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Mechanical Characterization and Micro-Wear of FeB-Fe_2B Layers on Boriding AISI D2 and AISI 4340 Steels

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Abstract

The mechanical behavior and wear of the different hardened phases with bore-induced changes in AISI 4340 and AISI D2 steels were investigated. The hardness and modulus of elasticity were measured by nanoindentation and the values obtained for the layers in AISI D2 steel were 18 GPa and 325 GPa in the Fe_2B boride phase, and 20 GPa and 360 GPa in the FeB boride phase, respectively. The AISI 4340 steel presented mainly the Fe_2B phase. It was then possible to analyze the coefficient of friction obtained in the Fe_2B phase of the steel AISI 4340 presented a range of 0.04 to 0.06. The AISI D2 steel presents two different phases in the boride layer being the coefficient of friction higher for the test in the FeB phase than for Fe_2B, and the values vary from 0.065 to 0.075. These parameters were obtained with micro-wear tests. No adhesion failures were observed after the sliding tests in the interface of the two different boride layers. Cracks in the FeB phase after the sliding test were much more frequent.

Keywords

Iron Boride, Micro-Wear Test, Mechanical Properties, Boriding

1. Introduction

The thermochemical process known as boriding is a good alternative for the improvement of surface properties due to the increase of hardness and decrease of wear in applications involving contact with other materials. Because of their relatively small size and high mobility, boron atoms diffuse in a variety of materials such as steel, cast iron, nonferrous metals such as nickel and cobalt alloys, refractory alloys, titanium alloys, molybdenum and cemented carbides [1]. The boriding process is widely used in applied machine elements where friction control and wear are important factors. The boriding produces a superficial layer of high hardness, high resistance to wear and low coefficient of friction. The boriding treatment can occur in solid, liquid, gas, fluid bed or plasma [2] media. The treatment is generally made at temperatures between 700˚C and 1050˚C [3].

The boron atoms in a ferrous substrate tend to dissolve interstitially, reacting with the iron to form the FeB and Fe_2B compounds. The boron layer may be formed of a single phase Fe_2B or a double phase FeB + Fe_2B [4]. In tribological applications, a boron layer composed of the Fe_2B phase is the most desirable, since the FeB phase, which is richer in boron, is more fragile, leading to the easy propagation of cracks in the layer. The strong covalent bond of most borides is responsible for their high values of melting point, elastic modulus and hardness [3]. The properties that FeB and Fe_2B boride phases are distinct, with relevance for the elastic modulus, hardness, and thermal expansion, so crack formation at the interface between these two phases can be possible. The borides in general have a high free negative energy for their formation, which presents excellent chemical and thermal stability [2]. However, the behavior of FeB/Fe_2B interface under wear test was not well known.

The commonly used tribological tests are the pin on disk and the reciprocal sliding [5], which use forces applied normally high and the contact with the piece is made by spheres with diameters of the order of millimeters [5] [6]. In these tests performed against a boriding surface, the dissipated energy is related to plastic deformation, adhesion processes and the creation of third body particles that influence the contact and considerably affecting the friction and the wear process. However, these tests do not permit to evaluate the difference in behavior between the FeB and the Fe_2B phase layers. They are present normally in a saw tooth geometry, grown from the surface. The behavior of the individual phases individually under wear test is difficult to infer from tribological tests with contact from top surface. In order to investigate the behavior of micro-wear in each phase, it is necessary to perform tests in the cross section piece in the boriding steel near the surface region. In these processes, the friction and the wear can be investigated by the contact of the tribological body against a single phase in the modified layer or in an analysis of a succession of phases in the wear groove. The results can then be analyzed considering only the behavior of the specific phase and not from a mixture of phases where the stress distribution is not clearly defined. The major problem to perform this tests the low thickness of the modified layer, of about 100 μm or less, being necessary to use spheres of small radius, with forces of the order of millinewtons, with the ability to control the movement in the range of micrometers and to measure lateral forces of small value [7].

The objective of this work was to evaluate the wear and the formation of cracks in the different phases formed as a function of the depth in the boride region after cross sections of AISI D2 and AISI 4340 steels submitted to boriding by thermochemical treatment in a salt bath. The surface regions of cross-sectional samples were subjected to reciprocal slip micro-wear tests with diamond spherical tip (r = 10 μm). The coefficient of friction and the aspect of the wear grooves were analyzed according to the composition of the measurement region, with emphasis on the formation of cracks in the different regions of the boride layer, as well as the interfaces between them. The wear analysis of FeB, Fe_2B, substrate, and the interfaces could be performed separately in this new way to investigate the treated surface. The mechanical properties, wear, friction coefficient, and crack formation of specific regions and in interfaces between the boride layers were investigated considering the composition of each region below the surface after boriding process in two different steels.

2. Materials and Methods

2.1. Materials and Treatment

Two types of steel were analyzed in this study: AISI D2 and AISI 4340 that present different Cr and C contents. The chemical composition of the steels was obtained by optical emission spectrometry according to **Table 1**. These steels were chosen because the formation of FeB and/or Fe_2B borides is dependent on the presence of Cr and C in the steel composition. Thus, for the chosen steels, the formation of FeB and Fe_2B borides occurs in the AISI D2 steel, whereas for the AISI 4340 steel the FeB formation occurred in a minimal amount, while the Fe_2B phase was the major one.

