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Microstructural and tribological characterization of niobium boride coating produced on AISI 1020 steel via multicomponent boriding

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Abstract

Niobium boride coating was produced on AISI 1020 steel by multicomponent boriding process. Boriding treatment at 1000 °C for 4 h was followed by thermo-reactive diffusion technique at 1000 °C for 6 h under argon atmosphere. Microabrasive wear tests were carried out using SiC abrasive particles at slurry concentrations of 0.5 and 1.0 g cm⁻³. Normal loads of 0.49 and 0.98 N were used. NbB and Nb₅B₆ phases were identified by XRD analysis. The niobium boride coating thickness was 2.0 ± 0.5 μm and its hardness was 1360 ± 200 HK₀.₀₁. Owing to the presence of a porous region on the niobium/iron boride layers the abrasive wear resistance decreased comparing to the borided and untreated AISI 1020 steel. Rolling abrasion was the main wear mechanism observed.

1. Introduction

Aiming low costs, high versatility and availability, carbon steels are extensively studied and applied [1]. Low carbon steels with surface treatments that promote higher hardness and wear resistance can substitute expensive high-alloyed steels in some applications [2, 3].

Machine elements and parts subjected to aggressive environments and severe wear conditions require surfaces with high hardness and corrosion resistance [4]. The use of surface treatment processes in engineering materials can thus improve their wear, oxidation and corrosion resistance, among other properties.

Thermo-Reactive Deposition Diffusion (TRD) treatments are alternative processes that can substitute coating processes of steels resulting in a hard and wear resistant layer of carbides, nitrides and borides [5]. The low cost is among the advantages of TRD treatments, since these treatments are carried out at atmospheric pressures, and do not require special equipments as the ones applied in Physical Vapour Deposition (PVD) and Chemical Vapour Deposition (CVD) of coatings [4]. In TRD processes, the carbon, nitrogen or boron from the substrate diffuses to the surface and reacts with a deposited layer of some carbide-, nitride- or boride-forming element such as Y, Nb, Ta, Cr, Mo or W. The process is carried out at high temperatures and can be performed in salt bath [6], fluidized bed [7] or solid media [8–10].

Considering the powder-pack method the TRD treatment powder usually consists of a metal element, which can be pure or in the form of iron-alloy, that will be deposited on the steel surface, an inert ‘diluent’ (Al₂O₃ or SiC) which does not take part in the formation reactions of the layers and avoids metal’s particle sintering; and an activator (NH₄Cl or KBF₄) which, when dissociated, chemically binds with the carbide former and forms chlorides/fluorides. These chlorides/fluorides then react with the substrate atoms to form carbides, nitrides or borides layer [11, 12].

Multicomponent boriding involves the boriding and the diffusion of some boride-forming element either solid or in a borax bath [13]. In this process, boriding can occur before or after the diffusion of a metallic element (through TRD technique). In multicomponent boriding, complex boron phases can provide increased hardness, corrosion resistance, wear and oxidation resistance at elevated temperatures compared to the simple iron boride phases obtained by conventional boriding thermochemical treatment [14]. These complex boron phases are obtained by the reaction of boron atoms with other metallic atoms such as chromium [15], aluminum [9, 16],...
vanadium [11, 17], titanium [12], niobium [5, 18], and others. Because it is a diffusion-controlled treatment, multicomponent boriding provides better adhesion of the boride layer to the substrate, compared to PVD and CVD treatments in which there is little or no diffusion of the desired coating [19].

Niobium borides are recognized among transition metal borides as potential candidates for structural applications at elevated temperatures due to their melting temperatures, mechanical strength, thermal and electrical conductivity, chemical stability and wear resistance [20]. In the last few years some works have been carried out to obtain and characterize hard layers of transition metal borides. Usta et al [21] performed solid boriding treatment in pure niobium, tungsten and chromium substrates. Pin-on-disc wear tests were carried out without lubrication, with 6 mm AISI 52100 steel balls and 5 N applied load. NbB2, WB and CrB layers were formed on the surfaces of niobium, tungsten and chromium substrates, respectively after the boriding treatment. The tribological behavior was modified from ductile to brittle after surface treatment. Tantalum borides (TaB and TaB2) were obtained by powder-pack boriding treatment on pure tantalum substrates by Ribeiro et al [22]. The tribological behavior was evaluated by linearly reciprocating wear tests in simulated body fluid. The surface hardness increased from 870 HV to 3419 HV after the boriding treatment. This hardness increase makes tantalum prone to nucleation and crack propagation due to surface brittleness. The boriding treatment was considered unsuitable for tribological applications.

