Microabrasive wear behavior of borided steel abraded by SiO\textsubscript{2} particles

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\section*{ABSTRACT}
Boriding treatment was applied to AISI 1020 steel to improve its wear resistance. The samples were characterized by X-ray diffraction (XRD), scanning electron microscopy, Vickers microhardness testing, fracture toughness, and confocal microscopy. The microscale abrasive wear behavior was also investigated. SiO\textsubscript{2} abrasive particles were used as abradant with slurry concentrations of 0.5 and 1.0 g/cm\textsuperscript{2}. Normal loads of 0.49 and 0.98 N were used. Fe\textsubscript{2}B phase was identified in the boride layer via XRD analysis. The Fe\textsubscript{2}B layer was 169 \textmu m thick with a mean hardness of 1608 ± 101 HV\textsubscript{0.05} and fracture toughness of 5.35 ± 1.43 MPa m\textsuperscript{1/2}. A reduction in the hardness of the outermost surface of the boride layer was observed owing to the formation of a porous region. Boriding treatment improved the wear resistance of the steel substrate. Sliding abrasive wear was the main mechanism under all tested conditions. The presence of micro-rolling abrasion and fracture-based mechanisms was observed for untreated and borided samples, respectively.

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\section{1. Introduction}
Carburizing [1], nitriding [2,3], and boriding [4,5] are well-known diffusion treatments used to improve the surface properties of metallic substrates such as corrosion resistance, hardness, and wear resistance where it is mostly needed.

Boriding (boronizing) is a thermochemical surface treatment that allows the formation of the FeB and Fe\textsubscript{2}B boride phases owing to the diffusion of boron atoms in ferrous substrates [6,7]. The boriding treatment of ferrous alloys is generally performed between 840 and 1050 °C [8] in powder-packed [9,10], paste [11], gaseous [12], and salt [13,14] media. The powder-packed processes have the advantages of simplicity and cost effectiveness compared with the other boriding media [15].

Since boriding is a well-established surface improvement process, there are a number of studies focused on its abrasive
[13,16–18] and sliding [19–22] wear behaviors. The microabrasive wear behavior of borided steels, however, is not very well understood and there are a limited number of studies on this area [23–25].

Martini et al. [23] performed boriding thermochemical treatment on the surface of pure iron and medium carbon steel substrates by a powder-packed process at 850 ºC for 15 h. Polyphase (FeB–Fe2B) boride coatings were grown on the ferrous substrates. The abrasive wear resistance was evaluated using a microabrasive wear tester with the fixed-ball configuration. A silicon carbide (SiC) (4–5 µm) abrasive slurry was used. The normal load and sliding speed were 0.2 N and 0.05 m/s, respectively. Owing to the presence of an outer layer composed of disordered crystals, the authors reported that the wear rate in borided coatings was initially high, under both sliding and microabrasive conditions. According to their work, the minimum wear rate was obtained in the compact inner regions of the coatings, which comprised highly ordered Fe2B crystals.

A microscale abrasive wear tester with free-ball configuration was used by Gunen et al. [24] to evaluate the microabrasive wear behavior of borided AISI 304 stainless steel. The boriding treatments were carried out at 950 and 1000 ºC for 2 and 4 h under each condition (temperature). FeB and Fe2B phases were observed after thermochemical treatments. A SiC (5 µm) abrasive slurry was used, and the sliding speeds of the ball were 0.1, 0.14, and 0.2 m/s. According to the authors, after the boriding, the abrasive wear resistance improved slightly. Rolling abrasion was the dominant wear mechanism. The wear coefficients were not determined in the study.

Krelling et al. [25] reported that for AISI 1020 samples borided at 1000 ºC for 4 h, the wear resistance worsened compared with untreated samples. There was only the presence of Fe2B phase in the boride layer. A microscale abrasive tester with a fixed-ball configuration was used. The SiC abrasive slurry concentration varied between 0.5 and 1.0 g/cm³, and the applied loads used were 0.49 and 0.98 N. Rolling abrasion was the wear mechanism observed in borided samples, and grooving abrasion with microrolling occurred in the untreated samples. The authors attributed the better wear resistance of the untreated samples to the embedment of the SiC abrasive particles on the surface of the wear craters. In this way, the untreated samples were covered with a SiC layer, improving their wear resistance compared with the borided specimens.