The boriding treatment was carried out in a bath containing borax ($Na_2B_4O_7 \cdot 10H_2O$) and boron carbide (B_4C), with the following composition 90% wt $Na_2B_4O_7 \cdot 10H_2O$ + 10% wt B_4C. Borax is a carrier for dissolved forming elements and also a boron supplier, being an active agent in the process. The hardened layer is formed by combining the iron present in the substrate with the boron present due to the chemical reduction of the borax by the B_4C. For the test, samples with dimensions of 10 × 10 × 10 mm were used, and these were prepared through sanding strips up to 600 meshes, followed by polishing with 1 μm diamond paste. An ultrasonic cleaning was performed with the samples immersed in ethyl alcohol. The boriding treatment was carried out in an electric resistance furnace. The treatment temperature was 1000˚C for 4 h. Immediately after being withdrawn from the boriding bath the samples were cooled in oil. The samples were cut, polished and attacked with nital reagent. The boride layers were analyzed by optical and scanning electron microscopy. The thicknesses of the modified layers were measured by optical microscopy and the values

Table 1. Chemical compositions of AISI D2 and AISI 4340 steels.

Steel	C	Cr	Ni	Mo	V	Si	Mn	Fe
AISI D2	1.48	11.91	-------	0.98	0.76	0.96	0.45	Bal.
AISI 4340	0.39	0.76	1.74	0.26	------	0.15	0.73	Bal.

determined by the average of five measurements. The determination of the crystal structure was performed by X-ray diffraction using Bragg-Brentano diffractometry in a Shimadzu XRD7000 apparatus, with 2θ ranging from 20˚ to 100˚ using CuKα radiation.

2.2. Hardness and Elastic Modulus Determination

The microhardness and elastic modulus of the different regions of the boride layer were measured using an instrumented indentation technique with applied loads up to 100 mN. In the region under evaluation indentations were performed in a matrix with about 80 indentations separated by distances of 30 µm, in a manner that indentations were performed in all phases, FeB, Fe_2B, and also in the transition region in the matrix, depending on the distance from the surface in the cross section of the boron modified layer. The equipment used was a Zwick-Roell ZNH nanoindenter. The method used was the QCSM that allows obtaining the hardness and the modulus as a function of the penetration of the tip during the loading. A diamond tip with Berkovich geometry was used. From the images of the region after the indentations, the values of hardness and modulus in each phase were defined from each indentation.

2.3. Micro-Wear Tests

In order to perform the micro-wear tests, a Zwick-Roell ZNH instrumented indentation equipment was used, which has an independent system for lateral force application and lateral displacement measurement, keeping the normal force applied constant. In this equipment, it is possible to control lateral force independently from normal force. The lateral displacement can also be defined independently. In the test the oscillation movement had amplitude of 30 µm resulting in a wear displacement with a total displacement of 60 µm in each cycle, resulting in grooves of about 80 µm. Fifty cycles of sliding movement were performed by applying a lateral force, corresponding to a total distance of 50 × 120 µm, or 6000 µm. The counter body used was a diamond sphere with a nominal radius of 10 µm. During the wear test, lateral force, normal force, tip penetration and lateral position data were recorded continuously. The normal force applied during the slip micro-wear test was 200 mN. The width of the grooves was measured using optical microscopy. In addition, the final aspect of wear grooves was examined using a scanning electron microscope. The applied lateral force ranged from 8 mN to 20 mN. This value was not predefined but was defined by the equipment being the necessary load to be applied to achieve a lateral sliding of the piece, while the normal force applied to the diamond ball was constant and equal to 200 mN. Figure 1 shows a schematic draw of the micro-wear process.