Sen et al [18] obtained NbB and Nb3B2 on AISI M2 steel substrate by multicomponent boriding treatment. The niobium boride layer reached 2700 HV, n.0, hardness and thickness of 3.25 μm. Multicomponent boriding treatments were also carried out on AISI 319 L stainless steel by Samadi and Habibolahzade [12]. As a first step, solid TRD treatments were used to diffuse chromium, titanium and vanadium on the metal surface. After the TRD treatment, solid boriding was performed. CrB, TiB2 and VB borides layers were obtained. Borochromatized specimens showed the lower wear rate in pin-on-disc wear tests under 75 N and 115 N applied loads. All specimens presented better wear behavior after the multicomponent boriding treatment in comparison to untreated steel. After the surface treatments, the wear mechanism changed from grooving and plastic deformation to a predominant fatigue/oxidation mechanism.

More recently, multicomponent boriding has been applied to Inconel 625 alloy to form titanium boride coatings. When compared to untreated alloy, borotitanized alloy displayed higher wear resistance in micro-abrasive wear tests [23].

Low carbon steels have received attention from as substrates for the application of multicomponent boriding treatments. Boro-chromo-nickelising [24], borocarburizing [25], boron-nitriding [26] and boron-nitro-carburizing [27]. These treatments proved to be efficient in order to improve the ductility and toughness, corrosion resistance, tribological behavior and microhardness comparing to untreated substrates.

The formation and characterization of niobium boride coatings over low alloy and low carbon steels by TRD treatment technique or the tribological characterization of these coatings under microabrasive wear test conditions is not described in literature. Thus, in the present study, multicomponent boriding was carried out on AISI 1020 steel in order to obtain a niobium boride surface layer. The surface roughness, microstructure, microhardness and microabrasive wear behavior were examined and the results were compared with that of untreated steel results from our previous work [28]. This investigation can elucidate important questions about the abrasive wear behavior of niobium boride coated low carbon steels and contribute to the expansion of the application field of these steels.

2. Experimental procedure

2.1. Materials and treatments

An AISI 1020 steel rod with a diameter of 12.7 mm was cut into samples 6 mm thick. The samples were sanded up to 600 SiC emery paper and polished with 1 μm alumina suspension before the surface treatments.

Multicomponent boriding treatment was performed in two steps: I) Solid boriding thermochemical treatment and, II) Niobizing TRD treatment.

The boriding treatment was performed in solid medium with Ekabor 1-V2 boriding agent with a chemical composition of 5 % wt. B4C, 5 % wt. KBF4 and 90 % wt. SiC [29]. The samples were placed in a stainless steel crucible and heated up to 1000 °C during 4 h in a muffle furnace and covered with a minimum of 15 mm of boriding powder. After the treatment, the samples were cooled in air.

Before the niobizing treatment the borided samples were ultrasonically cleaned for 10 min with ethyl alcohol. Based in the work of Samadi, et al [12], niobizing treatment was performed in solid medium with a powder mixture of 25 % wt. FeNb, 6 % wt. NH4Cl and 69 % wt. Al2O3. Each sample was placed in an alumina crucible sealed with refractory cement. The treatment was carried out in an electrical resistance furnace under an argon atmosphere at 1000 °C for 6 h. After the treatment the samples were cooled inside the furnace until room temperature. Niobizing treatment parameters were selected from previous experimental work in order to obtain...
the best results of coating thickness. For treatment temperatures and times below 1000 °C and 6 h, respectively, the coating thickness and morphology were thin and irregular.

### 2.2. Characterization

X-ray diffractometry (XRD) was used to identify phases on the surface layer with Cu-Kα radiation in the 2θ range of 25°–120°. The samples were cut along the cross section, polished with alumina suspension and etched with 3% Nital solution to evaluate the morphology and thickness of the surface layer by scanning electron microscopy (SEM). Three Knoop hardness profiles were made to determine the microhardness profile of the boronized AISI 1020 steel. Microhardness measurements were made with 10 gf load and 10 s dwell time. Roughness measurements were obtained via confocal microscopy at 10x magnification with a Gaussian filter and a 0.25 mm cut-off length.