In addition to the greater realism of SiO2 abrasive particles [26] and their common presence in industrial applications as contaminants [27], the works that address the microabrasion topic in borided steels usually use SiC as the abrasive. These abrasive particles, however, are rarely seen in real slurries. The use of abrasive particles that do not match the characteristics of real abrasives can make it difficult to obtain particular wear features observed in real applications [28]. However, only a few studies about microabrasion use SiO2 as an abrasive agent [29,30]. Therefore, the aim of this paper is to improve the understanding on the microscale abrasive wear behavior of borided steels when they are abraded by soft, fine SiO2 abrasive particles in highly abrasive slurry concentrations.

2. Experimental procedure

2.1. Powder-packed boriding treatment

Samples of AISI 1020 mm thick steel were cut from a bar of 12.7 mm in diameter, sanded with silicon carbide abrasive paper up to 600 grit, and polished with 1 µm alumina suspension before the boriding thermochemical treatment. The powder-packed boriding treatment was carried out at 1000 ºC for 4 h. The samples were placed in a stainless steel container and covered with 15 mm of Ekabor 1-V2 boriding powder with a composition of 5 wt.% B4C, 5 wt.% KBF4, and 90 wt.% SiC [22,31]. After the treatment, the samples were cooled in air. Boriding parameters were selected from a previous work [25].

2.2. Sample characterization

The stoichiometry and morphology of the borides formed on an AISI 1020 steel surface were analyzed by X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM), respectively. XRD analysis was performed in a Shimadzu 6000 diffractometer with Cu Kα radiation and 2θ range of 25–120°. For SEM analysis, the samples were cut along their longitudinal section, sanded with SiC abrasive paper up to 600 grit number, polished with 1 µm Al2O3 suspension, and etched with 3% Nital solution for 5 s.

The surface topography analysis was performed in a Leica DCM 3D confocal microscope at a magnification of 10 ×. A Future-Tech FM-800 microhardness tester with 50 gf and 500gf loads applied for 10 s (dwell time) was used to measure the micro-Vickers hardness profile and fracture toughness of the boride layer, respectively. Five indentations were made on each position from the surface to determine the hardness profile. For fracture toughness, 15 measurements were made in the boride layer. The Vickers half-diagonals (l), microcrack lengths (g), and total crack length (c) were measured according to Fig. 1.

![Image](image-url)

Figure 1 – Palmqvist microcracks produced at the corners of the indentation on the boride Fe2B layer.
The Palmqvist crack model proposed by Laugier [32], Eq. (1), was used to determine the fracture toughness of the boride layer:

$$K_c = k^p \cdot \left( \frac{g}{1} \right)^{-1/2} \cdot \left( \frac{E}{H} \right)^{2/3} \cdot \frac{p}{c^{3/2}}$$

(1)

where $K_c$ is the fracture toughness, $k^p$ to 0.015, $E$ is Young’s modulus of the Fe$_2$B phase (290 GPa), $H$ is the microhardness, and $P$ is the applied load.

2.3. Microscale abrasive wear tests

The microscale abrasive wear tests were performed using a fixed-ball configuration TE-66 SLIM Tribometer from Phoenix Tribology. The tests were performed with abrasive slurry concentrations of 0.5 or 1.0 g/cm$^3$ (grams of SiO$_2$ per cm$^3$ of distilled water). The applied loads were 0.49 or 0.98 N, and the rotary speed was 70 rpm, resulting in a 0.1 m/s sliding speed to avoid hydrodynamic effects. As with the boriding parameters, the values of abrasive slurry concentration and the applied load were selected from the literature [25]. Three repeated tests (same conditions) were performed for each sliding distance in the range 50–1200 ball revolutions. The wear crater diameters were optically measured in steps of 50 ball revolutions up to 1200 revolutions, to identify the steady-state regime (SSR). Eq. (2) was used to calculate the wear coefficient $k$ as a function of wear crater diameter $b$, radius of the ball $R$, sliding distance $S$, and normal applied load $N$. The wear coefficient value is the mean of 39 values (at least) for each condition:

$$k = \frac{n b^4}{64 S}$$

(2)

where $b$ and $R$ are the wear crater diameter and the radius of the ball, respectively:

$$h = \frac{b^2}{8R}$$

(3)

Before each set of experiments up to 1200 revolutions, the 25.4 mm diameter AISI 52100 steel ball was conditioned by manually shaking it in a mixture containing silica sand and distilled water for 10 min. Following this, the ball was ultrasonically cleaned in ethyl alcohol for 10 min.

The abrasive used in this work was SiO$_2$ from Sigma-Aldrich with a mean particle diameter of 3 μm. According to the manufacturer, the particle size distribution is approximately 99% between 0.5 and 10 μm and 80% between 1 and 5 μm. Fig. 2 shows an SEM image of the abrasive particles.

3. Results and discussion

3.1. Microstructure

The XRD pattern for borided AISI 1020 steel is shown in Fig. 3. There is only the presence of Fe$_2$B boride phase over the surface of the AISI 1020 steel after the boriding thermochemical treatment. This result is in agreement with the studies of Béjar and Moreno [13] and Krelling et al. [25] on borided AISI 1020 steel. However, depending on the matrix composition, treatment temperature, and time, boriding with Ekabor powder could lead to the formation of a dual-phase layer consisting of FeB (external) and Fe$_2$B (internal) phases [22].

Owing to its low amount of alloying elements, the Fe$_2$B boride layer presented a sawtooth morphology (Fig. 4) with a thickness of 169 ± 15 μm.

The large surface boron concentration leads to the formation of a porous outermost region [33]. This porous region observed at the surface of the boride layer is due to the formation of the initially disordered boride crystals formed over the steel surface and the transformation of Fe into Fe$_2$B, which is associated with a 16% increase in volume [7]. The presence of this porous zone was observed in other works [34,35] and is very common in boriding processes.

The microhardness profile curve for borided and untreated AISI 1020 steel is shown in Fig. 5. A hardness of 174 ± 8
values, which were more sensitive than $S_o$ to large deviations from the mean line [37], measured for untreated and borided specimens were $0.23 \pm 0.13$ and $3.16 \pm 0.04 \mu m$, respectively. Initially, smooth surfaces saw an increase in their roughness values owing to the Fe$_2$B crystal formation. This behavior was also observed in other works [8,22,25]. An example of the untreated and borided surface topography is given in Fig. 6.

The fracture toughness achieved in this work for Fe$_2$B boride phase was $5.35 \pm 1.43 \text{ MPa m}^{1/2}$, which is 2–20% higher than the values obtained in the works cited in Table 1. The fracture toughness value depends strongly on the boride types [38], alloying elements, boriding time, and temperature [39]. The vast majority of the works in Table 1 used steel substrates with high amounts of alloying elements (tool steels), which lead to the formation of a borided layer composed of FeB and Fe$_2$B and, consequently, diminish the fracture toughness. Higher boriding times and temperatures also diminish the fracture toughness of steel substrates [40]. In addition, treatment time seems to have a stronger influence than temperature on the reduction of fracture toughness when low-alloy steels are compared.

Another important feature that might be considered is that for high-alloy steels (tool steels), the presence of a dual-phase boride layer makes it difficult to discern what kind of interactions exists between different borides and, consequently, the influence of each one [39]. Only the cracks parallel to the surface were considered in the fracture toughness calculation, because it is the critical condition of the fracture toughness of Fe$_2$B boride phase [41].