3. Results

3.1. Microstructural Characterization

Figure 2 shows the images by optical microscopy of the boron layer showing the

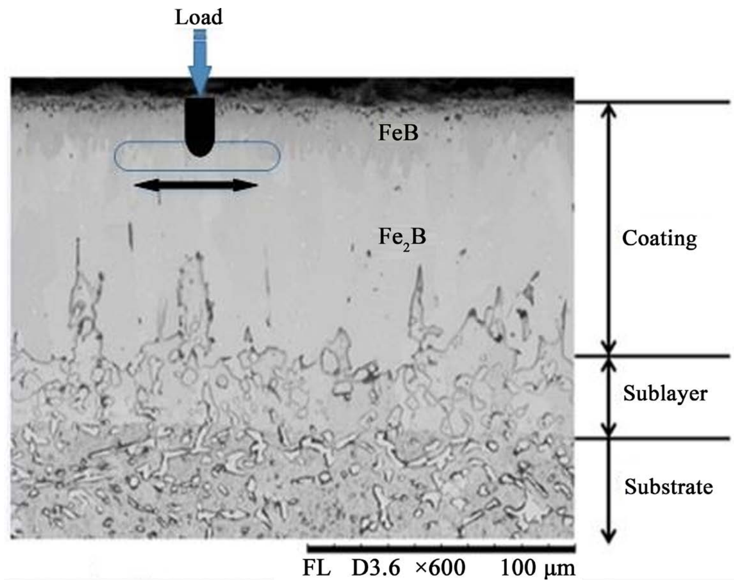
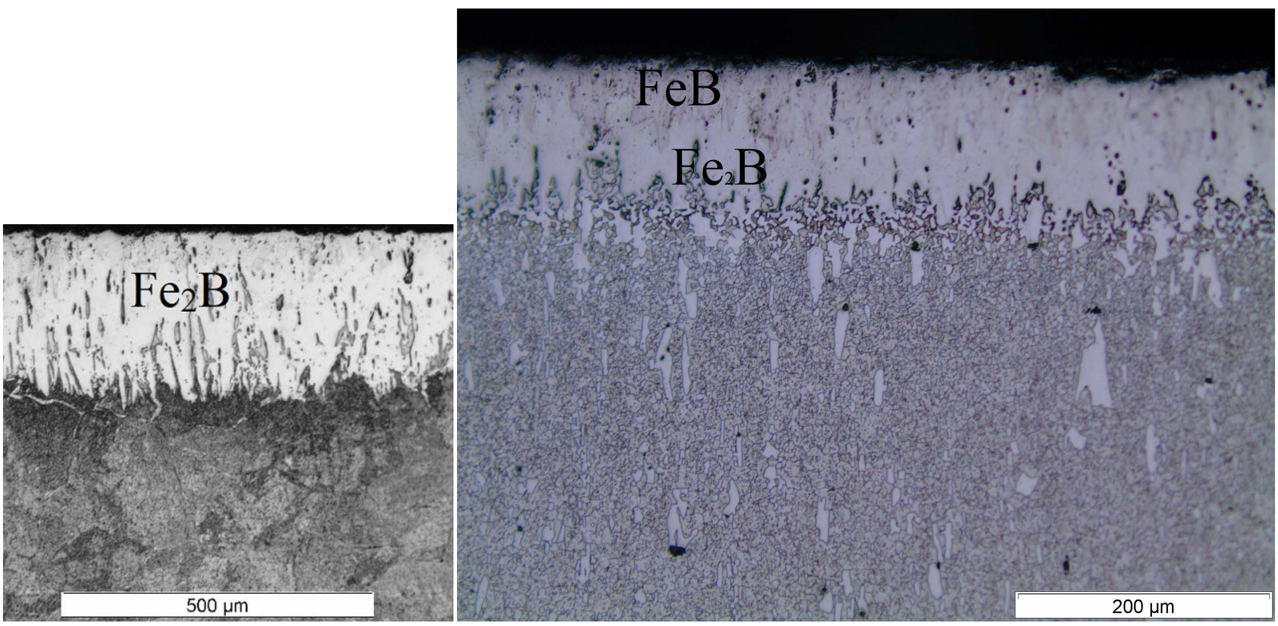


Figure 1. Illustrative schematic draw of the micro-wear test with reciprocal sliding in the different layers of the region modified by boriding.



(a) (b)

Figure 2. Bound steel. (a) AISI 4340; (b) AISI D2.

FeB and Fe_2B phases in the AISI D2 and Fe_2B phase in the AISI 4340 steels. The boride layer of the AISI 4340 steel formed on the substrate shown a saw-tooth type morphology. This type of morphology is due to a pronounced anisotropy of the boron diffusion coefficient in the tetragonal network of the Fe_2B [8]. It is possible to see for D2 steel a flat, compact, and porosity-free morphology. The presence of FeB and Fe_2B in the boride layer was confirmed by the X-ray diffraction analysis, as shown in **Figure 3**. The presence of FeB is more pronounced in AISI D2 steel, since it presents a greater amount of alloying elements and then the grown of this phase is favored.

The thickness of the boride layer Fe_2B on AISI 4340 steel was approximately 240 μm. In comparison, the FeB + Fe_2B layer on AISI D2 steel show a thickness of approximately 126 μm. The lower thickness of the AISI D2 steel layer is due to its chemical composition, which presents a greater amount of alloying element, considering that these alloying elements restrict the boron diffusion and retard the growth of the boride layer due to the formation of a diffusion barrier [2].

Figure 4 shows the hardness and modulus of elasticity of the Fe_2B phase obtained in the AISI 4340 steel. The hardness value was approximately 18 GPa and the elastic modulus approximately 325 GPa.

Figure 5 shows the hardness and the elastic modulus for the FeB and Fe_2B phases observed on AISI D2 steel. The hardness value of the Fe_2B phase was approximately 18 GPa, which was the same as for AISI 4340 steel. The FeB phase is

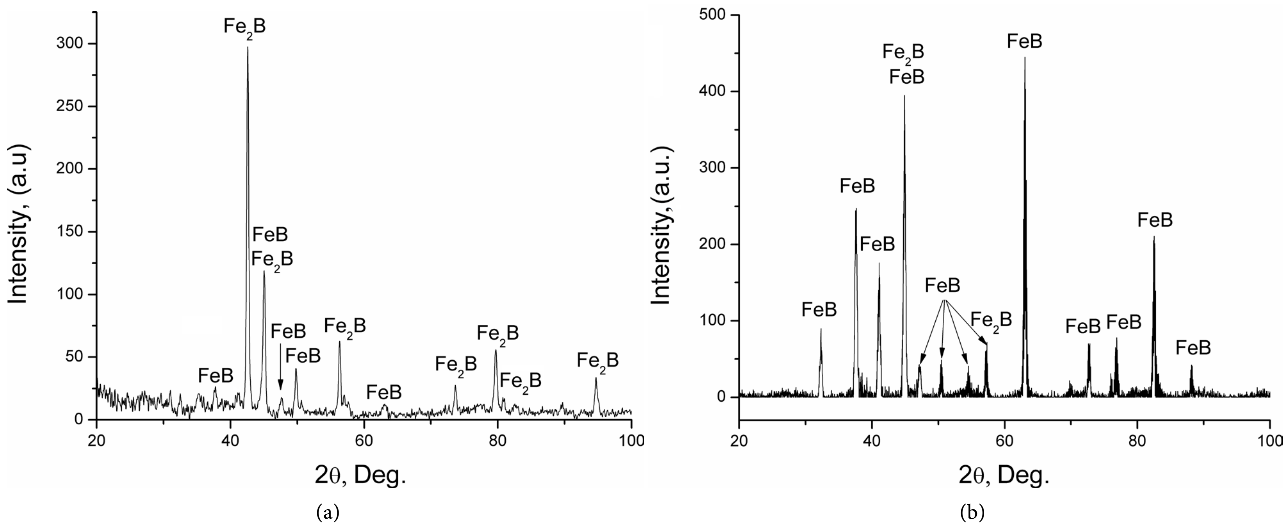


Figure 3. X-ray diffraction after boriding (a) AISI 4340 steel (b) AISI D2 steel.