### 2.3. Microabrasive wear tests

The investigation of the tribological performance was carried out using micro-scale abrasion tests in a fixed ball configuration TE-66 SLIM tribometer from Phoenix Tribology. Normal loads of 0.49 N and 0.98 N were used in the experiments with a rotary speed of 70 rpm. The tests were performed using abrasive slurry concentrations of 0.5 g cm⁻³ and 1.0 g cm⁻³ (grams of SiC cm⁻³ of distilled water). The abrasive used in this work had a diameter of approximately 5 μm. Three sets of experiments were performed for each combination of normal load and abrasive slurry concentration. After the niobizing treatment (before microabrasive wear tests) the samples did not undergo any kind of surface preparation. More details about the microabrasive wear tests is given in our previous work [28].

### 3. Results and discussion

#### 3.1. Coating characterization

The XRD pattern for the multicomponent borided AISI 1020 steel is shown in figure 1. Despite the formation of the Fe₂B boride phase that was promoted by the boriding thermochemical treatment (first step in the multicomponent boriding process), the niobizing treatment (second step in the multicomponent boriding process) promoted the formation of the NbB and Nb₅B₆ niobium boride phases. Other authors [5, 18, 30] also report the formation of the Nb₃B₂, NbB₂ and Nb₅B₄ phases, which were not identified in this work.
The Al₂O₃ phase identified in figure 1 comes from the niobization powder and, although it does not take part in the chemical reactions, it can be incorporated to the surface during the treatment and favor the formation of a porous region in the iron/niobium borides interface, as shown in figure 2. This characteristic occurred, similarly, in the work of Mu et al [31] in the boriding of CoCrMo alloys.

The formation region of these pores, according to Jha et al [32], is very close to the original surface of the sample. In substitutional binary alloys it is possible that one atomic species diffuses faster than another. This effect is known as the Kirkendall effect [33]. In the niobization treatment, the Fe diffusion coefficient (outward) is lower than the Nb diffusion coefficient (inward) [34]. This creates a supersaturation of vacancies that favors the formation of pores. The formation of the pores also depends on other factors such as heterogeneous nucleation in particles of impurities [32]. Thus, the presence of Al₂O₃, which was present in the niobization powder and incorporated at the surface, formed preferential sites for the pores formation. In our work the porosity level was estimated by image software analysis at 28% ± 7% in the first 20 μm from the surface of the samples. Below this distance there is no significant level of porosity.

When smaller magnifications are used, it is possible to note the sawtooth morphology of the boride layer (figure 3), which was due to the presence of low amounts of alloying elements [35]. Although there was the formation of the niobium boride coating on the sample surface (figure 3(b)) after the niobizing treatment, there were not significant changes in the iron boride coating morphology (figure 3(a)) besides the increasing in the level of porosity at the Fe₂B surface. Below the iron boride layer there is the presence of proeutectoid ferrite (darker grains) and pearlite (lighter grains).

The niobium boride layer thickness was 2.0 ± 0.5 μm while the iron boride layer thickness reached 158.9 ± 9.5 μm. The niobium boride layer thickness is not in agreement with the work of Sen et al [18] who carried out niobization treatment in pre-borided AISI M2 steel and obtained a layer of niobium borides of 3.25 μm thickness after 4 h at 900 °C, nor with the work of Pazarlioglu et al [3], which obtained 7.19 μm thick niobium borides layers in the niobization of pre-borided AISI 1010 steel at 900 °C for 4 h. Although the authors did not report, there was a porous region below the niobium boride layer. The higher thicknesses for the niobium boride layer obtained by these works may be due to the fact that, after boriding, both the AISI M2 and AISI 1010 steel presented the FeB phase in the iron boride layer. Because it is more saturated with boron atoms, the FeB layer acts as a better donor source of B atoms to the formation of the niobium boride layer that will be deposited on the surface of the material. In the present paper, only Fe₂B phase was identified in the X-ray diffraction (figure 1).
The microhardness profile of boroniobized AISI 1020 steel is shown in figure 4. The hardness of the niobium boride layer was $1360 \pm 200$ HK$_{0.01}$. Hardness measurement of the niobium boride layer was impaired by the presence of the pores, decreasing the expected value for the hardness. Below the niobium boride layer, the Fe$_2$B layer showed an average hardness of $2014 \pm 169$ HK$_{0.01}$. The increase in hardness of the boride layers in relation to the substrate is caused by the formation of the hard boride phases. Other important feature observed in figure 4 is that in the transition region, i.e. in the Fe$_2$B/substrate interface ($151–161$ $\mu$m) there are greater values of the deviation from the mean value. This is a characteristic of the sawtooth morphology obtained after boriding low alloyed steels.

