Investigation of the structure and properties of the surface composite layer on VKS-5 steel

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Abstracts. In this work, were studied the structure and properties of the surface composite layer on heat-resistant steel VKS-5, obtained by combined chemical and thermal treatment, combining liquid electrolysis-free boriding with gas and ion-plasma nitriding. The properties of the surface composite layer are analyzed depending on various parameters of chemical and thermal treatment to determine the optimal mode. The study of the microstructure and properties of borated layers has shown that the most optimal properties can be obtained in the boron mode at a temperature of 1010 °C with an exposure time of 8 hours. The study of the microstructure of composite layers has shown that gas nitriding produces a layer of 80 microns in size, which penetrates deep into the borated layer, and in ion-plasma nitriding, the layer size is so small that it is not possible to determine it using optical microscopy. A study of the properties of composite layers obtained by combined chemical-thermal treatment has shown that combined chemical-thermal treatment, combining boriding and ion-plasma nitriding, allows you to get the best combination of high hardness and reduced microfragility.

Key words: microfragility, boriding, nitriding, combined chemical and thermal treatment, microhardness

1. Introduction

Due to the tightening of operating modes, previously used for the manufacture of gears, high-nickel steels no longer meet the increased requirements and gave way to heat-resistant steels of the VKS group, which make it difficult to break up martensite and shift the processes of low tempering towards higher temperatures, by reducing the concentration of Ni, adding carbide-forming elements such as Cr, W, Mo, V, Nb, and adding Si.

VKS group steels are widely used for manufacturing gears of gas turbine engines in aircraft and rocket engineering. The most rational alloying is typical for VKS-5 steel, due to which it is able to maintain high hardness for a long time and ensure reliable operation of gearboxes and aggregates [1].

To obtain the necessary set of properties and increase the durability and reliability of gears, it is necessary to strengthen the surface of the parts. Surface hardening is possible in several ways: chemical and thermal treatment (cementation [2] or corona discharge treatment [3], nitriding [4]), Electromechanical treatment [5], plastic deformation [6].

Recently, there has been an increased interest in boriding, which makes it possible to obtain diffusion layers characterized by extremely high hardness (about 2000 HV) [7]. Borated layers can significantly increase wear resistance compared to other diffusion layers [8-10]. But at the same time, borated layers are characterized by critically high brittleness, which makes it impossible to apply this type of chemical and thermal treatment in many areas of mechanical engineering [11, 12]. One of the promising methods for reducing the brittleness of borated layers is combined chemical and thermal treatment, which allows creating a multi-layer surface composite layer that does not have a significant micro-moisture gradient, which will increase the reliability of parts processed by boriding and expand the scope of boriding application [13, 14]. In [15-17], successive and simultaneous saturation of the surface of parts made of 4x3mvme and 2X9V6 steels with boron and nitrogen was performed, which led to a decrease in the brittleness of the borated layer.
2. Materials and methods of research
Heat-resistant complex-alloyed martensite hardening steel VKS-5 was selected for research. The composition of the steel under study is given in [18].

Liquid electrolysis-free boriding was performed in a melt based on sodium tetraborate and alkali metal halides at a temperature of 910 °C with an exposure time of 4, 6, 8 hours and at a temperature of 1010 °C with an exposure time of 2, 4, 6, 8 hours.

After boriding, the samples were quenched in oil, followed by low tempering at a temperature of 250 °C with an exposure time of 3 hours to relieve stress.

Gas nitriding was performed at a temperature of 550 °C with an exposure time of 30 hours. Ion-plasma nitriding was performed at a temperature of 550 °C with an exposure time of 18 hours.

After combined chemical and thermal treatment, the samples were not subjected to mechanical processing.

The study of the microhardness distribution over the depth of the diffusion and composite layers was performed on cross sections using an automatic Durascan-70 microhardness meter at a load of 100 g.

The microstructure and depth of the diffusion layer were studied using an Olympus optical microscope at various magnifications.

X-ray phase analysis was performed on a DRON-6 diffractometer in iron Kα radiation using a manganese filter.

The study of microdroplet carried out according to the method described in [19]. The brittleness factor was evaluated depending not only on the number of prints with cracks or on the number of cracks in the print, but also on their nature. We determined the average fragility score, which is set on a 5-point scale given in [20]. The total brittleness score $Z_p$ was determined by the formula:

$$Z_p = 0 \cdot n_0 + 1 \cdot n_1 + 2 \cdot n_2 + 3 \cdot n_3 + 4 \cdot n_4 + 5 \cdot n_5$$

(1)

Where $n_0$, $n_1$, $n_2$, $n_3$, $n_4$, $n_5$ – relative numbers of prints out of the total number with a given fragility score.

To take into account the rate of increase in brittle material destruction with increasing load $P$, the ratio of the increment of the total brittleness score to the increment of the load was calculated using the formula:

$$\frac{\Delta Z}{\Delta P} = \left(\frac{\partial Z}{\partial P}\right)_P$$

(2)

The product of the total brittleness score by the value of its load derivative is considered to be an indicator of the brittleness of a material that reflects the nature of brittle failure and the rate of its increase with increasing load. The brittleness index was determined by the formula:

$$\gamma_p = Z_p \left(\frac{\partial Z}{\partial P}\right)_P$$

(3)

For each sample, at least 30 measurements were performed after different values of the microhardness meter load.