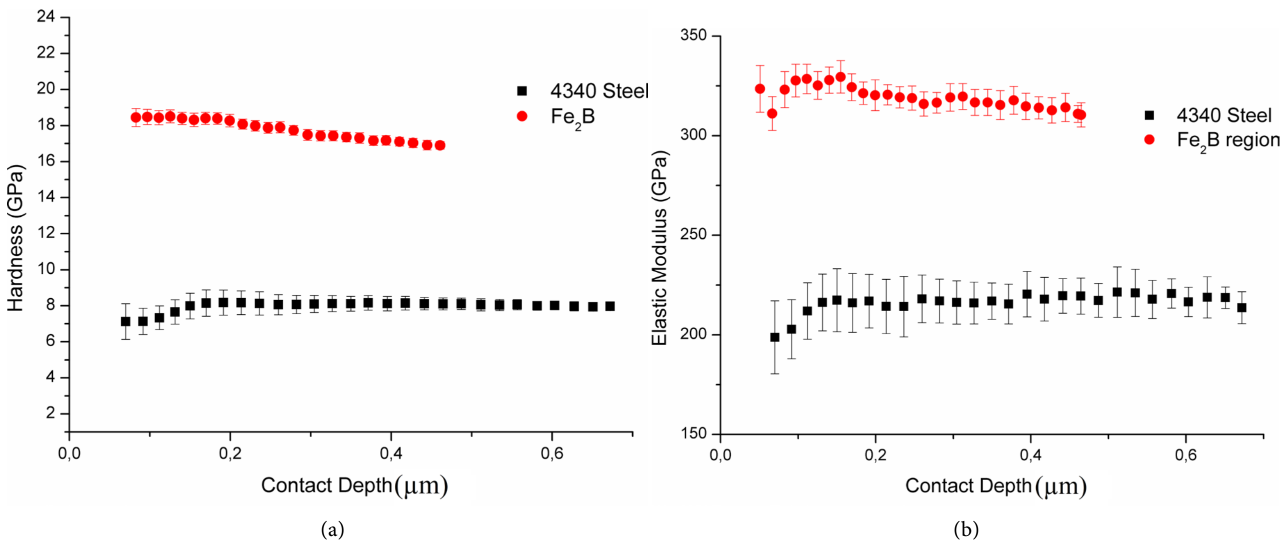


Figure 4. (a) Hardness and (b) elastic modulus of borides in AISI 4340 steel.

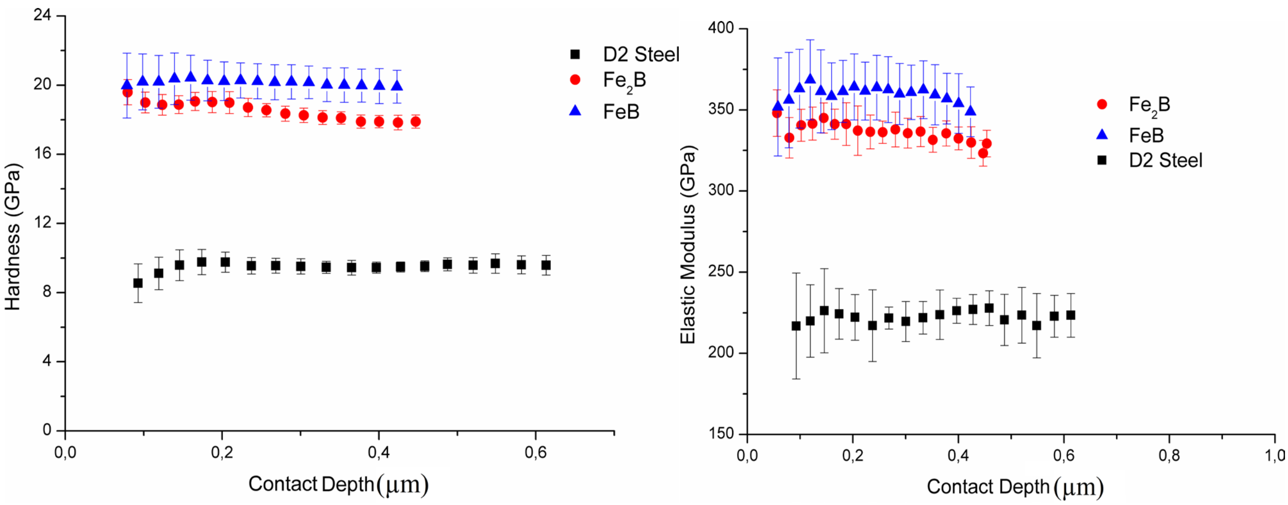
harder with a value of approximately 20 GPa. The elastic modulus for the FeB and Fe_2B phases were approximately 360 GPa and 325 GPa, respectively.