Figure 5 shows the samples surface roughness. The initial roughnesses, before boro-niobizing treatment, were $0.02 \pm 8 \times 10^{-3}$ $\mu$m for the average surface roughness (Ra) and $0.03 \pm 3 \times 10^{-3}$ $\mu$m for the root mean square roughness (Rq). Skewness (Rsk) and kurtosis (Rku) were $-0.87 \pm 0.67$ and $9.30 \pm 3.39$, respectively [28]. After the boro-niobizing treatment, the surface roughness increased to: $0.96 \pm 0.05$ $\mu$m (Ra), $1.42 \pm 0.08$ $\mu$m (Rq), $1.23 \pm 0.20$ (Rsk) and $8.16 \pm 1.12$ (Rku).

Despite the roughness increase in relation to the untreated condition, if compared with the borided condition described in [28], the boro-niobized samples presented statistically the same roughness values. A low roughness variation occurs between these two conditions. This fact indicates that, since niobization is a material deposition treatment, the surface characteristics of the borided samples are maintained during the deposition of the niobium atoms and diffusion of the boron atoms from the substrate to the formation of a superficial layer of niobium borides on the sample surface. The increase in roughness parameters after the boriding thermochemical treatment (before niobization), as found in this work, is in agreement with other works [36, 37].
The values measured for skewness (positive) and kurtosis (>3) are attributed to profiles with many high peaks and low valleys [38]. Figure 4 shows the surface topography of the boroniobized AISI 1020 steel. Many high peaks can be seen in figure 6 (lighter points).

3.2. Tribological behavior

Equation (1) was used to calculate the wear coefficient $k$. In this equation, $k$ is given as a function of the wear crater diameter $b$, the radius of the ball $R$, the sliding distance $S$ and the normal applied load $N$.

$$ k = \frac{\pi b^4}{64 R S N} \quad \text{for} \quad b \ll R \quad \text{(1)} $$

The wear crater depth $h$ was calculated according equation (2), where $b$ is the diameter of the wear crater and $R$ is the radius of the ball.

$$ h = \frac{b^2}{8R} \quad \text{(2)} $$

In order to validate the use of equations (1) and (2), the topographical analysis of the wear craters produced during the microabrasive wear tests of the boroniobized samples were made. As shown in figure 7, a circle of 12.7 mm radius, which is exactly the radius of the AISI 52100 steel ball, was obtained in the cross-section of the wear crater (figure 7(b)).

The steady state regime (SSR) was determined according to the evolution of the wear coefficient ($k$) value (figure 8(a)) and the volume of removed material (VRM) (figure 8(b)) with test duration. In microabrasive wear tests, the SSR is achieved when the wear coefficient values remain constant or when the VRM has a linear relation.
with the increase in the test duration. In this work it was considered that the SSR was achieved when, visually, the wear coefficient remained constant (Figure 8(a)). To validate this assumption, the correlation between VRM and sliding distance was plotted (Figure 8(b)). Linear regression coefficients ($R^2$) near the unit were obtained for all tested conditions, allowing to assume that the SSR was really achieved after a sliding distance of 48 m despite the wear test parameters. Some authors assume that the SSR is achieved when the variation of the wear coefficient do not exceed 5% [39]. The variation of the wear coefficient obtained in this work did not exceeded 7% during the SSR, which is in agreement with other authors [40]. The wear coefficient values shown in Table 1 are the averages of 39 values for each test condition in the SSR.

