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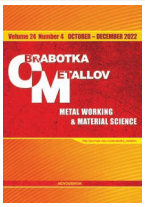

The features of residual stresses investigation in the hardened surface layer of die steels after diffusion boroaluminizing

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ABSTRACT

Introduction. Diffusion boroaluminizing provides improved performance properties of the die steels' surface such as wear resistance, high hardness, and corrosion resistance. Surface hardening can significantly contribute to the occurrence of technological residual stresses (*TRS*) on the surface. Currently, there are no studies on the topic of the stress state of diffusion boroaluminizing. **The purpose** of this work is to develop a method for determining the *TRS* and a nature of its distribution in the diffusion layers on the surface of *5CrNiMo* and *3Cr2W8V* die steels after boroaluminizing by a mechanical method. **The paper considers** the results of experimental studies on the determination of the normal components of *TRS* by the mechanical method in diffusion layers of die steels. The conducted studies showed that the formation of unfavorable tensile *TRS* occurs along the depth of the hardened layer in the case of the investigated *TCT* method and types of steels. **Results and discussions.** The main approaches for determining the *TRS* in the surface layer of *3Cr2W8V* and *5CrNiMo* die steels after *TCT* are considered. Problems in the determination of *TRS* by the mechanical method on the *UDINON-2* unit are identified, and its solution is proposed. The efficiency of using the anodic dissolution method for the continuous removal of stressed layers during the *TRS* study by the mechanical method on the *UDION*-2 unit is shown. The optimal electrolyte composition is selected for the process of anodic dissolution consisting of: $NaNO_3$ – 60 g/l; $NaNO_2$ – 5 g/l; Na_2CO_3 – 5 g/l; $C_3H_8O_3$ – 15 g/l; H_2O – the rest. The distributions of the normal *TRS* components in the diffusion layer of die steel specimens are revealed. It is established that, during the *TCT* of these steels predominantly tensile *TRS* are formed in the surface layer. Further research will be aimed at developing measures to reduce tensile *TRS* during diffusion boroaluminizing of die steels.

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Introduction

Chemical and thermal treatment (*CTT*) of metals is widely used to improve the mechanical properties of machine parts and tools [1]. Carburizing, chrome plating and nitriding are thermochemical processes that improve the wear resistance and corrosion resistance of components [2–12]. One of the effective

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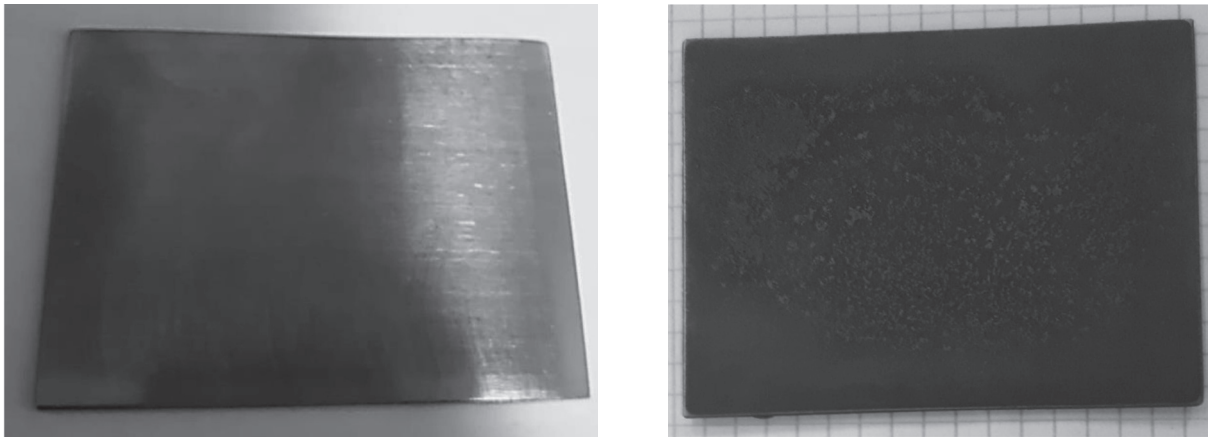
technologies for modifying the surface layers of die steels is multicomponent *CTT*, such as boroaluminizing, which allows increasing significantly the wear resistance, as well as heat resistance, corrosion resistance and a number of other properties of the surface layers of machine parts and tools [13–16]. The service life of products after hardening largely depends on the distribution of technological residual stresses (*TRS*) in the diffusion layer and adjacent layers of the base material and on the general nature of the microstructure of the entire section changed during the *CTT* process [8,17–20]. Control and management of *TRS* is one of the most important tasks of mechanical engineering [21, 22]. Therefore, when developing *Fe-Me-B* coatings by diffusion alloying methods on the surface of steel products, efforts should be directed to finding *TRS* distributions that improve the operational properties of products.