3.2. Microscale abrasive wear behavior

To validate the use of Eqs. (2) and (3), topographical measurements of the wear craters produced during the microscale abrasive wear test were carried out. As shown in Fig. 7, for the untreated condition, the wear crater reflected the counterbody geometry (ball of 12.7 mm radius). Fig. 7c was obtained from the profile identified by the black line observed in Fig. 7a. The average wear crater radius, measured after the abrasive wear tests, was 12.9 and 12.2 mm for untreated and borided conditions, respectively.

The SSR was achieved between 400 and 600 revolutions (32–48 m) of the ball for the untreated and borided conditions, respectively. The SSR was determined by analysis of the variation of the wear coefficient ($k$) and the volume of the material removed (VMR) according to the sliding distance. Fig. 8a shows the variation of the wear coefficient with test duration. In

| Table 1 – Fracture toughness values of Fe$_2$B layer obtained in this study in comparison with other works. |
|-------------------------|-----------------|-----------------|-----------------|
| $K_c$ (MPa m$^{1/2}$)  | Substrate       | Boriding conditions | Reference       |
| Time (h) | Temperature (°C) |
|-------------------------|-----------------|-----------------|-----------------|
| 4.50 | AISI 1045 | 8 | 1000 | [41] |
| 5.24 | AISI W4 | 6 | 1050 | [38] |
| 4.65 | AISI D2 | 3 | 1000 | [34] |
| 4.46 | AISI H13 | 5 | 900 | [40] |
| 4.79 | AISI P20 | 2 | 950 | [42] |
| 5.35 | AISI 1020 | 4 | 1000 | This study |
Figure 6 – Surface topography of (a) untreated and (b) borided samples.

Figure 7 – Topographical analysis of the wear crater produced during the micro-scale abrasive wear test for untreated condition. (a) Wear crater; (b) SEM of the wear crater shown in (a); (c) profile in the center of the crater (black line).
According to Table 2, the abrasive slurry concentrations have no influence in the wear coefficient value for untreated specimens. For borided specimens, increasing the abrasive slurry concentration resulted in increases of 120 and 200% for 0.49 and 0.98 N of applied load, respectively. The wear coefficient decreased with increasing applied load for all tested conditions. The boriding treatment resulted in a reduction of the wear coefficient up to 80% (0.98 N and 0.5 g/cm³) compared with untreated specimens.

The better abrasive wear resistance is attributed to the formation of a hard (1608 ± 101 HV0.05) Fe2B boride layer during the boriding treatment of the AISI 1020 steel. Because the higher wear crater depth obtained for borided specimens after 1200 ball revolutions was 11.3 ± 0.8 μm, it is reasonable to assume that the wear resistance is determined by the boride layer abrasive wear behavior. Although a porous boride layer was formed, it can be assumed that the boriding treatment was effective for increasing the wear resistance of the AISI 1020 steel under the conditions established in this study because this outermost porous layer was not completely ripped off.

Gomez et al. [43] evaluated the effect of the abrasive particle size distribution on the wear rate and wear mode in microscale abrasive wear tests. In their study, the authors used SiC as the abradant. The size of the SiC particles ranged from 2.1 to 6.6 μm. Samples were made of AISI 1020 steel, the rotational speed of the ball was fixed at 150 rpm, the abrasive slurry concentration was 0.1 g/cm³, and the applied loads were 0.2 and 0.4 N. For particles size of 3 μm and 0.4 N (18% less than the load used in this work), they found a mean wear coefficient of 0.2 × 10⁻³ mm³/N m (43% greater than the k value achieved in this work).

In terms of the wear modes, in [43] the presence of embedded SiC abrasive particles could be observed in the wear crater. This embedment, according to other works [25,44–46], can be the responsible for the reduction in the wear coefficient of ductile materials compared with harder surfaces. In all these works, the authors used SiC as the abradant.

