allows, along with qualitative microscopic examination, to assess the properties of microsites; it changes when moving from the central zones of microgranules to the peripheral ones; 2) determination of the hardness of thin surface layers; conventional hardness tests assess the properties of relatively thick surface layers [11].



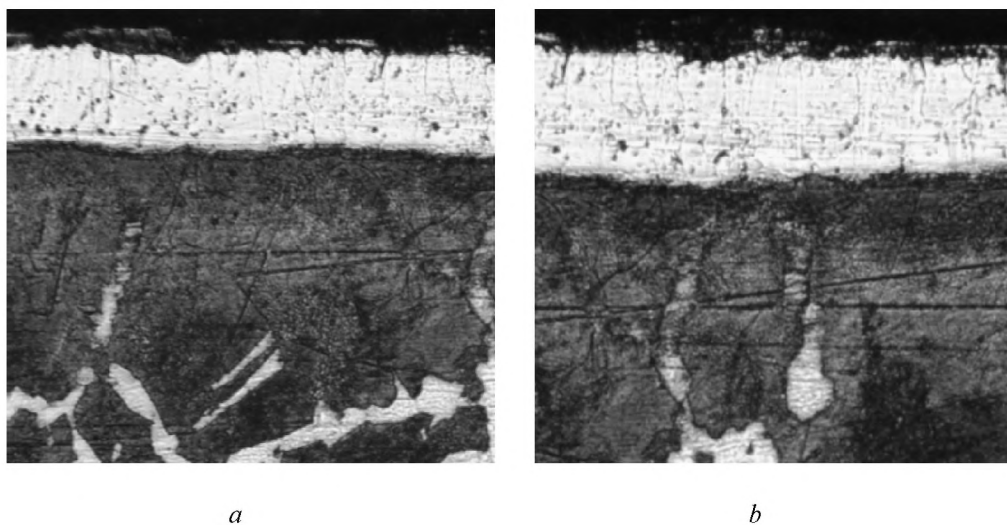
The highest microhardness is found on the surface of the coatings, which gradually decreases to the surface of the material and is equal to the hardness of the material being processed (Fig. 1). This distribution of microhardness minimizes surface punching in service conditions.

The surface microhardness of multicomponent chrome layers alloyed with silicon and boron is 20000–22000 MPa, which is due to the presence of the FeB phase. The microhardness of the alloyed Fe<sub>2</sub>B phase is 15000–16000 MPa. The obtained results indicate that silicon is displaced from the boride zone to the sublayer of the α-phase, alloying only the Fe<sub>2</sub>B.



**Fig. 3.** Distribution of microhardness over the thickness of chrome plating of titanium alloyed coatings on structural materials

Surface microhardness of aluminum-doped titanium coatings (Fig. 1) is: on steel 20–H<sub>100</sub>=8000 MPa (phases: on the surface Fe<sub>2</sub>Al<sub>5</sub>, alloyed Ti and TiAl, under it is a zone of solid solutions FeAl and Fe<sub>3</sub>Al, alloyed with titanium with inclusions of Ti<sub>3</sub>Al, Fe<sub>2</sub>Al<sub>5</sub>), on steel 45–H<sub>100</sub>=8300 MPa (phases: Fe<sub>2</sub>Al<sub>5</sub>, TiAl, Fe<sub>3</sub>Al, Ti<sub>3</sub>Al), on steel U8–H<sub>100</sub>=9000 MPa (phases: Fe<sub>2</sub>Al<sub>5</sub>, TiAl, Fe<sub>3</sub>Al, Ti<sub>3</sub>Al, TiC). According to the research results, it was found that, compared to coatings obtained under isothermal conditions, the microhardness of steel 45 with SHS coatings is 1.8–2.0 times higher.



**Fig. 4.** Microstructures (x100) of titanium and aluminum alloyed coatings on steel 45, depending on the type of alloying ( $t_n = 1000$  °C,  $\tau_0 = 30$  min): a – Cr; b – Al

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Microstructure of structural materials with protective coating was studied from cross-sections on metallographic microscopes “Neophot-21” and “Neophot-32”. The range of image magnification varies from  $\times 100$  to  $\times 500$  times. The preparation and processing of the cross-sections was carried out according to the standard method. To reveal the microstructure of the coatings obtained on steels, a 3 % alcohol solution of picric acid in ethyl alcohol was used.