It is known that the study of the stress-strain state (*SSS*) of borated layers can be carried out by the methods of destructive and non-destructive testing [23–26].

In our work [27], the first data on the assessment of the stress state of boride layers were presented. In this paper, we review the methods for determining the *TRS* that can be used after hardening *CTT*, in particular, in *Fe-Me-B* coatings in the surface layer of carbon and alloy steels, and also consider the problems of measuring residual stresses by mechanical method and its solution. The results of experimental studies on the detection of the *TRS* distribution in the diffusion layers of *3Cr2W8V* and *5CrNiMo* tool steels after high-temperature boroaluminizing (*HBA*) are presented.

Research methodology

CTT was carried out in saturating pastes containing powders of boron carbide, aluminum and sodium fluoride as an activator of the following composition: 80 % B_4C + 16% *Al* + 4% *NaF* [15]. Samples with overall dimensions of 1.8×80×60 mm (Fig. 1, *a*) were made of *3Cr2W8V* and *5CrNiMo* tool steels (see Tables 1, 2). After tamping, the molds were removed, and the resulting briquettes were dried at a temperature of 50–100 °C for two hours in a drying chamber. After that, the briquettes were loaded into a furnace preheated to the process temperature. The treatment duration was 2 hours, the temperature was 950 and 1,050 °C. The samples were cooled outside the furnace in calm air at room temperature. The sample after *CTT* is shown in Fig. 1, *b*.



a *b*

Fig. 1. Flat samples

Table 1

Chemical composition of *5CrNiMo* steel, wt. %

C	*Si*	*Mn*	*Cr*	*Mo*	*Ni*	*P*	*S*	*Cu*
0.50–0.60	0.10–0.40	0.50–0.80	0.50–0.60	0.15–0.30	1.40–1.80	≤ 0.03	≤ 0.03	≤ 0.30

Table 2

Chemical composition of *3Cr2W8V* steel, wt. %

C	*Si*	*Mn*	*W*	*V*	*Cr*	*Ni*	*P*	*S*	*Cu*
0.3–0.4	0.15–0.4	0.15–0.4	8.5–10.0	0.3–0.6	2.2–2.7	< 0.35	≤ 0.03	≤ 0.03	≤ 0.03

Metallographic analysis was performed on an optical microscope *"METAM RV-34"* with a digital camera *"Altami Studio"*.

The stress state of boride coatings after *CTT* was measured using an installation for determining residual stresses *UDION-2* (Fig. 2), developed at *IRNTU* [23–27].

Elementary strip samples were cut from the initial plate samples on the *Discotom-10* cutting machine (*Struers*, *Denmark*) using an abrasive disc cutting wheel with the use of abundant cooling (Fig. 3, *a*). The strips were cut in a mutually perpendicular direction along the *X* and *Z* axes (Fig. 3, *b*), the nominal length of the strips was 60 mm, width – 8 mm.

After cutting out the strip samples, its geometric parameters (width *b* and thickness *h*), bend of deflection and initial mass were measured. This data is necessary for the subsequent calculation of the *TRS*.

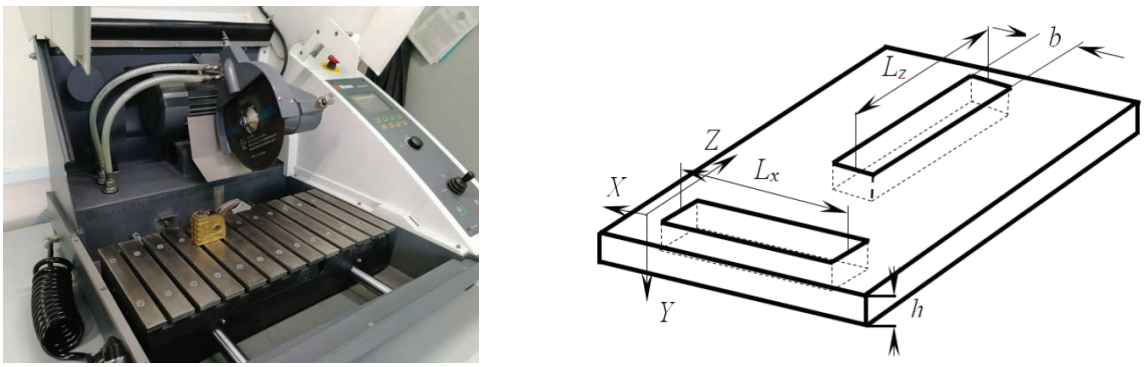
The sample strips were mounted in the devices of the *UDION2* installation and its electrochemical etching (anodic dissolution) was carried out in the electrolyte of composition No. 3 (Table 4) with simultaneous recording of the real time sample deformation. After etching, the samples were dismounted and re-weighed; the average etching rate was determined by formula (1). The final stage was the calculation of the *TRS* in the *XUdion* software [28] and the construction of *TRS* distribution plots.

