

Investigation of surface properties and wear resistance of borided steels with different B₄C mixtures

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Boriding is a thermo-chemical surface hardening treatment applied to iron based materials. This treatment is performed by boron element diffusing into iron based materials at high temperatures. Wear resistance increases by forming ferro-boron phases on the material surface. In addition, a very hard layer forms on the material surface. As a result, the tribological properties of the material increase. In this study, 6 different mixtures are prepared with B₄C and SiC powders. In these mixtures steels of AISI 1020 and AISI 1040 are borided with a stainless steel crucible at a temperature of 900°C for 3 h. Depending on the boriding mixtures, the thickness of the borides formed on the surface of borided AISI 1020 and AISI 1040 steels ranged from 14 to 71 μm and from 10 to 64 μm, respectively. It is observed that the layer thickness increases with increase in B₄C% ratio. The wear rate of steels increased with decreasing SiC ratio. The average value of obtained microhardness for borides of AISI 1020 and AISI 1040 steels are 1776 and 1860 HV_{0.05}, respectively.

Keywords: AISI 1020, AISI 1040, B₄C, SiC, Wear, Fe₂B phase

Boriding refers to a surface diffusion treatment by which a boride coating is formed on the component surface. Formation of iron borides on steel surfaces is a well-known example of boriding¹. The boriding process improves hardness, fatigue, corrosion, and wear properties of surface. Industrial boriding processes can be applied to a wide range of steel alloys including carbon steel, low alloy-steel, tool-steel and stainless-steel^{2,3}. Typically, boriding is carried out in the 700-1000°C temperature range by using solid, liquid or gaseous boron-rich atmospheres⁴. The most frequently used method is pack boriding, a process similar to pack carburizing⁵. Often the pack contains a source of boron, usually boron carbide (B₄C) or amorphous boron, an activator to deposit atomic boron on the workpiece and a diluent. Pack boriding involves placing the component in the powder mixture and sealing it in a container. The container is then heated up to the required temperature for the required time and cooled in air^{5,6}.

Boride layers have typical high hardness values in the range of 1600-2100 HV_{0.1}. The hardness achieved by boriding increases the resistance against abrasive wear⁷. The thickness and the proportion of each of

those borides depend on the chemical composition of the boriding environment, the temperature and the duration of treatment⁸. The existence of sufficient boron in the environment increases boride layer thickness. If there is not sufficient boron in the environment, that is, if boron is used up in the boron source during the boriding process then the formation of the layer ceases. This way, a thin layer of boride is formed. For the carbon reduction of B₄C, there must be a substance (reductant) in the environment. Thus, with the combination of SiC which is a reductant substance and B₄C, B (elemental boron) is obtained. Carbon-free B demonstrates a very high oxygen affinity and forms B₂O₃ with oxygen. There should be enough SiC in the environment to prevent this. SiC, which provides reduction, also reduces iron, this way, carbon-free boron spreads over the iron. If the concentration of boron on the exterior of the initial Fe₂B layer is maintained at around 9%, the Fe₂B phase continues to grow. On the other hand, if the concentration of boron on the exterior of the Fe₂B layer reaches approximately 16%, FeB forms and grows on top of the Fe₂B layer resulting in a two-phase iron boride layer. The presence of silicon carbide in the boriding pack is essential in order to dilute the boron carbide to a level that ensures that the concentration of free boron in the system is conducive

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to the formation of the single-phase Fe₂B layer⁹⁻¹¹. Usually, the formation of a monophase (Fe₂B) with sawtooth morphology is more desirable than a double phase layer with FeB and Fe₂B. The boron rich FeB phase is not desirable because FeB is more brittle than the iron subboride, the Fe₂B phase. Furthermore, since the FeB and Fe₂B phases exhibit substantially different coefficients of thermal expansion, CTE ($\alpha_{\text{FeB}}=23 \times 10^{-6}/^{\circ}\text{C}$, $\alpha_{\text{Fe}_2\text{B}}=7.85 \times 10^{-6}/^{\circ}\text{C}$), crack formation is often observed at the FeB/Fe₂B interface of a double phase layer. These cracks often lead to flaking and spalling when a mechanical load is applied. Through the control of boronizing process parameters, i.e., boronizing powder composition, temperature and time, the Fe₂B phase can be consistently achieved during pack boriding^{10,12}. At the same time the Fe₂B layer produces superior wear resistance and mechanical properties. Research has mainly focused on two peculiar characteristics of boride coatings. They are as follows: (i) high hardness that is expected to give a high wear resistance and (ii) columnar morphology that is required for a good adhesion between the coating and the substrate¹³.