The microscale abrasive wear mechanisms were identified by SEM analysis. Sliding (2-body, grooving) abrasion was the main microscale abrasive wear mechanism observed for untreated specimens (see Fig. 9). The sliding abrasion mechanism is characterized by the formation of grooves aligned in the sliding direction [47], as observed in Fig. 9. The presence of adhered material over the wear crater surfaces was also observed for an applied load of 0.98 N (Fig. 9d) promoted by the

![Figure 8](image)

**Figure 8** – Example of test evolution in a borided specimen at a normal load of 0.49 N and abrasive slurry concentration of 1.0 g/cm³. (a) k = f(S); (b) VMR = f(S) in SSR.

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**Table 2** – Wear coefficients (k) and linear regression coefficients (R²) for all tested conditions.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Load (N)</th>
<th>Slurry conc. (g/cm³)</th>
<th>k × 10⁻³ (mm³/N m)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Borided</td>
<td>0.49</td>
<td>0.5</td>
<td>0.05 ± 2.6 × 10⁻⁶</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>0.98</td>
<td>0.5</td>
<td>0.02 ± 2.7 × 10⁻⁶</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>0.49</td>
<td>1.0</td>
<td>0.11 ± 6.0 × 10⁻⁶</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>0.98</td>
<td>1.0</td>
<td>0.06 ± 5.2 × 10⁻⁶</td>
<td>0.96</td>
</tr>
<tr>
<td></td>
<td>0.49</td>
<td>0.5</td>
<td>0.14 ± 6.7 × 10⁻⁶</td>
<td>0.98</td>
</tr>
<tr>
<td></td>
<td>0.98</td>
<td>0.5</td>
<td>0.14 ± 8.4 × 10⁻⁶</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>0.49</td>
<td>1.0</td>
<td>0.09 ± 7.8 × 10⁻⁶</td>
<td>0.99</td>
</tr>
<tr>
<td>Untreated</td>
<td>0.98</td>
<td>1.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
plastic deformation of the AISI 1020 substrate. The difficulty of the abrasive particle entrainment in the contact region is a reasonable explanation for these phenomena, as described previously in [48]. The ridge formation is more likely to be present when grooving abrasion occurs, at high loads (over than 0.5 N) and in ductile materials [49,50].

When the wear crater surfaces are observed at higher magnification (Fig. 10), the presence of the microrolling abrasion mechanism can be observed. In this mechanism, there are some abrasive indentations along the grooves, promoted by the rolling motion of the abrasives in the wear contact. The microrolling abrasive wear mechanism was observed by Cozza et al. [51] and described with more detail in [52]. In Fig. 10a and b, the presence of some embedded SiO$_2$ abrasive particles can be observed. Only the 0.49 N, 0.5 g/cm$^3$ (Fig. 10a), and 0.98 N, 1.0 g/cm$^3$ (Fig. 10b) conditions are shown.

Krelling et al. [25] used the same test conditions in their work, except for the type of abrasive (SiC). The authors found many SiC abrasive particles embedded in the wear craters of untreated specimens; according to their work, this explains the higher wear resistance of untreated specimens compared to borided ones. In this work, a rounder and softer (SiO$_2$) abrasive was used. Thus, it is expected that it is more difficult for the SiO$_2$ particles to become embedded in the AISI 1020
surface to protect the surface against abrasion. The wear coefficient obtained in this work is up to 26× lower than that reported by Krelling et al. [25].

The severity of contact (Sc) for untreated specimens was estimated in the range 0.007–0.025, depending on the test conditions, according to the model proposed by Adachi and Hutchings [53]. In their model, the severity of the contact can be calculated by Eq. (4), where $W$ is the normal load, $\phi$ is the abrasive slurry volume fraction, and $A$ is the region over which the separation of the ball and the specimen is less than the diameter of the abrasive particles. The parameters $H'$ and $A$ are calculated according to Eqs. (5) and (6):

$$Sc = \frac{W}{A\phi H'}$$

$$\frac{1}{H'} = \frac{1}{H_b} + \frac{1}{H_s}$$

$$A = \pi \left( a^2 + 2nd \right)$$

In Eq. (5), $H_b$ is the hardness of the ball and $H_s$ is the hardness of the specimen. In Eq. (6), $D$ is the diameter of the abrasive particles, $R$ is the radius of the ball, and $a$ is the radius of the Hertzian contact area.