Fig. 2. Installation for measuring residual stresses by mechanical method (*UDION-2*)

Results and discussions

The problem of research of TRS by mechanical method

Let us denote the problems associated with the measurement of residual stresses by the mechanical method after strengthening thermal and chemical treatment. The determination of *TRS* by the mechanical method at the *UDION-2* installation [23–28] is associated with the registration of elementary samples-strips motion (when measuring residual stresses in plates) during the removal of stressed layers of the material



a *b*

Fig. 3. Cutting elementary samples-strips from initial plates

under study as a function of the thickness of the removed layer. Removal of stressed layers from the surface under study in this installation is realized by the means of chemical etching – gradual dissolution of the material in electrolyte solutions. Basically, the compositions of electrolytes used in chemical etching are a combination of solutions of inorganic acids with water. At the same time, in order to ensure accuracy and minimize measurement error, it is necessary to ensure the following surface quality parameters during the etching process: low roughness, absence of pitting, undercuts under the protective coating, pits, non-scratches, and, if possible, to achieve a constant etching rate throughout the process. For a stable flow of the process and the establishment of the optimal duration of the experiment, the recommended etching rate should be 0.005–0.01 mm/min.

When processing products by *CTT* methods [1, 13–16], the surface layer of the metal acquires the following properties:

– decrease in magnetic permeability;
– increase in electrical resistance;
– increase in torsional stiffness;
– increase in hardness;
– increase in wear resistance;
– increase in corrosion resistance, in particular acid resistance.

The latter property makes it difficult to study the *TRS* in products subjected to the types of *CTT* under consideration, at least during chemical etching in acid solutions. To confirm this, we conducted a number of studies on the chemical etching of test samples made of 3*Cr2W8V* steel after boroaluminizing in various compositions of acid electrolytes (Table 3). The test samples were made in the form of strips with overall dimensions of 1.7×8×60 mm. An area of 80 mm^2 was etched, rubber enamel was used to protect the rest of the sample. Etching was carried out in a small volume of electrolyte, not exceeding 200 ml, in a thermostatic bath of the *UDION2* installation with a control of the average rate of removal of layers (etching), calculated by the formula [29]:

$$\bar{V}_{\text{etching}} = \frac{1000\Delta m}{S\rho\tau}, \text{ mm/min} \quad (1)$$

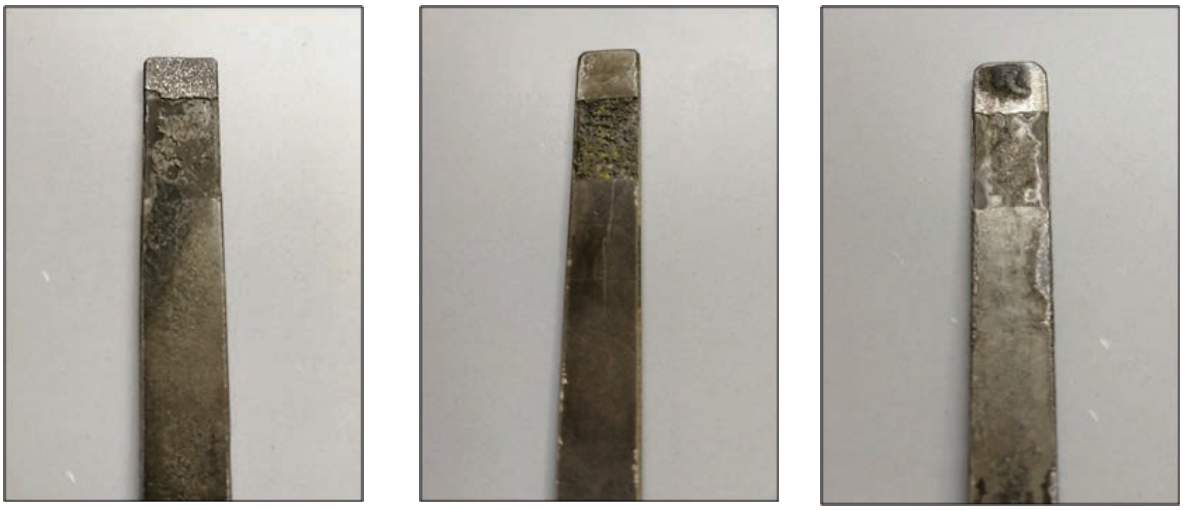
where Δm – weight of the removed material, g; S – the surface area from which the material was removed, mm^2; ρ – material density, g/sm^3; τ – test time (etching), min.