Ekabor commercial powders are used in industrial applications: (90% SiC, 5% B₄C, 5% KBF₄). When the studies conducted with commercial powders were analyzed it was observed that the FeB and Fe₂B phases were formed¹⁴⁻¹⁹. Chemical composition of 5% activator (KBF₄) is used in the Ekabor commercial powders. This accelerates the spread of boron over iron. It is known that the layer thickness increases with an increase in the rate of the activator (KBF₄)¹⁰. The probability of the occurrence of the Fe₂B and FeB phases increases as the layer thickness increases.

In this study attempted to obtain a single-phase (Fe₂B) boron layer with no activator and wear behavior of the Fe₂B phase was investigated. AISI 1020 and 1040 steels were borided in B₄C and SiC powders in different proportions at 900°C for 3 h with no subsequent activators. The characterization of the formed boride layer was investigated by optical microscope analysis, X-ray diffraction and a microhardness test. As a result of the wear tests, the effect of Fe₂B phase on friction coefficient, surface roughness and wear rate was studied. Worn surfaces of the samples were characterized by scanning electron microscopy and the results were investigated with those obtained for pack borided samples.

Experimental Procedure

The chemical composition of the AISI 1020 and 1040 steels with the dimensions of Ø 20×7 mm used in this study are shown in Table 1. The M1, M2, M3, M4, M5 and M6 (Mx) mixtures shown in Table 2 were used for the boriding process of steels. The boriding process was carried out by putting the study samples (Table 2) into a stainless steel crucible which was then heated in an electrical resistance furnace. Boriding was performed at a temperature of 900°C for 3 h. Cross-sections of boriding steels were prepared metallographically to observe morphological details using an Olympus BX-60 microscope. The presence of borides formed in the coating layers was confirmed by means of X-ray diffraction (Shimadzu XRD-6000) using Cu K α , ($\lambda= 1,5406 \text{ \AA}$) radiation. The thickness of the layers formed on the steels was measured by an optical micrometer attached to the optical microscope (OlympusBX-60). Thickness values given in the results section are averages of at least 15 measurements. The hardness of the layers was measured on the cross-sections using a Micro-Vickers indenter (Shimadzu HVM-2) with a 50 g load. The tribological properties of the borided steels were examined using a ball-on-disk test device.

To perform friction and wear of borided samples a ball-on-disc test device was used. In the wear tests, WC-Co balls of 8 mm in diameter supplied from H.C. Starck Ceramics GmbH were used. The wear experiments were carried out in a ball-disk arrangement under dry friction condition at room temperature with applied load of 10 N and with a sliding velocity of 0.2m/s at sliding distance of 500 m. After the test, the wear volumes of the samples were quantified by multiplying cross-sectional areas of

Table 1—Chemical composition (wt%) of the materials used

Steel	C	Mn	Si	Cr	Mo	Ni	Fe
AISI 1020	0.20	0.4	0.19	—	—	—	Balance
AISI 1040	0.40	0.7	0.19	—	—	—	Balance

Table 2—Compositions (wt%) of boriding mixtures

Mixture	B ₄ C	SiC
M1	95	5
M2	80	20
M3	60	40
M4	40	60
M5	20	80
M6	5	95

wear by the width of wear track obtained from the device Taylor-Hobson Rugosimeter. Wear rate was calculated with using Eq. (1).

$$\text{wear rate} = \text{worn volume} / (\text{applied load} \times \text{sliding distance}), \text{ mm}^3/\text{Nm} \quad \dots (1)$$

The friction coefficient depending on sliding distance was obtained through friction coefficient program. As a result of the wear tests, graphs of friction coefficient, surface roughness and wear rate depending on the boriding mixtures are plotted. Surface profiles of the wear tracks on the samples and surface roughness were measured by a Taylor-Hobson Rugosimeter.