According to the results of the study, it was found that, in comparison with coatings obtained under isothermal conditions, the microhardness of steel 45 with SHS coatings is 1.8–2.0 times higher, the obtained coatings have a multiphase structure, and are characterized by uniformity in thickness. The surface microhardness of titanium-alloyed chromium coatings is: on steel 20– $H_{100}=16000$  MPa (phases:  $(Cr, Fe)_{23}C_6$ ,  $(Cr, Fe)_7C_3$ , alloyed with titanium,  $Fe_2Ti$ ,  $Cr_2Ti$ ,  $\alpha$ -solid solution Ti and Cr y  $\alpha$ -iron), on steel 45– $H_{100}=18000$  MPa (phases:  $(Cr, Fe)_{23}C_6$ , titanium-doped,  $\alpha$ -solid solution of chromium in iron with inclusions  $Cr_2Ti$ ), on steel Y8– $H_{100}=19500$  MPa (phases: directly on the outside of the coating is a zone of carbides  $(Fe, Cr)_{23}C_6$ ,  $(Fe, Cr)_7C_3$ ,  $Cr_3C_2$ ,  $(Ti, Cr)C$ ). When chromium coatings are alloyed with titanium on the carbon steels under consideration, the following phases are formed  $(Fe, Cr)_{23}C_6$ ,  $(Fe, Cr)_7C_3$ ,  $Cr_3C_2$ , as well as phases:  $Fe_2Ti$ ,  $Cr_2T$ . Such a phase composition is explained by the fact that aluminum and chromium can be in a liquid state at the temperature of SHS treatment and diffuse into the substrate more actively than titanium. In addition, it should be noted that during the treatment process, aluminum can partially be in a liquid state, so titanium diffusion in this case will pass through the liquid phase, thus intensifying the coating formation process.

### **Conclusions**

We obtained a titanium-doped chromium coating to strengthen the working surfaces of parts used in aggressive conditions on equipment for vulcanizing products made of new elastomeric materials based on a copolymer of vinyl dell fluoride and hexopropylene, ethylene-propylene rubber. When chrome coatings are alloyed with titanium on the steels under consideration, the following phases are formed  $(Fe, Cr)_{23}C_6$ ,  $(Fe, Cr)_7C_3$ ,  $Cr_3C_2$ , and phases:  $Fe_2Ti$ ,  $Cr_2Ti$ . It was found that the alloying elements diffuse deep into the steel to a depth of 210 microns. Al and Ti are evenly distributed over the thickness of the protective chromium coating. Unsteady-state temperature conditions lead to an increase in the concentration of alloying elements in chromium coatings, in contrast to isothermal conditions. the surface microhardness of aluminum-alloyed titanium coatings is: on steel 20– $H_{100}=8000$  MPa (phases: on the surface of  $Fe_2Al_5$ , doped with Ti and TiAl, under it is a zone of solid solutions of FeAl and  $Fe_3Al$ , alloyed with titanium inclusions  $Ti_3Al$ ,  $Fe_2Al_5$ ), on steel 45– $H_{100}=8300$  MPa (phases:  $Fe_2Al_5$ , TiAl,  $Fe_3Al$ ,  $Ti_3Al$ ), on steel U8– $H_{100}=9000$  MPa (phases:  $Fe_2Al_5$ , TiAl,  $Fe_3Al$ ,  $Ti_3Al$ , TiC). According to the results of the research, it was found that, compared to coatings obtained under isothermal conditions, the microhardness of steel 45 with SHS coatings is 1.8–2.0 times higher. According to the results of X-ray diffraction and metallographic analyses, it was found that the obtained coatings have a multiphase structure and are characterized by uniformity in thickness. The surface microhardness of the titanium-alloyed chromium coatings is as follows: on steel 20– $H_{100}=16000$  MPa (phases:  $(Cr, Fe)_{23}C_6$ ,  $(Cr, Fe)_7C_3$ , alloyed with titanium,  $Fe_2Ti$ ,  $Cr_2Ti$ ,  $\alpha$ -solid solution of Ti and Cr in  $\alpha$ -iron), on steel 45– $H_{100}=18000$  MPa (phases:  $(Cr, Fe)_{23}C_6$ , titanium-doped,  $\alpha$ -solid solution of chromium in iron with inclusions  $Cr_2Ti$ ), on steel Y8– $H_{100}=19500$  MPa (phases: directly on the outer side of the coating is a zone of carbides  $(Fe, Cr)_{23}C_6$ ,  $(Fe, Cr)_7C_3$ ,  $Cr_3C_2$ ,  $(Ti, Cr)C$ ). When chromium coatings are alloyed with titanium on the carbon steels under consideration, the following phases are formed  $(Fe, Cr)_{23}C_6$ ,  $(Fe, Cr)_7C_3$ ,  $Cr_3C_2$ , and phases:  $Fe_2Ti$ ,  $Cr_2T$ . The surface microhardness of multicomponent chromium layers doped with silicon and boron is 20000–22000 MPa, which is due to the presence of FeB phase. Microhardness of the alloyed phase  $Fe_2B$  is 15000–16000 MPa. The results indicate that silicon is displaced from the boride zone into the  $\alpha$ -phase sublayer, alloying only the  $Fe_2B$  phase.

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