3.2. Micro-Wear Test

Figure 6 shows an image of grooves that were taken in the layer of the boriding-modified region for AISI D2 steel. The penetrator displacement was 60 µm on the surface of the coating in the FeB, Fe_2B, and in the sublayer and the substrate, as previously indicated in a schematic draw shown in Figure 1.

The wear test grooves performed on AISI 4340 steel are shown in Figure 7. The groove 1, correspond to a test performed just below the borate layer. Test 2 was performed on the substrate. Tests 3, 4, 5, 6 and 7 were performed on boron-modified layer.

Figure 8 shows the eight grooves for tests made on the modified region in cross section of the AISI D2 steel. Test 1 was performed in the FeB region while test 2 was made in a FeB phase region with a small portion of the Fe_2B phase. Tests 3 and 4 encompasses the two phases, with the highest portion being in the



(a) (b)

Figure 5. (a) Hardness and (b) elastic modulus of borides in AISI D2 steel.

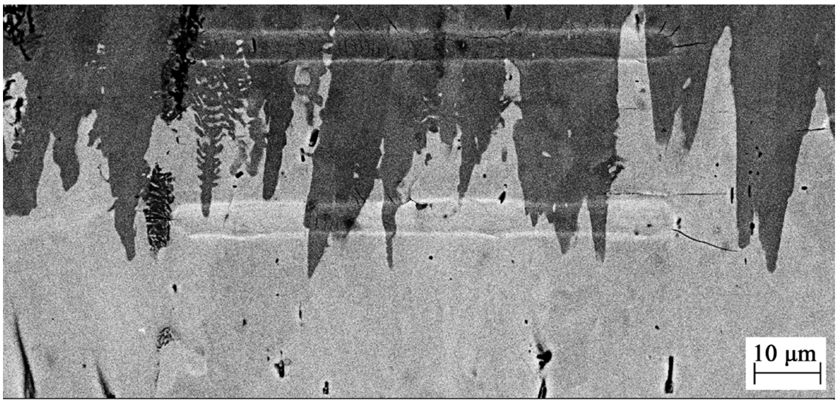


Figure 6. Micro-wear grooves in FeB and Fe_2B phases in AISI D2 steel.

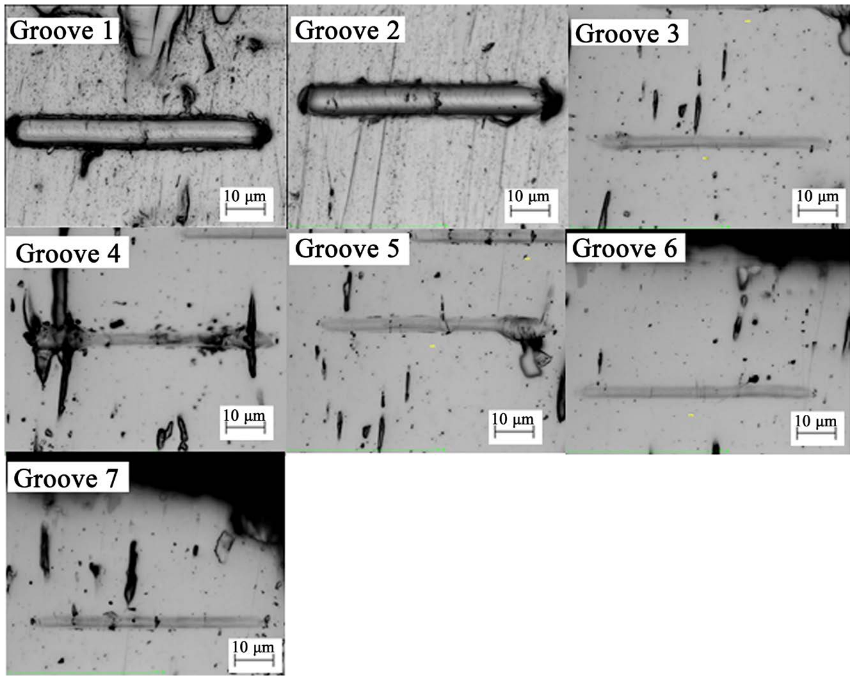


Figure 7. Morphology of grooves of micro-wear made on a cross section of boron modified layer of an AISI 4340 steel.