Results and Discussion

Characterization of boride coatings

Figures 1(a-f) and 2(a-f) show optical micrographs of samples of AISI 1020 and 1040 steels, borided at 900°C for 3 h in the M1, M2, M3, M4, M5 and M6 (Mx) mixtures. As shown in Figs 1 and 2, borides formed on the steels substrates had saw tooth morphology, which is typical in borided steels. The boride layer thickness of AISI 1020 and 1040 steels obtained as a result of boriding for 3 h at 900°C is

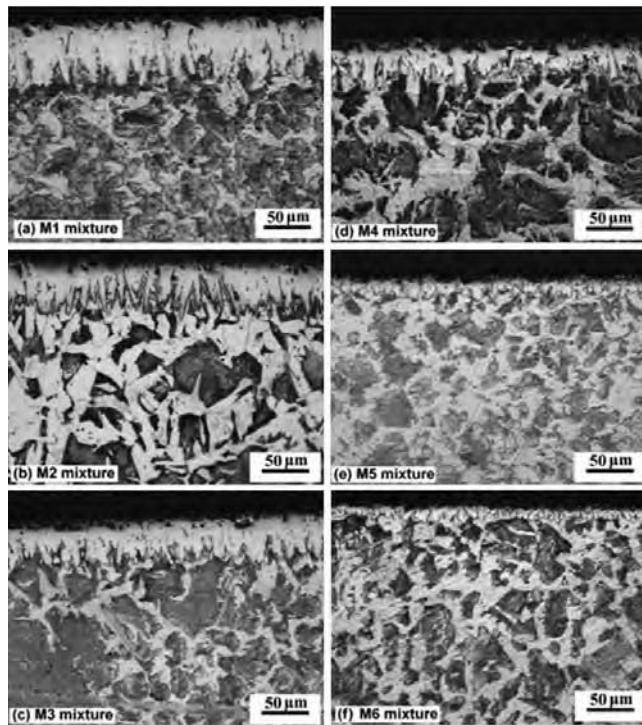


Fig. 1—Cross-section microstructure of borided AISI 1020 steel in different mixtures for 3 h at 900°C, (a) M1, (b) M2, (c)M3, (d) M4, (e) M5 and (f) M6

given in Fig. 3. By using the M1, M2 and M3 mixtures, the boride layer was relatively thicker than in the M4, M5 and M6 mixtures. While the highest boride layer thickness value was recorded for the M1 mixture, the lowest boride layer thickness value was obtained for the M6 mixture on the AISI 1020 and AISI 1040 steels. Together with the decreases in the B₄C% ratio of mixtures M1 to M6, a decrease occurred in the boride layer thickness values. While the increase of boron concentration in the environment also increased the boride layer formation, it is known that

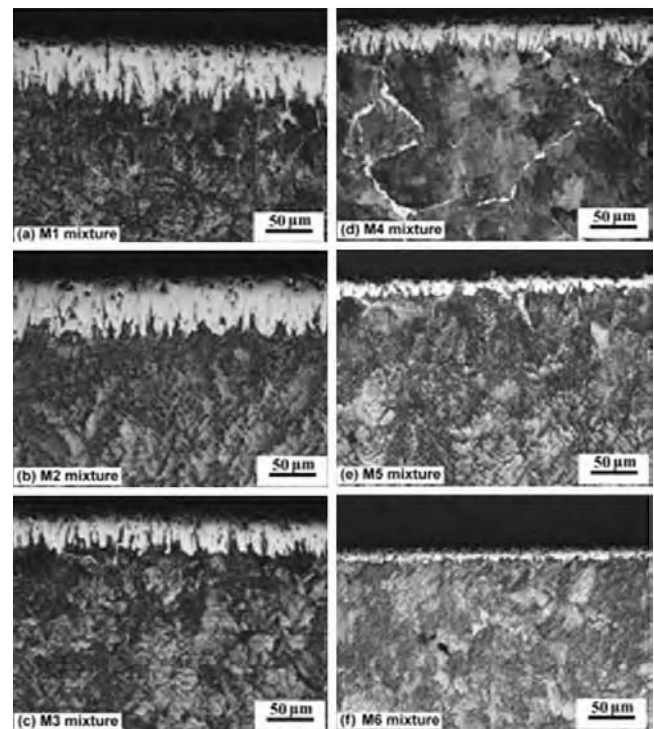


Fig. 2—Cross-section microstructure of borided AISI 1040 steel in different mixtures for 3 h at 900°C, (a) M1, (b) M2, (c)M3, (d) M4, (e) M5 and (f) M6