The values of $Sc$ obtained for untreated condition are close to the mixed (sliding + rolling) and rolling behavior shown in the author's wear mode map [53], different from the wear mechanisms observed by SEM in this work. It is important to note that Adachi and Hutchings used SiC as the abrasive agent, which is larger, harder, and stiffer than SiO$_2$.

The sliding microscale abrasive wear mechanism was identified as the wear mechanism for borided specimens. The presence of cracks is observed in some regions of the images presented in Figs. 11 and 12, which is a higher magnification of the center of the wear craters. Compared to untreated specimens, the grooves produced by the abrasive particles are shallower, which is attributed to the higher hardness of the borided specimen surface and its higher load-bearing capacity [54,55]. In addition, owing to its higher hardness, the presence of the microrolling mechanism was not observed in the wear surfaces of the borided specimens.

The fracture of the borided layer is a consequence of the porous region present in the surface region of the boride layer, as identified in Fig. 12b by the presence of crack propagation from a pore.

The fracture toughness value of Fe$_3$B phase has a direct influence on the wear mechanisms observed in borided specimens. The presence of a fragile phase (5.35 MPa m$^{1/2}$) changes

Figure 11 – Wear craters in borided specimens after 1200 revolutions: (a) 0.49 N, 0.5 g/cm$^3$; (b) 0.49 N, 1.0 g/cm$^3$; (c) 0.98 N, 0.5 g/cm$^3$; (d) 0.98 N, 1.0 g/cm$^3$. 
the wear mechanism from a deformation-based to a fracture-based mechanism. In addition, FeB and Fe₂B are formed under tensile and compressive residual stresses, respectively [6]. Because only Fe₂B phase was detected in XRD analysis (Fig. 3), it is expected that this compressive stress will retard crack growth, leading to higher Kc values compared to the FeB phase and better wear resistance. However, the influence of boriding time and temperature and, consequently, the fracture toughness of boride layers in the microabrasive wear resistance and wear mechanisms of borided steels should be further investigated.

Comparing the wear coefficient values obtained in this work with those described in [25] for borided specimens a decrease up to 150×, depending on the test condition, was observed when SiO₂ was used as the abrasive.

The severity of the contact estimated for the borided specimens is in the range of 0.001–0.004, which is in the rolling abrasion mechanism region on the Adachi and Hutchings [53] wear map. Because of the difference between the bulk and the boride layer hardness, 9.9 and 19 GPa, respectively, it is expected that the SiO₂ abrasive particles become embedded in the ball surface and, consequently, produce grooves on the specimen surface (2-body/sliding abrasive wear mechanism).

When softer deformable abrasives are used, combined with the higher initial roughness of borided specimens, particle entrainment becomes difficult in the contact region, leading to the occurrence of the sliding abrasive wear mechanism [30]. Although the load per particle decreases with increasing test duration [29,56], if the particles become embedded outside the contact region, the occurrence of the rolling mechanism will not occur although the sliding distance increases.

- After the boriding thermochemical treatment, there is an increase in the initial roughness owing to the formation of the boride layer;
- Boriding treatment increased the wear resistance of AISI 1020 steel;
- The main wear mechanism observed, regardless of the condition, was sliding abrasive wear, favored mainly by the characteristics of the abrasive;
- Microrolling abrasion was observed in the untreated specimens;
- The presence of a porous region in the Fe₂B layer affects the wear mechanism of the borided specimens, favoring the occurrence of cracks in the wear craters;
- More realistic abrasives such as SiO₂ allow better evaluation of the tribological behavior of ductile materials submitted to boriding thermochemical treatment.

5. Conflicts of interest

The authors declare no conflicts of interest.

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