Table 3

Electrolyte compositions, parameters, and results of chemical etching of samples after CTT

Composition No.	Electrolyte composition		t, °C	$\bar{V}_{\text{etching}}$, mm/min	Comments
1	(g/l):	HF – 30; H_2SO_4 – 150; H_2O – rest	23	–	There are practically no signs of poisoning
2	(g/l):	HF – 120; H_2SO_4 – 590; H_2O – rest	35	0.0014	The surface is dark gray with large amounts of non-etched areas (Fig. 4, *a*)
3	(% vol.):	HF – 10; H_2SO_4 – 8; HNO_3 – 9; H_2O – rest	35	0.0076	Black relief surface with numerous small bumps and depressions (Fig. 4, *b*)
4	(% vol.):	HCl – 565; HNO_3 – 237; NaF – 30; H_2O – rest	37	0.0063	Gray wavy surface with a non-etched area in the middle (Fig. 4, *c*)

Composition No. 1 was taken as the initial electrolyte (Table 3) [29], this electrolyte showed a poor result, the surface of the sample was practically not etched. Composition No. 2 was obtained by increasing the concentration of components of composition No. 1 with an increase in the temperature of the solution. Composition No. 3 is a modification of the previous composition with the addition of nitric acid as an oxidizer. Composition No. 4 is focused on chemical dimensional processing of steels [30]. The condition of the samples surface after chemical etching is shown in Figure 4.



a *b* *c*

Fig. 4. The surface of specimens as a result of chemical etching of specimens made of *3Cr2W8V* steel after *CTT*:

a – composition No. 2; *b* – composition No. *3*; c – composition No. 4

As can be seen from the experiments, chemical etching does not give acceptable results, therefore, for layer-by-layer removal of material from samples after *CTT*, it was proposed to use electrochemical etching, in particular anodic dissolution, when studying the *TRS* at the *UDION*-2 installation.

The removal of the material during electrochemical treatment occurs under the action of an electric current in the electrolyte medium. The electrochemical treatment process is based on the phenomenon of anodic dissolution of metals. Anodic dissolution can take place in electrolytes of different compositions, including non-aggressive ones – aqueous solutions of salts that are cheap and harmless [31, 32] compared to acidic ones.

Under the action of an electric current in the electrolyte, the anode material, which is the test sample, dissolves in the form of processing products. The cathode does not wear out, which is one of the positive features of the process. As a result of the reactions, hydrogen is released at the cathode, and precipitation in the form of insoluble metal hydroxide and oxygen occurs at the anode. Insoluble hydroxide clutters up the electrolyte and reduces the productivity of the process. In this regard, it is necessary to make provision for the processes of regeneration and purification of the electrolyte from the reaction products (settling, filtration, decantation).

To work out the process, parameters and composition of the electrolyte, a number of studies were conducted on the anodic dissolution of *3Cr2W8V* steel samples after *CTT* (Table 4). For this, an electrochemical cell was placed in the thermostatic bath of the *UDION-2* installation (Fig. 5), a lead cathode connected to the negative output of the power source, and a sample anode connected to the positive output of the power source, was placed in the thermostatic bath. As a power source for the electrochemical circuit, a *Mastech HY3010* laboratory *DC* power source was used with output voltage regulation in the range of 0–30 V at an output current of 0–10 A, with the function of a stabilized current source. As in the case of chemical etching during anodic dissolution, the average rate of layers removal was calculated according to the formula (1).