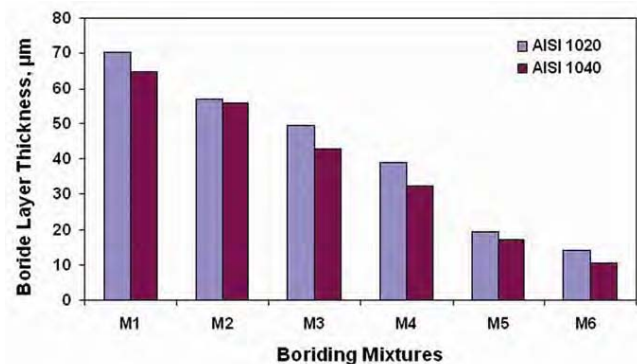


Fig. 3—Boride layer thickness versus different boriding mixtures for AISI 1020 and AISI 1040

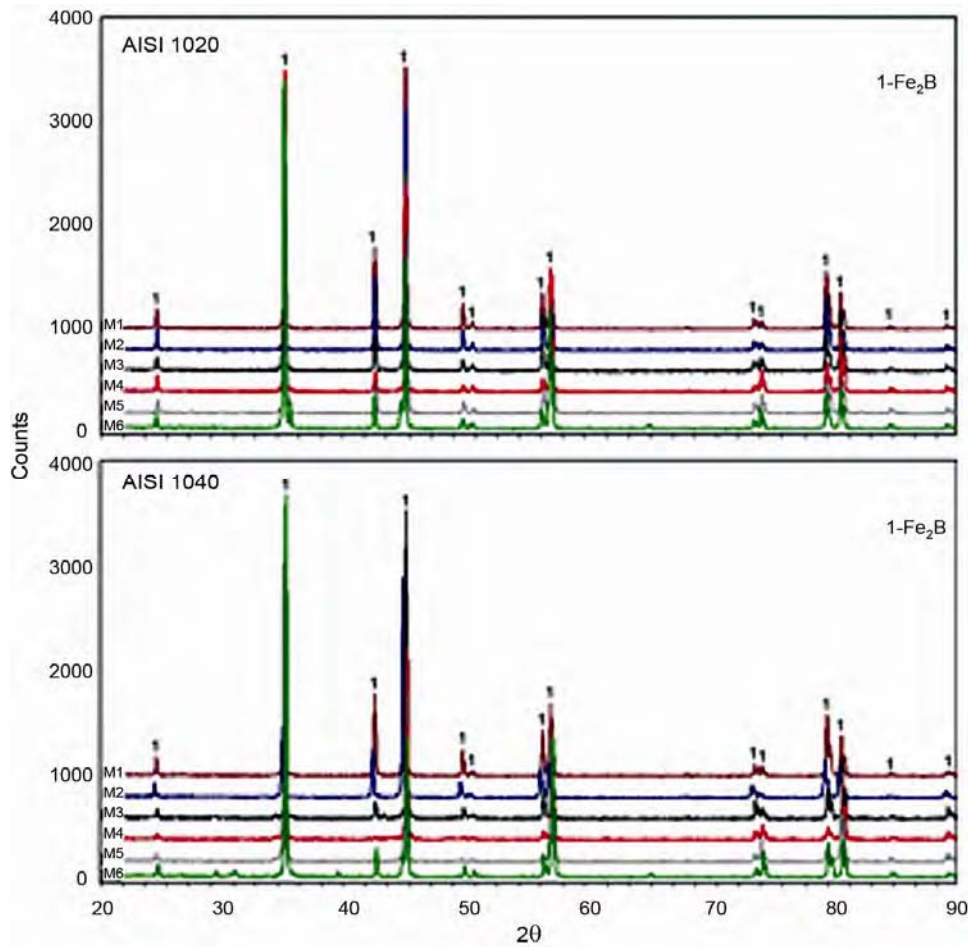


Fig. 4—XRD patterns of the borided steels in different mixtures for 3 h at 900°C, (a) AISI 1020 and (b) AISI 1040

