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The Influence of Thermal-Diffusion Saturation on the Properties of Boride Layers in AISI1095 Tool Steel

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This article presents a comprehensive analysis of the mechanical properties of boride diffusion coatings applied to AISI1095 steel. The study is conducted to enhance the wear resistance and durability of tools and machine parts. Using thermal-diffusion saturation, single-phase and two-phase boride layers are formed, for which detailed measurements of microhardness and microbrittleness are carried out. Tests are performed with the PMT-3 device under loads ranging from 0.4 N to 20 N, allowing the determination of the relationships between microhardness, brittleness, and load magnitude. Data analysis revealed three distinct regions of microhardness variation corresponding to different load levels and highlighted the behaviour of boride phases FeB and Fe_2B. Special attention is given to studying interphase boundaries, where maximum residual stresses, contributing to crack formation and coating degradation, are observed. The obtained results enable more accurate predictions of the operational characteristics of boride coatings and optimization of application regimes that is particularly important for components subjected to critical loads in industries such as heavy industry and mechanical engineering. This research contributes to the development of protective coating technologies aimed at improving the wear resistance and durability of products.

Key words: boride coatings, diffusion method, microhardness, microbrittleness, steel AISI1095, wear resistance, industrial applications.

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У цій статті представлено всебічну аналізу механічних властивостей боридних дифузійних покриттів, нанесених на крицю AISI1095. Дослідження проводилося з метою підвищення зносостійкости та довговічности інструментів і деталів машин. Методом термодифузійного насичення було сформовано однофазні та двофазні боридні шари, для яких було проведено детальні міряння мікротвердости та мікрокрихкости. З використанням приладу ПМТ-3 було проведено випробування під навантаженням у діяпазоні від 0,4 Н до 20 Н, що уможливило визначити залежності мікротвердости та крихкости від величини навантаження. Аналіза даних виявила три характерні області зміни мікротвердости, що відповідають різним рівням навантаження, і підкреслила особливості поведінки боридних фаз FeB і Fe_2B. Особливу увагу було приділено дослідженню міжфазних меж, де спостерігалися максимальні залишкові напруги, що сприяють утворенню тріщин і деґрадації покриття. Одержані результати дали змогу більш точно прогнозувати експлуатаційні характеристики боридних покриттів та оптимізувати режими нанесення їх, що особливо важливо для деталів, які піддаються критичним навантаженням у таких галузях, як важка промисловість і машинобудування. Це дослідження вносить внесок у розвиток технологій нанесення захисних покриттів, спрямованих на поліпшення зносостійкости та довговічности виробів.

Ключові слова: боридні покриття, дифузійний метод, мікротвердість, мікрокрихкість, криця AISI1095, зносостійкість, промислові застосування.

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1. INTRODUCTION

One of the most effective methods for increasing the wear resistance and durability of tools, machine parts, as well as technological and instrumental equipment, is the application of boride diffusion coatings on the surface of products. These coatings provide high hardness and resistance to mechanical and chemical effects, which significantly enhance the performance characteristics of products. For a qualitative and quantitative assessment of boride coatings, it is crucial to study their mechanical properties in detail, especially such parameters as microhardness and microbrittleness [1, 2].

In the present study, an analysis of the microhardness and microbrittleness of boride layers formed by the thermal-diffusion saturation method was conducted. The research established a clear relationship between microhardness, microbrittleness, and the load applied during testing. This allows conclusions to be drawn about the behaviour of coatings under various operating conditions.

The results of this study have important practical significance. They allow for more accurate predictions of the performance characteristics of boride coatings and for the selection of optimal application modes for various operating conditions. The obtained data can also be used to develop new surface treatment technologies aimed at improving the wear resistance and durability of products. In particular, the established dependencies can help in creating coatings with specified properties, which is especially important for critically loaded units and parts used in heavy industry, mechanical engineering, and other fields.

Thus, the studies of the microhardness of boride layers conducted in this work make a significant contribution to the development of protective coating technologies, which contributes to the increased reliability and durability of tools and equipment.

2. EXPERIMENTAL/THEORETICAL DETAILS

The research was conducted on AISI1095 steel coated with boride layers. The boriding process was carried out in powder media inside special containers with a melting lock at a temperature of 950°C for 4 hours. As a result of this process, single-phase and two-phase boride layers were formed on the samples. The thickness of the single-phase layer consisting of Fe_2B was about 100 μm. The two-phase layer had a total thickness of up to 200 μm, with the thickness of the FeB surface zone being 100 μm.

To measure the microhardness of the coatings, a PMT-3 device was used, where tests were conducted with loads on the indenter ranging from 0.4 N to 2.0 N. Indentation tests were performed according to two schemes: the first scheme included measurements along the surface of the sample along the axis of the boride needles, and the second scheme provided for measurements along the layer on cross-sections at a distance of 35 μm from the surface of the sample [3–5].

The cross-sections for determining microhardness were prepared according to standard methodology. This process included rough grinding, then fine grinding on abrasive paper, and polishing on cloth using chromium oxide. During the microhardness and microbrittleness measurements, 50 indentations were performed at each load.

