Investigation of the structure and properties of surface composite layers on multi-alloyed steels

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Abstract. In this work were studied the structures and properties of surface composite layers obtained by chemical-thermal treatment and combined chemical-thermal treatment, combining liquid-free electrolysis-free boriding with gas nitriding. The properties of surface composite layers on steels with different alloys are analyzed depending on various parameters of chemical-thermal treatment. The study showed that, during boriding, phases of iron borides FeB, Fe2B are formed on the surface of the samples, and their volume ratio strongly depends on the degree of steel alloying. A strong effect of carbon on the morphology of surface layers was revealed. Finely dispersed strengthening particles were found in the borated layers on the VKS-5 steel, large superhard particles on the Ch12MF steel near the borated layers. The study of the microstructure of the composite layers showed that during gas nitriding, a layer 80 μm in size is obtained, which penetrates deep into the borated layer. The study of the properties of surface layers showed that carbon has the greatest influence on the formation of borated layers. The resulting composite layers are characterized by reduced microbrittleness and increased corrosion resistance.

1 Introduction

Boriding, as a unique method of chemical-thermal treatment (CHT), has not found a wide distribution like cementation and nitriding, despite the fact that the hardness of diffusion layers is 2 times higher compared to cemented layers, and 3 times, compared to with nitrided [1].

It is known that borated layers are characterized by extremely high brittleness [2], which significantly limits the range of processed products. Modern research is aimed at a fundamental change in the structure of borated layers. Instead of traditional compact boride layers, heterogeneous boron layers are obtained, consisting of a mixture of dispersed borides and an α-solid solution [3–9]. Studies are being carried out [4, 5] on modeling the kinetics and properties of borated layers.

Alloying elements have a significant effect on the kinetics, structure, and properties of borated layers. The element that is considered to have the greatest impact is carbon [1, 5]. While some scientists argue [1, 6, 10] that carbon slows down diffusion and reduces the rate of boron penetration, others [8, 9] note that carbon has little effect on the depth of borated

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layers. In studies [1-3, 6-9], a regularity is traced - carbon has a fundamental effect on the morphology of borated layers, which in turn determines the mechanical properties.

Other alloying elements (Cr, Si, Mo, V, etc.) have little effect on the layer morphology, but have a strong effect on the phase composition of the borated layers [11–13]. It was shown in [14–17] that the presence of chromium in steel in a significant amount makes it possible to obtain compact boride layers in which the chromium content is comparable to that in steel. In complexly alloyed steels, one can observe the precipitation of a dispersed strengthening phase both in steel and in compact boride layers [13, 18].

Some works [19, 20] point out the possibility of reducing the brittleness of surface diffusion layers by combined chemical-thermal treatment.

The investigation of the complex effect of alloying elements both on the morphology of borated layers and on the phase composition is an important component for predicting the mechanical properties of products subjected to boriding and solving an important problem - reducing the brittleness of boride layers, in particular. In this work, boride layers obtained on steels with different content of alloying elements, their microstructure, properties, morphology, chemical and phase composition are studied. In addition, composite layers combining high-temperature electrolysis-free liquid boriding and gas nitriding have been studied.

2 Methodology and materials of the experiment

Steels 16Ch3NVFMB (VKS-5), 40Ch13, and Ch12MF were chosen as the material for research, the chemical composition of which is given in Table 1.

Table 1. The chemical composition of the studied steels

Steel	Content of elements, % mass									
VKS–5	C	Si	Mn	Ni	Nb	W	V	Mo	Cr	Ce
	0,14–0,19	0,6–0,9	0,4–0,7	1,0–1,5	0,1-0,2	1,0–1,4	0,35–0,55	0,4–0,6	2,6-3,0	0,01–0,05
40Ch13	C	Si	Mn	Ni			S	P	Cr	
	0,35-0,44	under 0.6	under 0.6	under 0.6			under 0.025	under 0.03	12-14	
Ch12MF	C	Si	Mn	Ni	V	S	P	Mo	Cr	Cu
	1,45-1,65	0,1-0,4	0,15-0,45	under 0,35	0,15-0,3	under 0,03	under 0,03	0,4-0,6	11-12,5	under 0,3

The choice of steels was due to different carbon content, as well as different content of alloying elements. So the range of carbon was from 0.16% by mass. for steel VKS-5 up to 1.5% by mass. for steel Ch12MF. The total content of alloying elements (excluding carbon) ranged from 8% by mass. for steel VKS-5 up to 14% by mass. for steel 40Ch13.