Table 4

Electrolyte compositions; parameters, and results of electrochemical etching (anodic dissolution) of samples after *CTT*

Composition No.	Electrolyte composition		t, °C	Current density j, A/dm^2	Voltage U, V	$\bar{V}_{TP}$, mm/min	Comments
1	(%weight.):	KNO_3 – 12; NaF – 2; $NaCl$ – 1; H_2O – rest	30	75	5	0.0062	The surface is dark gray, smooth, even in the center, with a slight undulation along the edges (Fig. 6, *a*)
2	(%weight.):	H_3PO_4 – 49,5; H_2SO_4 – 40; H_2O – rest	32	100	8.5	0.007	Textured light surface with metallic luster, case of electrolytic polishing (Fig. 6, *b*)
3	(g/l):	$NaNO_3$ – 60; $NaNO_2$ – 5; Na_2CO_3 – 5; $C_3H_8O_3$ – 15; H_2O – rest	30	100	10	0.0056	The surface is dark gray, matte smooth, smooth with a few minor micro-steps (Fig. 6, *c*)

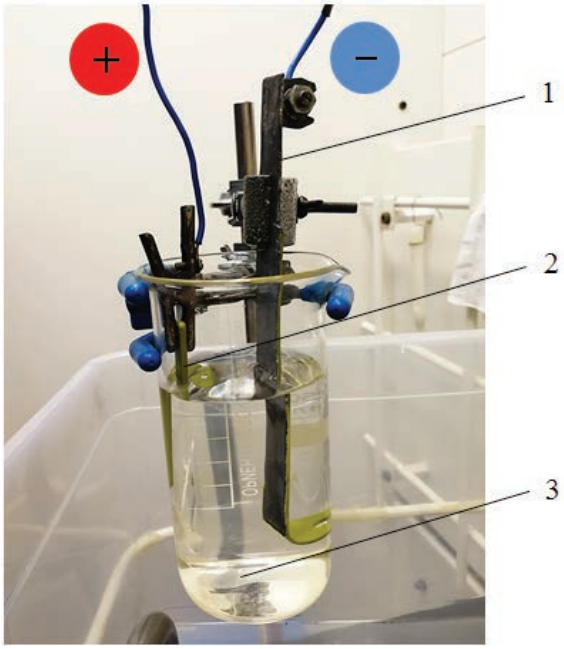
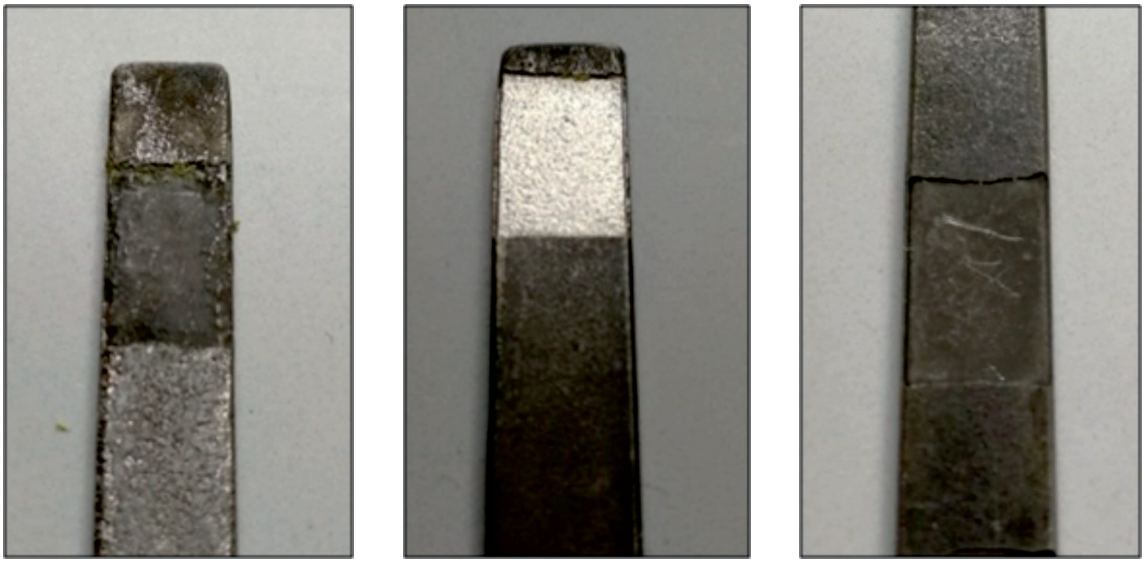


Fig. 5. Electrochemical cell for testing the process of anodic dissolution:
1 – cathode (lead plate); *2* – anode (sample); *3* – beaker with electrolyte solution

Composition No. 1 (see Table 4) [30], recommended for dimensional electrochemical machining of tool steels, generally showed good results, except for a slight surface waviness (Fig. 6, *a*). Composition No. 2 [29] gives a shiny, though textured, smooth surface (Fig. 6, *b*), but only for this type of steel. Composition No. 3 recommended for abrasive-electrochemical machining of metals [33] showed good results, the surfaces of the samples are matte smooth gray with minimal roughness (Fig. 6, *c*).