There is an increase of 63% in the wear coefficient when the abrasive slurry concentration doubled for a constant 0.98 N applied load. For an applied load of 0.49 N, there is a decrease of 45% in the wear coefficient with the increase in the abrasive slurry concentration. If compared with our previous work [28], which were obtained under exactly the same experimental conditions, the boroniobized AISI 1020 steel showed higher values of wear coefficient than untreated and borided samples. For comparison purposes, the highest wear coefficient obtained for untreated AISI 1020 steel samples was $3.59 \times 10^{-3} \pm 8.79 \times 10^{-5}$ mm$^3$ Nm$^{-1}$ [28], which is only approximately 9% higher than the lowest wear coefficient obtained for boroniobized samples. This could be due to the microstructure heterogeneity of boroniobized samples. While the AISI 52100 steel ball slides over an SiC-coated AISI 1020 surface in the untreated condition leading to an artificial reduction of the wear coefficient for this surface condition [28, 41–43], the presence of the porous region in the iron/niobium borides interface in the boroniobized samples may lead to a decrease in the load bearing capacity of the substrate, accelerating the beginning of the wear process through the delamination of the niobium boride layer [44–47]. Indeed, for a 0.98 N applied load and 0.5 g cm$^{-3}$ abrasive slurry concentration (lower $k$ according to Table 1), the

Table 1. Wear ($k$) and linear regression ($R^2$) coefficients for worn boroniobized AISI 1020 steel.

| Load [N] | Slurry concentration [g cm$^{-3}$] | $k \times 10^{-3}$ [mm$^3$ Nm$^{-1}$] | $R^2$ |
|----------|----------------------------------|--------------------------------------|-------|
| 0.49     | 0.5                              | $6.76 \pm 5.7 \times 10^{-4}$       | 0.98  |
| 0.98     | 0.5                              | $3.30 \pm 1.9 \times 10^{-4}$       | 0.96  |
| 0.49     | 1.0                              | $3.73 \pm 1.7 \times 10^{-4}$       | 0.99  |
| 0.98     | 1.0                              | $5.39 \pm 4.8 \times 10^{-4}$       | 0.92  |
wear crater depth obtained from equation (2) ranged from 22.5 to 87.3 μm for a sliding distance variation of 4–96 m. For the condition that showed the higher wear coefficient, 0.49 N applied load and 0.5 g cm⁻³ slurry concentration, the wear crater depth ranged from 32.3 to 84.5 μm for the same sliding distance variation. Thus, in the first 4 m the wear crater depth already exceeded the niobium boride layer thickness and the porous region (as shown in figure 2). The Fe₂B layer, whose thickness was 158.9 ± 9.5 μm, was not completely smeared off after a sliding distance of 96 m. Because the thickness of the niobium boride layer is approximately half the average size of the SiC particles used in this work, they would be able to completely penetrate the layer to the point of causing its delamination and consequent acceleration of the wear process at the initial instants of the wear test [48]. EDS mapping analysis for the abrasive wear condition for the lower wear coefficient (0.98 N and 0.5 g cm⁻³) at the end of the test confirm that the niobium boride layer is completely smeared-off (as shown in figure 9). It can be seen an increase in the presence of oxygen atoms inside the wear crater region, despite the good oxidation resistance of the iron boride phases [49, 50]. There is less amount of oxygen in the sample surface, which indicates that the niobium boride layers can be used as oxidation protection coatings [51].

Adachi and Hutchings [52] proposed a model based on the severity of the contact (Sc) to explain the wear mechanisms that are expected to occur in microabrasive wear tests. According to their model, the Sc is calculated by equation (3) where, W is the normal load, θ is the volume fraction of the abrasive in the slurry, and A is the region over which the separation of the ball and the specimen is less than the diameter of the abrasive particles D. In equation (4), a is the radius of the Hertzian contact area, and R is the radius of the ball. The parameter H’ is calculated according to equation (5), where H₀ is the hardness of the ball and Hᵣ is the hardness of the specimen.

\[
Sc = \frac{W}{A \theta H'}
\]

\[
A = \pi (a^2 + 2RD)
\]

\[
H' = \frac{H_0 + H_r}{2}
\]
The contact severity calculation resulted in values from 0.0007 to 0.0025, depending on the test conditions as shown in Table 2 ($H_b = 9.9$ GPa, $H_s = 2.6$ GPa, $R = 12.7$ mm and $D = 5$ μm). The severity of the contact values are related to the rolling abrasion according to the wear mode map of Adachi and Hutchings \cite{52}. In their work, they used many balls and specimens materials, but the sliding distance remained constant at 16 m.