Boriding was carried out by a liquid electrolysis-free method in a shaft resistance furnace at a temperature of 1000 °C in a melt based on sodium tetraborate (70% by mass) and boron carbide (30% by mass) with exposure from 2 to 8 hours.

Gas nitriding was carried out at a temperature of 550°C with an exposure of 30 h.

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

To identify the grain boundaries during the analysis of the microstructure of the microsection surface, etching with the Marble reagent was used [14].

The investigation of the distribution of microhardness over the depth of the diffusion and composite layers was carried out on transverse sections on an automatic microhardness tester Durascan-70 at a load of 100 g.

The investigation of the microstructure and depth of the diffusion layer was carried out on an Olympus optical microscope at various magnifications.

Scanning electron microscopy was carried out on a JSM-7500F scanning electron microscope with an energy dispersive attachment.

3 The results of experiments and discussion

The study of the microstructure and depth of the obtained borated layers made it possible to reveal the relationship between the holding time of the process and the depth of the resulting layers. All samples of the studied steels were processed in the melt at a temperature of 1000 ± 10 ˚C for 2, 4, 6, 8 hours. The graph of the dependence of the layer depth on the holding time is shown in Figure 1.

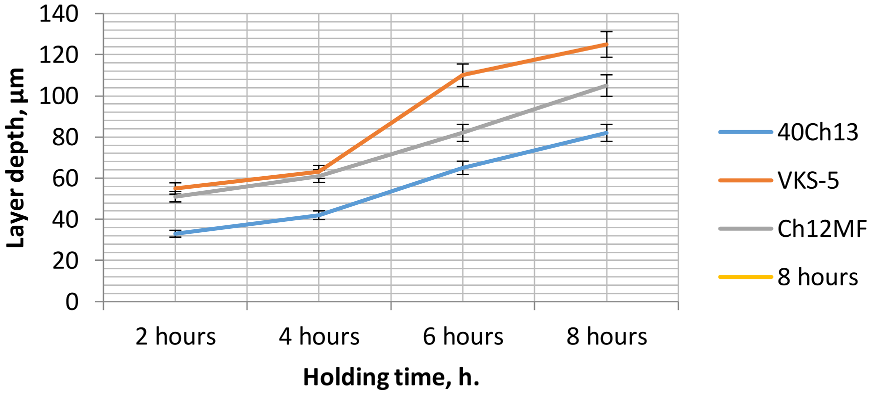

Fig. 1. Graph of the dependence of the layer depth on the holding time for the studied steels

It has been established that an increase in exposure time leads to an increase in the layer depth. The preferred holding time is 6-8 hours, since this holding time results in an optimum layer depth. Moreover, an increase in the holding time of more than 6 hours reduces the growth rate of boride layers, which is associated with the difficulty of further diffusion of boron deep into the steel. Thus, at the initial stages of the process, boron diffuses both over microstructure defects and over the grain volume, but when the FeB and Fe_2B phases form on the surface of boride needles, diffusion begins to pass along the boundaries of the needles, which greatly reduces the specific area of the diffusion flux. The accumulation of carbon in the transition layer has an effect, which also hinders the diffusion of boron.

The study of the microstructure of steel VKS-5 showed that the borated layers have a mildly pronounced needle-like structure. The microstructure of the borated layer obtained on VKS-5 steel with an exposure of 8 hours is shown in Figure 2.

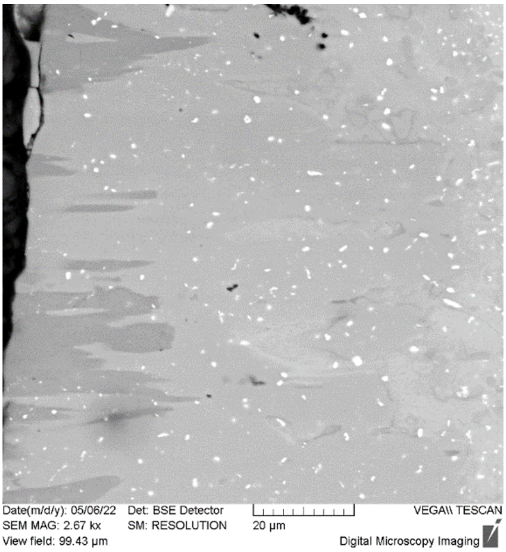

Fig. 2. Microstructure of the borated layer obtained on steel VKS-5

The structure is represented by two boride phases, the dark layer corresponds to the FeB phase, and the lighter layer corresponds to the Fe_2B phase. The presence of light particles of different sizes was revealed, presumably these are carbide, boride, carboboride phases of alloying elements.