Preparatory studies have shown that for the layer-by-layer removal of material in the study of *TRS* by mechanical method in samples after diffusion boroaluminizing, the most acceptable results are obtained by the process of anodic dissolution. It should be noted that in each specific case, the selection of the electrolyte composition and the necessary electrical and temperature conditions is carried out individually for each material, taking into account the type of its treatment; this procedure is time-consuming and requires test samples for testing the process.

In these studies, the composition of electrolyte No. 3 for anodic dissolution showed the best result (Table 4). This electrolyte also showed good results with anodic dissolution of the surface layer of *5CrNiMo* steel samples after *CTT*. To control the process of anodic dissolution of the material, in particular, to establish its required speed, adjustment of the electrolyte temperature and electrical parameters of the process (voltage, anode current density) can be applied.



a *b* *c*

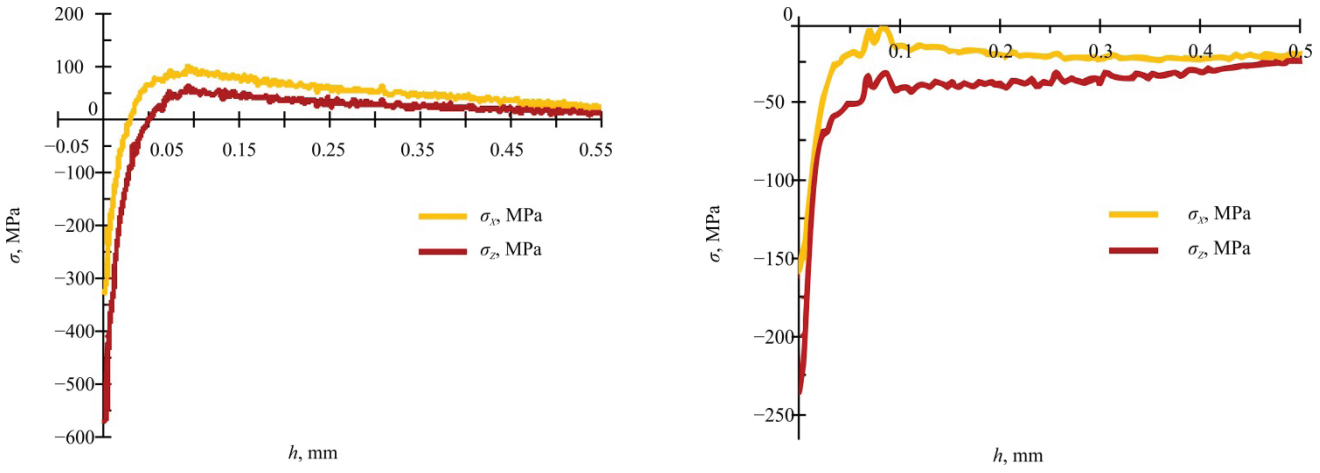
Fig. 6. The surface of specimens as a result of electrochemical etching of specimens made of *3Cr2W8V* steel after *CTT:*

a – composition No. 1; *b* – composition No. 2; *c* – composition No. 3

Investigation of TRS after diffusion boroaluminizing

Control samples after annealing (Fig. 7) are characterized by the presence of compression *TRS* on its surface: for *5CrNiMo* steel sample (Fig. 7, *a*) the compression *TRS* values lie in the range of −325…−570 MPa with a sharp rise and specifying near-zero values from a depth of 0.05–0.075 mm; for *3Cr2W8V* steel sample (Fig. 7, *b*) these values are in the range of −155…−235 MPa. Moreover, the *TRS* plot after annealing of the *5CrNiMo* steel plate is self-balanced (Fig. 7, *a*).