The occurrence of grooving (when the abrasive particles slide in the contact region) or rolling (when they roll in the ball-specimen contact region) wear mechanisms \cite{52, 53} has a great influence with the sliding distance \cite{54}. Cozza et al \cite{55} used the equation (6) to define the test severity $S_{test}$ and determine the transition between the wear mechanisms in the microabrasive wear test.

$$S_{test} = P \nu$$  \hspace{1cm} (6)

In the equation (6), $P$ is the contact pressure that can be calculated according to equation (7) and $\nu$ is the sliding velocity. In equation (7), $F_n$ is the normal applied load and $A_p$ is the total projected area.
The evolution of the test severity with test duration is shown in figure 10 for the microabrasive wear test conditions used in this work.

![Figure 10](image10.png)

**Figure 10.** Evolution of $S_{test}$ as a function of test duration.

**Figure 11.** SEM images of the center of the wear craters. (a) 0.49 N and 0.5 g cm$^{-3}$, (b) 0.49 N and 1.0 g cm$^{-3}$, (c) 0.98 N and 0.5 g cm$^{-3}$ and, (d) 0.98 N and 1.0 g cm$^{-3}$.

\[
P = \frac{F_a}{A_p}
\]  

(7)

The evolution of the test severity with test duration is shown in figure 10 for the microabrasive wear test conditions used in this work.
Test severity decreased with the increase in the sliding distance for all tested conditions, according to figure 10. This happens because the normal applied load $F_n$ remains constant during the test, and the projected area $A_p$ increases, resulting in a decrease in contact pressure $P$. Decreasing contact pressure is responsible for decreasing test severity $S_{test}$. Due to the application of a constant normal load, the contact pressure decreases and consequently the test severity also decreases. This leads to a variation in the wear mechanisms [56] from grooving/sliding to rolling abrasion [57]. Because the abrasive slurry concentration is maintained constant during the test, the reduction in the contact pressure due to the increased wear crater projected area causes the load per abrasive particle to decrease with increasing sliding distance [58, 59]. Therefore, the decrease of the load per abrasive particle favors the occurrence of rolling abrasion [54].

The microabrasive wear mechanisms were experimentally identified by SEM analysis. Figure 11 shows the center of the boroniobiized AISI 1020 wear craters for the test conditions used in this work. The presence of indentations, which is a rolling abrasion characteristic, can be noted at the surface of the wear craters. Scratches and grooves are not observed.

The edges of the wear craters are shown in figure 12. The lighter region (on the right) is referring to the niobium boride layer, while the darker region (on the left) refers to the wear crater. In figure 12(a) porosities and

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**Figure 12.** SEM images of the edges of the wear craters for an abrasive slurry concentration of 1.0 g cm$^{-3}$ at (a) 0.49 N and (b) 0.98 N applied loads.
embedded SiC abrasive particles are observed on the wear crater region. In figure 12(b), in addition to the porosities observed in the wear crater, the occurrence of fractures is detected in the edge of the wear crater. The presence of these porosities would affect the mechanical strength, weakening the niobium boride layer and decreasing the wear resistance, which is in qualitative agreement with Schiﬀmann et al [36].

4. Conclusions

The wear behavior of AISI 1020 steel samples coated with niobium boride layers was investigated using a microabrasive wear apparatus. The main findings indicated the following:

- Niobium boride layer was successfully deposited on AISI 1020 steel samples by multicomponent boriding process;
- Boro-niobized samples presented statistically the same roughness of borided AISI 1020 steel from previous work because niobization is a material deposition technique;
- The formation of a porous region in the niobium/iron boride layers contributed to a lower hardness at the surface in comparison to the internal iron boride layer;
- The porous region are formed due to Kirkendall effect and heterogeneous nucleation in Al₂O₃ particles;
- Owing to the low bearing capacity, the wear resistance of the boro-niobized samples was inferior to the borided and untreated AISI 1020 steel;
- In the first stages of the tests the niobium boride layer are completely smeared-off by fracture mechanism;
- Rolling abrasion was the wear mechanism observed for all tested conditions owing to the evolution in the wear test sliding distance and decrease of the contact pressure.

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