the boride layer decreases along with the decrease of the boron atom potential¹⁹⁻²¹. In addition, the values of the boride layer thickness of AISI 1020, which have low carbon concentration, are reduced^{4,22}.

The X-ray diffraction patterns of the borided steels are given in Fig. 4. In all patterns, the peaks are those of the Fe₂B phase only. Consequently, the FeB phase did not form.

Microhardness measurements were carried out on the cross-sections from surface to interior along a line (Fig. 5). The average values of the micro-hardness of borides on AISI 1020 and AISI 1040 steels were 1776 and 1860 HV_{0.05}, respectively. While the highest microhardness values were obtained for AISI 1040 in the M1 mixture, the lowest microhardness value was obtained for AISI 1020 in the M6 mixture.

Wear and friction

Figure 6 illustrates the variation of surface roughness for AISI 1020 and AISI 1040. It was shown that after the boriding treatment, roughness

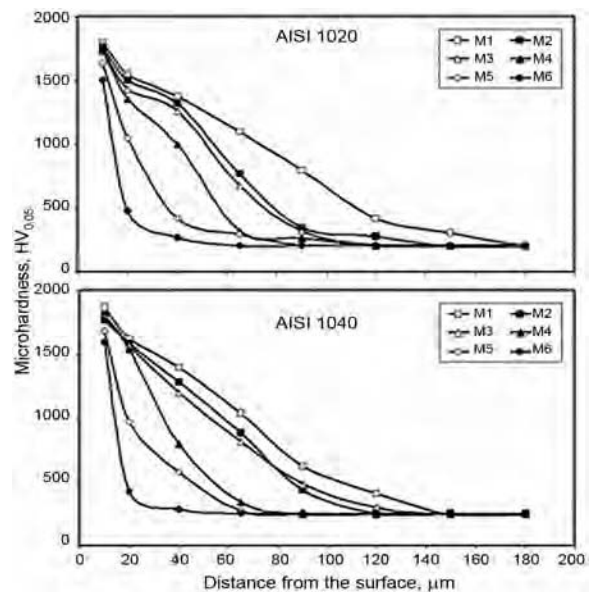


Fig. 5—From surface to core hardness distribution of the steels borided in different mixtures for 3 h at 900°C (a) AISI 1020 and (b) AISI 1040

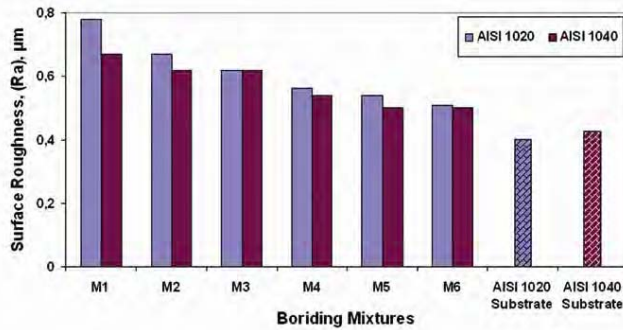


Fig. 6—Variation of surface roughness (R_a) of the AISI 1020 and AISI 1040 steels before and after 3 h in a 900°C boriding treatment