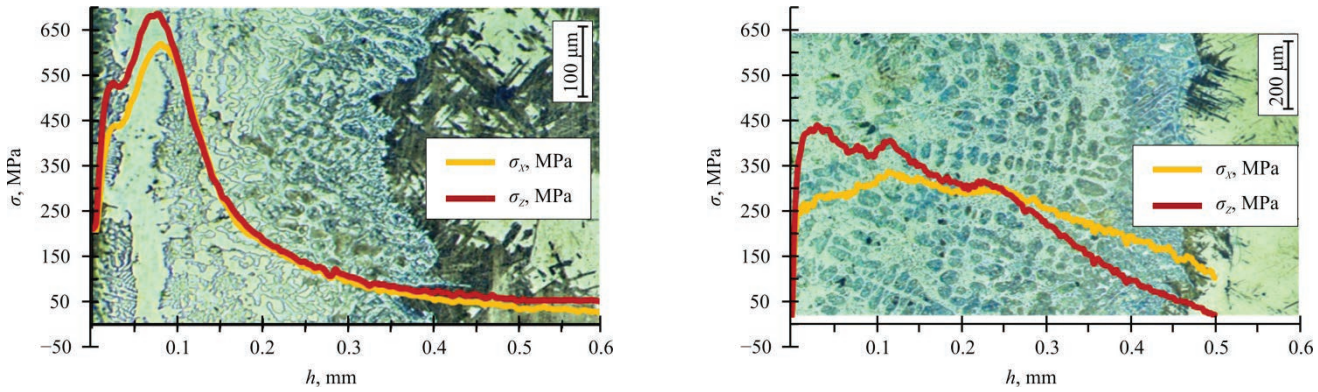
After the *CTT* of *5CrNiMo* steel at a temperature of 950 °C (Fig. 8, *a*), a diffusion layer with a thickness of 400–450 μm is formed [15], in which the *TRS* components are tensile. On the surface of the sample, it takes values of 210 MPa, at a depth of 0.08 mm it reaches a maximum of 620–687 MPa and gradually decrease, taking zero values at a depth of 0.9 mm. The depth of the *TRS* in this case is estimated to be within 0.5 mm.



a *b*

Fig. 7. Diagrams of residual stresses in a plate:

a – of steel *5CrNiMo* after annealing at 790 °C; *b* – of steel *3Cr2W8V* after annealing at 880 °C



a *b*

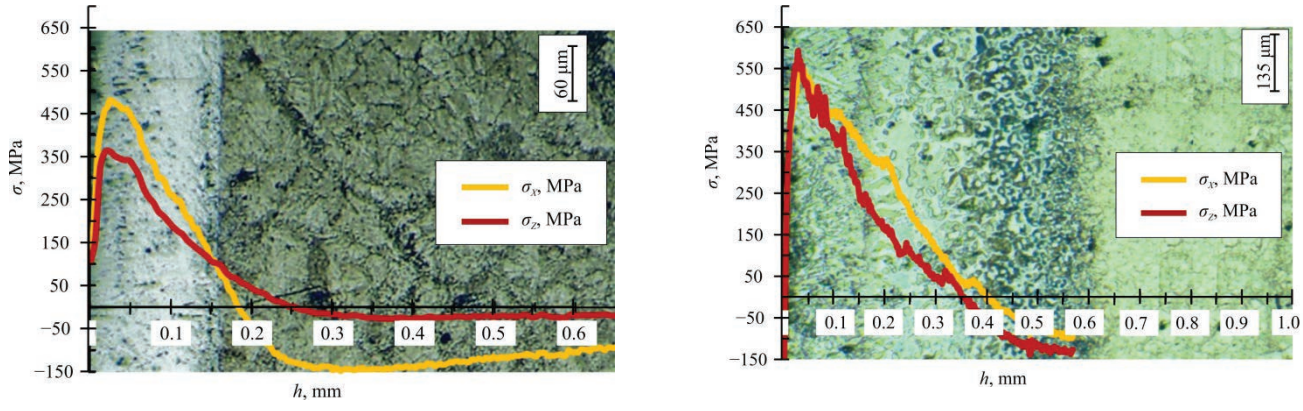
Fig. 8. Diagrams of residual stresses in a plate made of *5CrNiMo* steel at a treatment temperature:
a – 950 °C; *b* – 1,050 °C

After the *CTT* of *5CrNiMo* steel at a temperature of 1,050 °C (Fig. 8, *b*), layers with a thickness of 750–900 μm are formed [15], in which the components are tensile; on the surface take near-zero values. The component σ_Z has a maximum of 436 MPa at a depth of 0.05 mm with a further general downward trend, although with slight increases in values at depths of 0.2 and 0.375 mm. The σ_X component has at first a sharp rise to 250 MPa, and then a smooth rise to 330 MPa at a depth of 0.2 mm, and then a general downward trend with a slight increase in values at a depth of 0.4 mm. The depth of the *TRS* in this case is more than 0.8 mm.