These meticulous measurements and tests provided detailed data on the mechanical properties of boride coatings, which is important for assessing their performance characteristics and further use in various industrial fields [6].

The study of microbrittleness was conducted according to the methodology described in work [2]. To assess microbrittleness, the number of indentations with defects and the nature of these defects appearing around the indentation were analysed. The total brittleness score was calculated using the following formula:

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

where Z is the total brittleness score; n_1, n_2, n_3, n_4, n_4 are the numbers of indentations with the corresponding brittleness score.

During the study, careful statistical processing of the obtained results was carried out. Methods of statistical analysis were used to eliminate possible errors, including gross errors. All measurements were analysed to identify and eliminate anomalous data, ensuring high accuracy and reliability of the final results.

The methodology included the following stages:
– preparation of samples and application of indentations at various loads on the indenter;
– assessment of the nature of defects around each indentation using optical microscopy;
– counting the number of indentations with different levels of brittleness;
– calculation of the total brittleness score according to the specified formula;
– statistical processing of data, including the exclusion of anomalous values and calculation of average indicators.

3. RESULTS AND DISCUSSION

These studies provided objective data on the microbrittleness of boride coatings, which is important for further improving their characteristics and developing new coating application technologies [7]. This approach also allows for accurate prediction of material behaviour under operating conditions, which is a key factor for enhancing their durability and reliability.

The results of the surface microhardness measurements according to the first scheme for the single-phase and two-phase boride layers are presented in Table 1.

Measurements of the surface microhardness of the single-phase and two-phase boride layers showed that the surface microhardness changes depending on the load applied to the indenter (Fig. 1).

Figure 1 shows that three characteristic sections were identified in the microhardness measurements. The first section corresponds to a load of less than 0.5 N. In this section, there is a sharp decrease in microhardness when the load reaches 0.5 N. The second section lies in the range of 0.5–1 N, where a sharp increase in microhardness is observed (from 13900 MPa to 16100 MPa for FeB and from 9400 MPa to 15700 MPa for Fe_2B, respectively). The third section is characterized by a gradual increase in microhardness values for FeB and Fe_2B, respectively.

Additionally, the study included measurements of the microbrittleness of the FeB and Fe_2B phases by indenting along the layer on cross-sections.

TABLE 1. Surface microhardness of FeB and Fe_2B layers.

Load, N	FeB	Fe_2B
	Surface microhardness, MPa	
0.4	14800	9900
0.5	13900	9400
1.0	16100	15700
1.5	16300	16500
2.0	16500	17200
2.5	16600	17300
3.0	17100	17400

Changes in the microhardness of the boride layer on cross-sections depending on the load can be divided into three sections (Fig. 2). In the first section, with small loads (less than 0.5 N), there is an increase in microhardness values as the load decreases. This is because, with smaller loads, the indentation has fewer defects, and the microhardness is measured more accurately. In this section, the total brittleness score is the lowest: 36 for the FeB phase and 16 for the Fe_2B phase. The

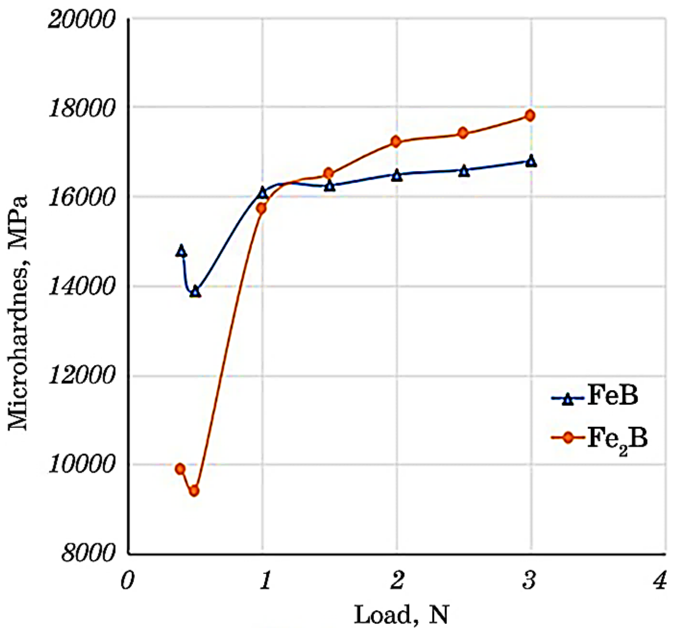

Fig. 1. Change in microhardness of boride layers depending on load.