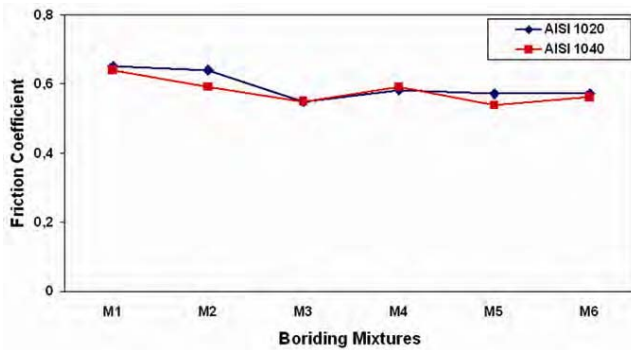


Fig. 7—Variation in the friction coefficient of the borided steels in different mixtures against a WC-Co ball for 10 N loads

increases for surfaces with initially low roughness. A “critical roughness” value was determined such that if surface roughness of a material was below this value before boriding, it reached the critical roughness value after boriding²³. The highest surface roughness value was recorded for the M1 mixture on the AISI 1020 steel. The lowest surface roughness value was recorded for the M6 mixture on the AISI 1040 steel. As can be seen in Fig. 6, for the AISI 1020 and AISI 1040 steels the surface roughness increased with an increase in boride thickness.

Wear tests were carried out in dry conditions at room temperature in air. The friction coefficient values for against the WC-Co ball for Mx mixture specimens borided for 3 h at 900°C are shown in Fig. 7. As can be seen in the graphics, the friction coefficient values in dry medium vary from 0.54-0.65.

The variation in the wear rate for borided steels in different mixtures against the WC-Co ball is indicated in Fig. 8. Figure 8 shows that the wear rate of the borided steels in different mixtures increases with a decrease of $B_4C\%$ into Mx mixtures. While the highest wear rate was recorded for AISI 1020 in the

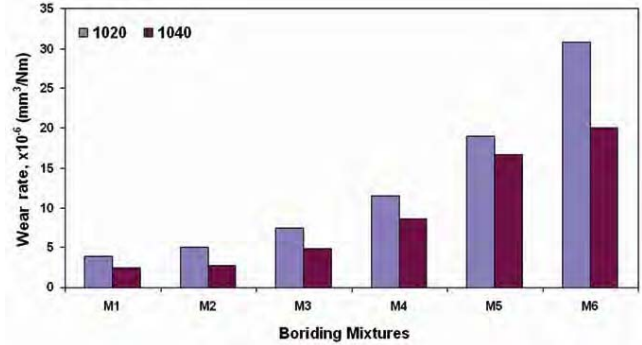


Fig. 8—Variation in the wear rate against a WC-Co ball for 10 N loads for borided steels in different mixtures

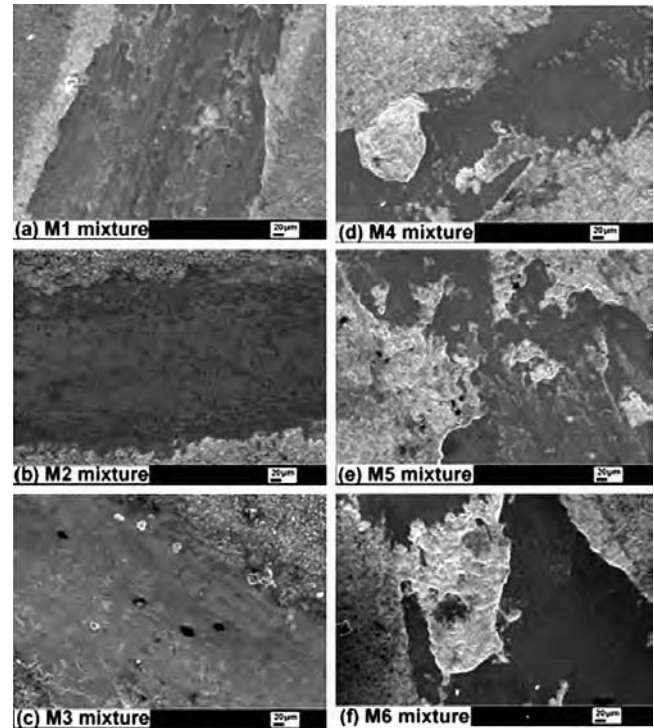


Fig. 9—SEM micrographs of the worn surfaces of the AISI 1040 steel borided in different mixtures; (a) M1 mixture, (b) M2 mixture, (c) M3 mixture, (d) M4 mixture, (e) M5 mixture and (f) M6 mixture

M6 mixture, the lowest wear rate was obtained for AISI 1040 in the M1 mixture. The wear rates of the steels for the M1 mixtures were four times lower than for the M6 mixtures. The relationship between the surface hardness and wear rates of the borided samples also confirms that the wear resistance improves as the hardness increases²⁴⁻²⁶. As can be seen in Fig. 8, while boride layer thickness increases, wear rate decreases²⁷.