3. Results and discussion
The study of the microstructure and depth of the obtained borated layers revealed the relationship between the parameters of the boron process and the depth of the resulting layers. The boriding modes and depth of the obtained layers are shown in table 1.

| Mode, № | $T_{bor}$, °C | Duration, h | Heat treatment | The depth of the layer, µm |
|---------|---------------|-------------|----------------|--------------------------|
| 1       | 910           | 4           | Hardening in oil, tempering at a temperature of 250 °C exposure 3 h | 70-75, 85-90, 40-45 |
| 2       | 910           | 6           | Hardening in oil, tempering at a temperature of 250 °C exposure 3 h | 60-70, 40-45, 40-45 |
| 3       | 910           | 8           | Hardening in oil, tempering at a temperature of 250 °C exposure 3 h | 70-75, 85-90, 40-45 |
| 4       | 1010          | 2           | 250 °C exposure | 40-45, 90-95, 140-150 |
| 5       | 1010          | 4           | 3 h           | 45-60, 90-95, 140-150 |
| 6       | 1010          | 6           | 3 h           | 45-60, 90-95, 140-150 |
| 7       | 1010          | 8           | 3 h           | 45-60, 90-95, 140-150 |

Found that the borated layer depth is significantly affected by temperature and duration of boriding, the minimum depth of the layer obtained at 1010 °C with exposure time of 2 h and maximum at the temperature of 1010 °C with 8 h aging temperature has a greater influence on the depth of the layer than the exposure time.
The microstructure study showed that the borated layers have a weakly pronounced needle-like structure. The microstructure of the boron layer obtained by mode 7 is shown in figure 1.

![Microstructure of the borated layer](image)

**Figure 1.** Microstructure of the borated layer

The structure is represented by two boride phases, the dark layer corresponds to the FeB phase, and the lighter layer corresponds to the Fe₂B phase. The presence of light particles of different sizes was revealed, presumably these are carbide, boride, and carboride phases of alloying elements. The study of the microhardness distribution over the surface of the section after modes 1, 2, and 3 is shown in figure 2.

![Microhardness distribution](image)

**Figure 2.** Microhardness distribution

The smoothest distribution of microhardness is found in samples processed according to mode 3. The study of the microhardness distribution over the surface of the section after modes 1, 2, and 3 is shown in figure 3.
The smoothest distribution of microhardness is found in samples processed according to mode 7. The distribution of microhardness over the surface of the section after modes 3 and 7 is compared. The samples processed according to mode 7 have the smoothest distribution of microhardness. The study showed that samples processed under different modes have equal values of microhardness, but the microhardness distribution curve becomes smoother with increasing depth of the borated layer.

The study of the microstructure of the composite layer obtained by combined chemical-thermal treatment, combining boriding and subsequent nitriding, is shown in figure 4.

It was found that after gas nitriding, a dark nitrogen-containing layer is superimposed on top of the borated layer, which penetrates to a depth of 90-95 microns, and after ion-plasma nitriding, there is no dark nitrogen-containing layer in the borated layer. The study of the microhardness distribution over the surface of the section after combined chemical-thermal treatment, combining boriding and gas and ion-plasma nitriding, is shown in figure 5.

**Figure 3.** Microhardness distribution

The smoothest distribution of microhardness is found in samples processed according to mode 7. The distribution of microhardness over the surface of the section after modes 3 and 7 is compared. The samples processed according to mode 7 have the smoothest distribution of microhardness. The study showed that samples processed under different modes have equal values of microhardness, but the microhardness distribution curve becomes smoother with increasing depth of the borated layer.

**Figure 4.** Microstructure of the composite layer, X200 (a - boriding and gas nitriding, b - boriding and ion-plasma nitriding)

It was found that after gas nitriding, a dark nitrogen-containing layer is superimposed on top of the borated layer, which penetrates to a depth of 90-95 microns, and after ion-plasma nitriding, there is no dark nitrogen-containing layer in the borated layer. The study of the microhardness distribution over the surface of the section after combined chemical-thermal treatment, combining boriding and gas and ion-plasma nitriding, is shown in figure 5.
Figure 5. Microhardness distribution
It is established that the microhardness distribution curve after gas nitriding has a significant gradient of microhardness values during the transition from the nitried layer to the borated layer and then to the core. After ion-plasma nitriding, the microhardness distribution curve is smooth.

The diffractogram of samples after gas nitriding is shown in figure 6.

Figure 6. Diffractogram of the sample surface after gas nitriding

It is established that on the sample surface provides the boride and nitride phases, revealed the presence carbobor.

The diffractogram of samples after ion-plasma nitriding is shown in figure 7.
Figure 7. Diffractogram of the sample surface after ion-plasma nitriding
It was found that, despite the absence of external changes in the microstructure, nitride phases were present on the sample surface, and the presence of carbobor and chromium nitrides was revealed. The results of the microfragility study are shown in figure 8.

Figure 8. The indication of the fragility
It has been found that gas nitriding can significantly reduce the micro-fragility of the layer than ion-plasma nitriding, but ion-plasma nitriding allows maintaining high hardness of borated layers.

4. Conclusion
In the studied processes of combined chemical and thermal treatment, the concept of sequential saturation of the surface with diffusants was implemented, which led to the creation of a multi-layer composite layer consisting of nitried and borated layers. Combined chemical-thermal treatment, which combines boriding and gas nitriding, can significantly reduce the values of microhardness, but the values of microhardness are greatly reduced. Combined chemical-thermal treatment, combining boriding and ion-plasma nitriding, allows to reduce the values of microfragility and maintain high values of microhardness.

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