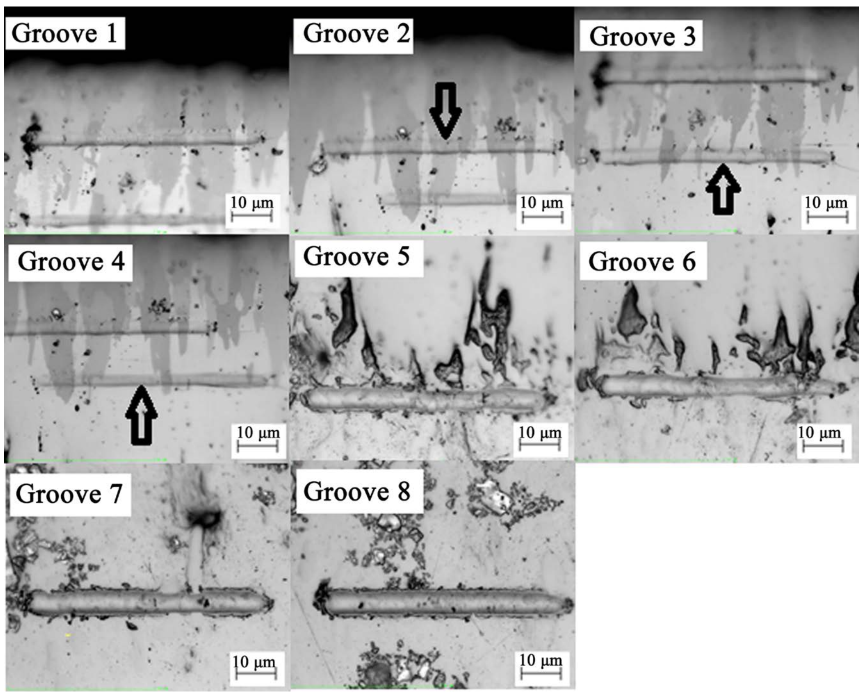


Figure 8. Morphology of grooves of micro-wear made on a cross section of boron modified AISI D2 steel.

Fe_2B phase and risk was practically on the Fe_2B phase. Tests 5 and 6 were made just below the boron modified layer. Finally the micro-wear tests 7 and 8 were performed on the substrate at approximately 50 μm below the modified region.

Figure 9 shows the graph of the coefficient of friction obtained in the AISI

4340 steel sample. The risks of 1-7 are shown in Figure 7.

The coefficient of friction in the steel AISI 4340 initially presented a higher value due to the initial surface where the penetrator slide since the groove is initially absent. After that, there is a tendency to stabilization in coefficient of friction. Table 2 presents the values of the coefficient of friction for test on cross section of a boron modified AISI 4340 steel.

Tests 1, 4, 6 and 7 presented a coefficient of friction in the range of 0.055 to 0.06. Curve for test 2, region of the substrate, shows a value of 0.05 relatively constant over the number of cycles. In grooves 3 and 5, performed in the Fe_2B hardened layer, the CoF values presented lower values, while for test 5 it showed a range of 0.055 to 0.045 curve 3 there was a decrease in this parameter that presented a range of 0.05 to 0.04.

The graph shown in Figure 10 shows the curves of the friction coefficients of

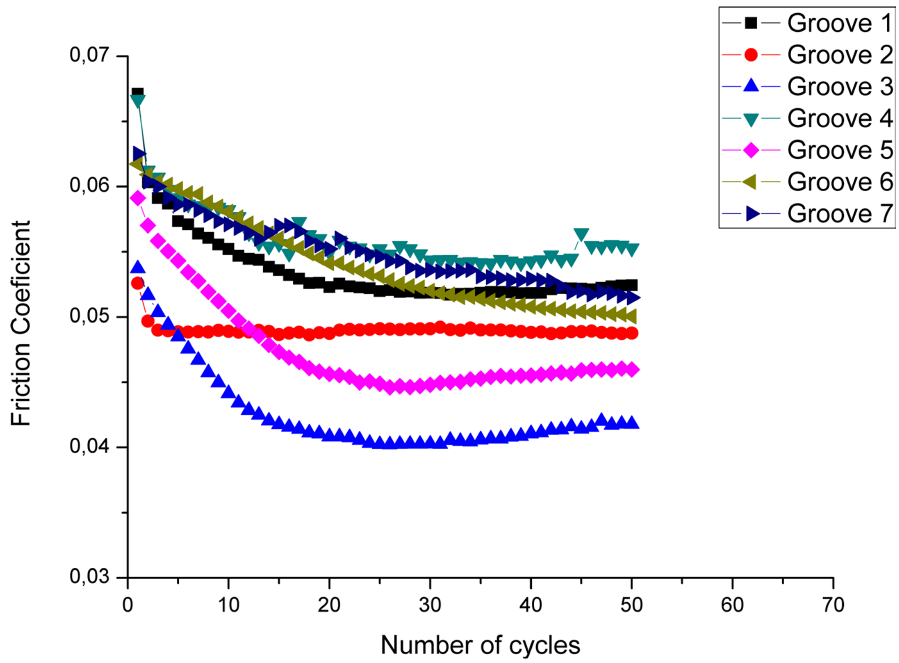


Figure 9. Coefficients of friction in function of cycle number for micro-wear tests performed on steel AISI 4340.

Table 2. Coefficient of friction of micro-wear on steel AISI 4340.

Test	Region	CoF
1	Below the boron layer	0.06
2	AISI 4340 Matriz	0.05
3	Fe_2B	0.05
4	Fe_2B	0.06
5	Fe_2B	0.05
6	Fe_2B	0.06
7	Fe_2B	0.06