The study of the microstructure of steel 40Ch13 showed that the borated layers have an atypical structure. The microstructure of the borated layer obtained on steel 40Ch13 with an exposure of 8 hours is shown in Figure 3.

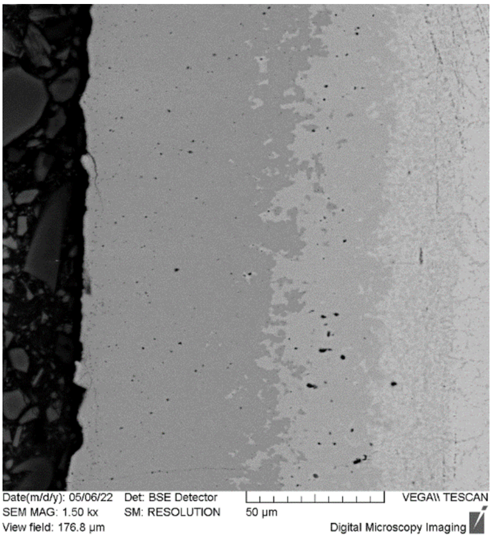

Fig. 3. Microstructure of the borated layer obtained on steel 40Ch13

The structure is also represented by two boride phases. Despite the large amount of chromium in the structure, no strengthening particles were detected, possibly due to the fact that chromium was dissolved in boride phases. It should be noted that the dark phase of FeB is dominant, which occupies about 70% of the volume of the boride layer.

The study of the microstructure of Ch12MF steel showed that the borated layers have a "hieroglyph" type structure. The microstructure of the borated layer obtained on Ch12MF steel with an exposure of 8 hours is shown in Figure 4.

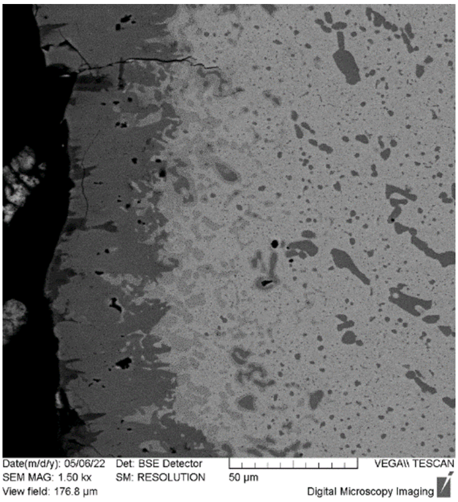

Fig. 4. Microstructure of the borated layer obtained on Ch12MF steel

The structure is represented by two boride phases, however, they differ in color from the previously obtained structures. Small inclusions of the FeB phase are fixed on the surface; the basis of the boride layer is the Fe_2B phase. The transition layer in this case is not continuous, it is interrupted in some places and is observed at a considerable distance from the boride layer. In some places, the transition layer envelops dark large hardening particles.

The conducted studies of the microhardness distribution over the cross section of the samples of the studied steels are presented in Figure 5 in the form of a histogram.

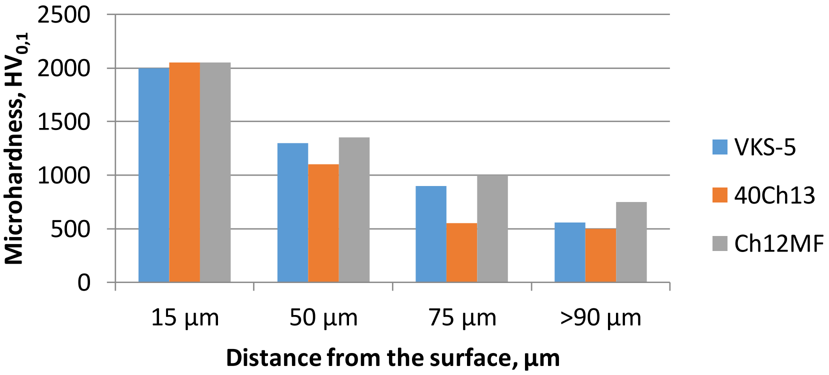

Fig. 5. Histogram of microhardness distribution over the layer depth investigated steels