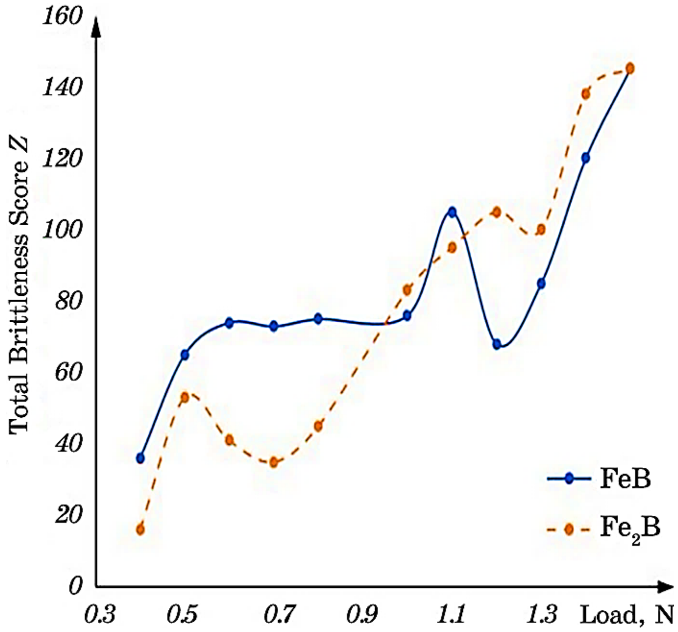

Fig. 2. Change in total brittleness score of boride layers depending on load.

number of indentations with defects is 19 and 12 for FeB and Fe_2B, respectively. These defects correspond to a brittleness score of 1–3 and are characterized by one or two cracks at the indentation corners.

At this stage of measurements, the root mean square error reaches its maximum values: 3700 MPa for the FeB phase and 4300 MPa for the Fe_2B phase. This is explained by the fact that, with small loads, the accuracy of measurements decreases due to the influence of microstructural heterogeneities and material defects. As a result, the microhardness measurement data can vary significantly, leading to increased error [6].

In the second section, with a load ranging from 0.5 N to 1.0 N, there is a further increase in microhardness values. During this period, the total brittleness score increases 2–3 times compared to the first section, indicating an increase in the materials' brittleness. At the same time, there is a decrease in the relative measurement error, indicating an improvement in the accuracy of the results (Table 2). The number of indentations with defects in this section is about 30, and they correspond to a brittleness score of 1–4. This means that the defects include both minimal cracks and more severe damage.

The third section is characterized by loads greater than 1 N. In this section, there is a further increase in the materials' microbrittleness. The number of indentations with defects approaches 40, indicating a

TABLE 2. Change in microhardness and brittleness of boride layers.

Load, N	FeB		Fe_2B	
	Microhardness, MPa	Total brittleness score Z	Microhardness, MPa	Total brittleness score Z
0.4	1310	36	1230	16
0.5	1250	65	1160	53
0.6	1300	74	1300	41
0.7	1340	73	1330	35
0.8	1390	75	1600	45
1.0	1490	76	1620	83
1.1	1540	105	1530	95
1.2	1600	68	1500	105
1.3	1720	85	1430	100
1.4	1530	120	1450	138
1.5	1440	145	1500	145

significant deterioration of the materials' condition under high loads. At a load of 1.5 N, the total brittleness score doubles compared to the second section. This significant increase indicates a sharp deterioration in the materials' brittleness. The number of indentations with defects reaches 40, among which there are indentations corresponding to a brittleness score of 5. This means that the indentation shape is destroyed, which is a serious indicator of the materials' property degradation.

More than half of the indentations in this section have a brittleness score of 4, characterized by one or two chips at the edges of the indentation. These chips indicate significant damage to the material structure under the load. As a result, the microhardness values in this section decrease to 14.400 and 14.200 MPa for the FeB and Fe_2B phases, respectively. This decrease indicates material degradation under high loads, which must be considered when using it in real operating conditions [8].

The analysis of the research results indicates that the destruction of the diffusion layer most often occurs along the interphase boundary between the high-boron phase FeB (with rhombic symmetry) and the low-boron phase Fe_2B (with tetragonal symmetry). In this zone, the maximum residual stresses are observed, which contribute to crack formation [9]. This occurs particularly due to the anisotropy of the thermal expansion coefficients of the FeB and Fe_2B borides. Within the interneedle space in AISI1095 steel, regions with a pearlitic structure are preserved, and the amount of borocementite is insignificant.

Fig. 3. Microstructure of boronised layer of AISI1095 steel, ×200.

The microstructure of boronised AISI1095 steel is shown in Fig. 3.

This fact underscores the importance of understanding the mechanisms of diffusion layer destruction and the need to consider anisotropy and other physical properties of materials when designing and operating products with such coatings [10].

4. CONCLUSIONS

As a result of the study, it was established that the microhardness of the boride layer directly depends on the load applied by the indenter. Changes in the surface microhardness of the FeB and Fe_2B boride layers showed similar trends with changing loads. These dependences can be divided into three main sections.

1. At low loads (less than 0.5 N), an initial increase in microhardness is observed. This may be due to the materials' response to small mechanical loads and initial changes in its structure.
2. In the range of 0.5 N to 1.0 N, there is a significant increase in microhardness. This is associated with deeper penetration of the indenter and possibly phase transformations in the boride phases.
3. In the load range of 1 N to 2 N, microhardness remains almost unchanged. This may indicate material saturation with deformation or

reaching its strength limit.

In addition, the study revealed the anisotropy of the microbrittleness of the FeB and Fe_2B boride phases, which is confirmed by the total brittleness score along the axis of their needles. These data not only deepen our understanding of the mechanical properties of boride layers but also highlight the importance of considering the directionality of tests when assessing their industrial and technical applications.

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