It can be noticed that the component σ_X does not have the first peak value, as the component σ_Z does, although they both have insignificant areas of increasing values with a general downward trend at almost the same depths.

After the *CTT* of *3Cr2W8V* steel at a temperature of 950 °C (Fig. 9, *a*) at a depth of the diffusion layer up to 130–150 mm [15], two zones are observed on the *TRS* plot: tensile and compressing – so it can be said that the *TRS* plots in this case are self-balanced. On the surface, the *TRS* components have values of 100 MPa and reach a maximum of 360–470 MPa at a depth of 0.025 mm, then a dropping occurs and at a depth of 0.18–0.25 mm it turns into compressive (σ_X = –150 MPa at a depth of 0.3 mm).The depth of the *TRS* is 0.3 mm.

After *HBA* of *3Cr2W8V* steel at a temperature of 1,050 °C (Fig. 9, *b*) in a diffusion layer with a depth of 500–600 μm [15], the components of the *TRS* on the surface are compressive with values of −125...−210 MPa, but abruptly turn into tensile and reach a maximum of 565 MPa at a depth of 0.03 mm. Then there is a smooth dropping of the components to zero at a depth of 0.35–0.4 mm and a transition to the



a *b*

Fig. 9. Diagrams of residual stresses in a plate made of *3Cr2W8V* steel at a processing temperature:
a – 950 °C; *b* – 1,050 °C

compressive region up to −100...−130 MPa. The depth of occurrence of the *TRS* at the same time is more than 0.55 mm.

Since the *CTT* processes take place in the entire volume of the material and form a flat *SSS* of a homogeneous type, i.e. any direction in the processing plane is the main one, the components of the *TRS* should be the same in all directions.

Similar diffusion layers obtained on the surface of *5CrNiMo* and *3Cr2W8V* steels by boroaluminizing can be classified as composite layers with heterogeneous dispersed morphology of the boride crystals arrangement [35, 36]. These layers are characterized by a complex distribution of microhardness in depth, explained by the gradient distribution of *B*, *Al* and alloying elements from the base steel [15]. It was shown in [15] that as a result of *CTT* on *5CrNiMo* and *3Cr2W8V* steels at a temperature of 1,050 °C and on *5CrNiMo* steel at a temperature of 950 °C, diffusion layers are formed on the surface of the steel, in which solid structural components (borides and carbides) are arranged in a matrix of plastic phases (aluminides, solid solutions of aluminum and carbon in *α-Fe*). The obtained properties have a positive effect on the wear resistance of the working surface, however, the nature of the *TRS* distribution in the diffusion layers and its dependence on the *SSS* obtained as a result of the strengthening *CTT* remains understudied.

As is known, tensile properties are unfavorable and can lead to cracks and destruction of the product, increase intercrystalline corrosion, contribute to fatigue failure, especially for parts operating under shock and alternating loads. In this case, it is necessary to provide a set of measures to reduce the tensile *TRS* after *CTT* or to form favorable compression *TRS*. As such measures can be proposed: subsequent heat treatment (tempering), quenching with subsequent tempering, plasma or laser treatment, elaboration of modes and technologies of *CTT*, etc. which will be a continuation of further research.

Conclusions

The main methods for determining the *TRS* in the surface layer after strengthening by the *CTT* methods of tool die steels *5CrNiMo* and *3Cr2W8V* are considered. Problems are identified in determining the *TRS* by mechanical method on the *UDINON-2* installation in samples after diffusion boroaluminizing, and its solution are proposed. The expediency of using the anodic dissolution method for continuous removal of stressed layers from treated samples in the study of *TRS* by mechanical method on the *UDION-2* installation is shown. The optimal electrolyte composition for the anodic dissolution process is selected, consisting of: $NaNO_3$ – 60 g/l; $NaNO_2$ – 5 g/l; Na_2CO_3 – 5 g/l; $C_3H_8O_3$ – 15 g/l; H_2O – the rest. The distributions of *TRS* normal components in the diffusion layer of samples from die steels after boroaluminizing are revealed. It is established that the formation of predominantly tensile *TRS* occurs in the surface layer of the *CTT* of these steels. Further research will be aimed at developing technological methods to reduce the tensile forces during diffusion boroaluminizing of die steels.

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Conflicts of Interest

The authors declare no conflict of interest.