An analysis of the distribution of microhardness over the cross section showed that the surface microhardness of all the studied steels is practically the same. With distance from the surface, the microhardness for different steels differ. Thus, the microhardness for Ch12MF steel is maximum at different distances from the surface, the microhardness of VKS-5 steel on boride layers is slightly inferior, significant differences are visible at a greater distance from the surface, where the microhardness of the matrix is measured. The microhardness of steel 40Ch13 is significantly inferior to other steels, especially in boride layers, which is obviously due to the alloying of boride layers with chromium. From the point of view of reducing the brittleness of the boride layers, microhardness distributions for VKS-5 and Ch12MF steels are preferable, which make it possible to reduce sharp drops in microhardness values over the sample cross section.

The study of the microstructure of composite layers after combined chemical-thermal treatment, which combines boriding and subsequent gas nitriding, suggests a decrease in microbrittleness and an increase in the corrosion resistance of the coating [13]. An example of the microstructure of the composite layer is shown in Figure 6.

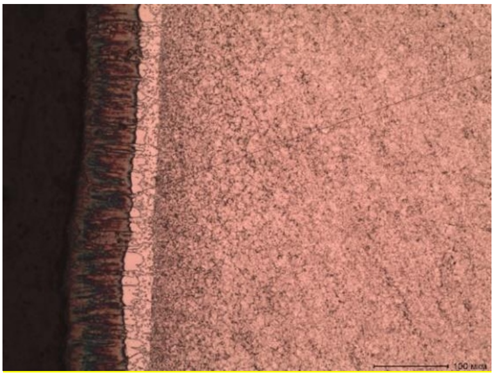

Fig. 6. Microstructure of the composite layer, x200

It has been established that after gas nitriding, a dark nitrogen-containing layer is superimposed over the borated layer, which penetrates to a depth of 90–95 µm.

The study of the distribution of microhardness over the surface of the section after combined chemical-thermal treatment, which combines boriding and gas nitriding, is shown in Figure 7.

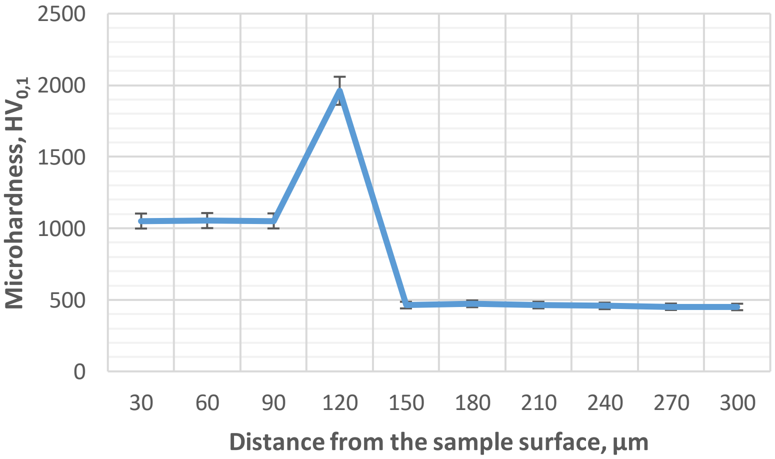

Fig. 7. Distribution of microhardness over the cross section of a sample of VKS-5 steel after combined chemical-thermal treatment

It has been established that the microhardness distribution curve after boriding and gas nitriding has a significant gradient of microhardness values in the transition from the nitrided layer to the boron layer and then to the core. Such a distribution of microhardness, based on previous studies [13], suggests a decrease in microbrittleness and an increase in wear resistance.

4 Conclusions

The study showed that with an increase in the amount of carbon in steel, the morphology of the borated layer changes. For example, for VKS-5 steel with a low carbon content, the boride layers have an acicular structure; for steel 40Kh13 with an average carbon content, the boride layers have an atypical continuous structure; steel Ch12MF is very different from the transition layers in other steels. The study of microhardness distributions showed that the surface microhardness on all steels is practically the same, and significant differences in microhardness begin with distance from the surface. An analysis of the microhardness distribution histogram showed that the most preferable microhardness distribution was observed for samples of VKS-5 and Ch12MF steels.

Combined chemical-thermal treatment, combining boriding and gas nitriding, makes it possible to obtain multilayer composite layers consisting of nitrided and boronized layers. The study of the microstructure and microhardness of the composite layer suggests a decrease in microbrittleness and an increase in corrosion resistance and wear resistance.

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