As can be seen in Fig. 9 (d-f), it is clear that the wear scars are deeper and wider than in Fig. 9 (a-c) at

SEM micrographs. As the boride layer thickness decreases, wear scars become deeper in the samples. Dark regions (compact layer) on the wear track increased with the decrease in boride layer thickness from the M1 to the M6 mixture. Moreover, there seems to be some debris particles on the surface from the M3 to the M6 mixture.

Borided AISI 1020 and AISI 1040 steels from the M1 to the M3 mixtures are extremely resistant to adhesion on account of their high hardness when compared with M4, M5 and M6. Borided steels are extremely resistant to abrasion and adhesion on account of their great hardness values. Furthermore, the boride layer has a low welding tendency. This property is of great consequence for adhesive wear and explains why borided samples show higher wear resistance^{8,28,29}.

Conclusions

From this study, the following conclusion can be drawn:

- (i) Depending on the boriding mixtures, the thickness of borides formed on the surfaces of borided AISI 1020 and AISI 1040 steels ranges from 14 to 71 μm and 10 to 64 μm , respectively.
- (ii) The boride layers formed on the surfaces of the AISI 1020 and AISI 1040 steels have tooth structures and it was observed that the layer thickness increased with increases in the $\text{B}_4\text{C}\%$ ratio.
- (iii) As the $\text{B}_4\text{C}\%$ ratio decreases from M1 to M6, a decrease in the boride layer thickness of the steels was observed.
- (iv) X-ray diffraction analysis showed that the single-phase Fe_2B saw-tooth shaped boride layer was obtained on the surface of all specimens.
- (v) The average values of micro-hardness of borides on the AISI 1020 and AISI 1040 steels were 1776 and 1860 $\text{HV}_{0.05}$, respectively.
- (vi) It was detected that the values of surface roughness increase with an increase in the values of boride layer thickness.
- (vii) Under dry conditions, the wear resistance of borided AISI 1040 steel is higher than that of borided AISI 1020 steel due to the high surface hardness. Adhesive wear occurred in these steels. The best wear resistance was obtained for

the samples borided from higher of B_4C proportion.

- (viii) The wear rate of the AISI 1020 and AISI 1040 steels increased with the decrease in boride layer thickness from M1 to M6.

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2013		
February	April	June
August	October	December
[Full Text Articles]		

2012		
February	April	June
August	October	December
[Full Text Articles]		

2011		

<u>February</u>	<u>April</u>	<u>June</u>
<u>August</u>	<u>October</u>	<u>December</u>
[Full Text Articles]		

2010		
<u>February</u>	<u>April</u>	<u>June</u>
<u>August</u>	<u>October</u> Special issue on Multifunctional Carbon Materials for 21 st Century	<u>December</u>
[Full Text Articles]		

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<u>February</u>	<u>April</u>	<u>June</u> Special issue on Luminescence and its Applications
<u>August</u>	<u>October</u>	<u>December</u>
[Full Text Articles]		

2008		
<u>February</u>	<u>April</u>	<u>June</u>
<u>August</u>	<u>October</u>	<u>December</u>
[Full Text Articles]		

2007		
<u>February</u>	<u>April</u>	<u>June</u>
<u>August</u>	<u>October</u>	<u>December</u>
[Full Text Articles]		

2006		
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<u>August</u>	<u>October</u>	<u>December</u>
[Full Text Articles]		

2005		
<u>February</u>	<u>April</u>	<u>June</u>
<u>August</u>	<u>October</u>	<u>December</u>
[Full Text Articles]		

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<u>February</u>	<u>April</u>	<u>June</u>
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2003		2002	
<u>February</u>	<u>April</u>	<u>February</u>	<u>April</u>
<u>June</u>	<u>August</u>	<u>June</u>	<u>August</u>
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