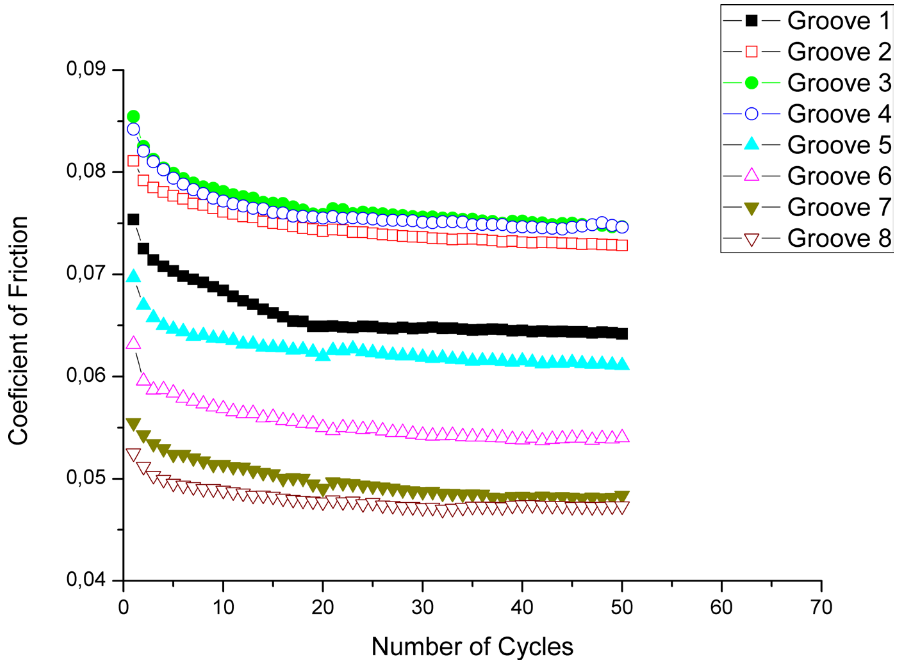


Figure 10. Coefficient of friction variation with the cycles in AISI D2 after boriding for the tests 1 - 8 made on different regions of modified layer.

Table 3. Coefficient of friction of micro-wear on steel AISI D2.

Test	Region	CoF
1	FeB	0.7
2	FeB e Fe_2B	0.08
3	Fe_2B e FeB	0.08
4	Fe_2B e FeB	0.08
5	Below the boron layer	0.07
6	Below the boron layer	0.06
7	AISI D2 Matriz	0.05
8	AISI D2 Matriz	0.05

AISI D2 steel. In this steel, because it did not present porosities the coefficient of friction presented a tendency of decrease depending on the region in which the risk was realized.

Table 3 shows the values for coefficient of friction relative a tests in different regions of the boron modified layer.

4. Discussion

The boriding process used in this work resulted in different characteristics depending on the substrate used. In the AISI 4340 steel, the predominant phase was Fe_2B, while in the AISI D2 steel it presented a double layer of borides, and on the external surface of the layer modified by boriding FeB was formed and, more internally, the formation of the Fe_2B phase. The formation of the FeB phase is favored in AISI D2 steel by the presence of alloying elements, mainly Cr, in high contents [8]. In the AISI 4340 steel, because it had a lower percentage of carbon and alloying elements, the hardened layer showed a serrated morphology, like saw-tooth, but without FeB formation. According to Selçuk *et al.* [8] this type of morphology is due to a pronounced anisotropy of the boron diffusion coefficient in the tetragonal network of the Fe_2B phase. This type of morphology tends to promote higher adhesion between the boride layer and the substrate [9]. The thickness of the layer modified by boriding in the AISI 4340 steel was approximately 240 μm, whereas in the AISI D2 steel the layer had a thickness of about 126 μm. The lower thickness of the layer on AISI D2 steel is due to a greater amount of alloying elements, as these restrict the diffusion of boron and the growth of the boride layer due to formation of a diffusion barrier [2]. The AISI 4340 steel presented a layer modified by boriding with high amount of porosity. This behavior is common in the iron boride layers formed on the surface of low carbon steel when compared to high alloy or high carbon steels [10] [11] [12].

The X-ray diffraction analysis showed that in the AISI 4340 steel the predominant phase was F_2B, whereas in the AISI D2 steel the structure of the layer modifies by boriding a double layer of iron borides characterized by FeB and Fe_2B phases. **Figure 5** shows the hardness of approximately 18 GPa and the an elastic modulus of 325 GPa of the Fe_2B boron obtained in the AISI 4340 steel. In the AISI D2 steel having the double borates layer the FeB hardness was approximately 20 GPa and the modulus of elasticity around 360 GPa while for the Fe_2B phase values of hardness and elastic modulus where 18 GPa and 335 GPa, respectively. For comparison, in the literature values of 590 GPa for the FeB module and about 300 GPa for Fe_2B [2]. The differences from literature are not clearly understood, but it could be related to porosity in Fe_2B phase for AISI4340 steel and FeB compounds that are not completely stoichiometric as indicated by X-ray diffraction for AISI D2 steel.

The coefficient of friction (CoF) was measured in the micro-wear tests against a diamond sphere of 10 μm radius and applied load of 200 mN. The stresses are much higher than the limit values for elastic deformation, resulting then in a large amount of plastic deformation. These CoF values were measured during the movement of the diamond tip in contact with the different regions on a cross section of each sample of the two steels used in this work. In the AISI 4340 steel, 7 tests were performed, while in the AISI D2 steel 8 tests were performed, as shown in **Figure 7** and **Figure 8**.

In the AISI 4340 steel, the micro-wear tests were carried out in the region where only the Fe_2B iron boride layer was present and in the innermost region corresponding to the matrix. CoF values ranged from 0.04 to 0.06. The lower CoF of 0.04 was observed for a region where the Fe_2B layer was more uniform without porosities. In this type of test the measured coefficient of friction was very low. It is related to the low adhesion due to the counter body was diamond, with a spherical geometry. Diamond is a material with high elastic modulus and high hardness. It can be seen in **Figure 7** for groove 3 that an insignificant number of cracks were no generated after the micro-wear test. In regions with pronounced porosity on the surface or just below the layer, an increase in CoF is observed.

In tests on the cross section of the modified layer on AISI D2 steel, the regions presenting a single FeB phase, a FeB/Fe_2B interface, and a transition region below the boron layer and in the matrix showed different coefficients of friction ranging from 0.048 to 0.078. The lower CoF was observed in the base matrix steel material, while the higher value was observed in the region alternately passing through FeB and Fe_2B dendrites. Wear trails in this interface region showed CoF of 0.078 and cracks were observed in the FeB phase. Cracks could be expected at the interfaces between the FeB/Fe_2B phases but no significant cracks were observed at these interfaces. The CoF found on the substrate of AISI D2 steel, groove 8 in **Figure 8**, presented the lowest value of all the regions in which the wear tests were performed. The CoF values of the substrates are similar between 0.04 and 0.055. CoF is mainly related to surface roughness, hardness, lubrication conditions and other surface effects, such as local chemical interactions at the interface of the pair in contact (iron boride and diamond tip) [13] [14].

The highest coefficients of friction are related to the presence of the FeB/Fe_2B interface. Possibly, there is a need for a greater lateral force to cause the sample to move against the diamond tip when the passage of one FeB rich region to another Fe_2B occurs and vice versa. Observing the image of the wear grooves, we can see the presence of cracks in FeB but not in the interfaces between the FeB (darker) and Fe_2B (brighter) phases in **Figure 11**.

In the AISI 4340 steel, which presented a great amount of porosity, the value of CoF in tests that reached these porosities presented higher values, as can be observed in **Table 2**. Carrera-Espinoza *et al.* [9] studied the tribological behavior of borides on an AISI 1018 steel and obtained in the plane slip wear test with a tribological Al_2O_3 sphere pair on the iron boride a coefficient of friction 0.088 in the slip condition with lubricant and in the slip condition without lubricant this value was 0.6. This is very diferent from obtained in this study, where values similar to lubricated tests were obtained for an unlubricated slide of diamond in the material. Saduman Sen *et al.* [15] studied the tribological properties using a pin-to-disk process. The iron boride layer formed on an AISI 4140 steel was subjected to oxidation and the coefficient of friction found was 0.50 to 0.60. However, this value decreased to 0.12 when the oxidation time was decreased. This indicate that the coefficient of friction is very dependent of the pair of materials in contact in a surface modified by a boriding process. The values of the coefficients of friction obtained in this study presented lower values than those found by Espinosa [9] and Saduman [15] due to two factors. In this work, the tribological pair used was boride against diamond, while in the mentioned works it was boride against alumina. In addition, the test used in this work was in the cross-section of the layer hardened differently from the mentioned works that

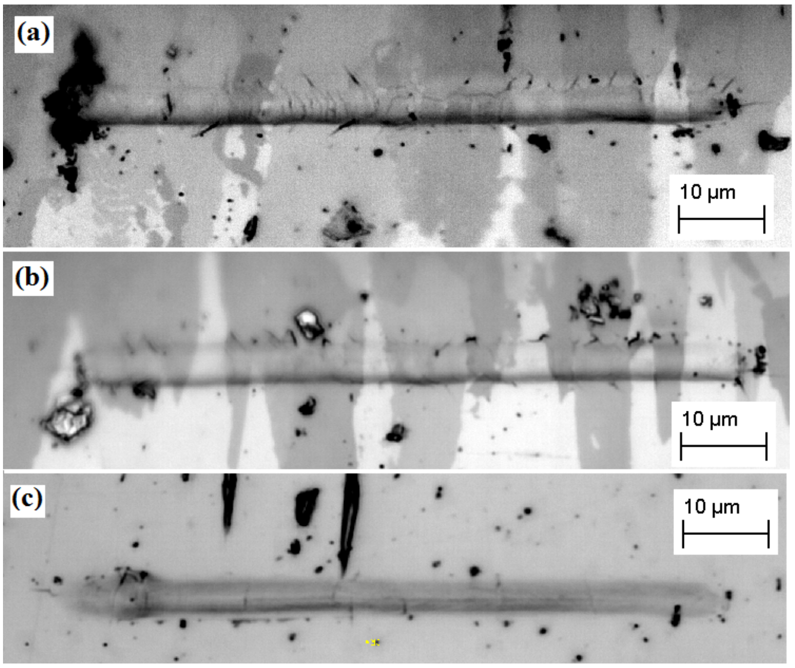


Figure 11. Cracks on grooves of micro-wear tests on (a) FeB phase (b) FeB and Fe_2B phase (c) Fe_2B phase.

the test was carried out at the top of the layer modified by boriding. The samples used in this work were polished with diamond paste, a fact that should have contributed to the lower values of the coefficients of friction.

5. Conclusions

The effects of boriding treatment on the microstructure, hardness and wear on AISI 4340 and D2 steels with a diamond spherical penetrator were investigated. The hardness and modulus of elasticity were measured by nanoindentation and the values obtained for the layers in the AISI D2 steel were 18 GPa and 325 GPa in the Fe_2B phase and 20 GPa and 360 GPa in the FeB phase.

The coefficient of friction obtained in the Fe_2B phase of the AISI 4340 steel presented a range of 0.04 to 0.06 while in the AISI D2 steel the coefficient of friction for the test in the FeB phase, values were between 0.065 to 0.075. No adhesion failures were observed between the diamond tip and the different boride layers and/or in the steel matrix.

Cracks formed in the different phases and regions of the layer modified by the boriding. Cracks in the FeB phase were much more frequent. However, no cracks were observed at the interfaces between FeB and Fe_2B phases during tests where the tip passes from one phase to another in AISI D2 steel. This indicates that despite the FeB be more brittle than Fe_2B the interface FeB-Fe_2B presents high levels of cohesion.

Ackowledgements

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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