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# Evaluation of mechanical wear in bismuth-titanium coatings synthesized by the sol-gel method and applied on 316 L stainless steel substrates

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**Abstract.** Steels are part of the basic equipment of most of the industrial framework, including very diverse sectors such as the oil, chemical and automotive industries. However, steels are susceptible to wear phenomena limiting their life in use and losing efficiency in the applications to which it is intended leading to a technological and economic problem. In mechanical systems, lubrication requires effective strategies to ensure that the increase in critical contact voltages does not cause material failure during operation. Although there are different ways to avoid the wear of steel parts, the development of material technology has allowed the manufacture of new alloys with anti-wear properties. Replacing steels with other higher cost materials is very unfeasible. For this reason, one of the alternatives of greatest interest to reduce wear is based on the surface modification of the metal through the use of coatings. The sol-gel method allows the manufacture of coatings on steel parts to reduce wear. The objective of this work was to manufacture, using sol-gel, bismuth-titanium coatings on 316 L stainless steel substrates in order to analyze the mechanical wear of the system and evaluate its wear rates and friction coefficients. The study focused on determining the effect of precursor concentration on performance as a film to reduce wear. It was determined that the friction coefficients show significant variations due to adhesive wear processes. With respect to wear, it is concluded that the coatings offer substrate protection by indicating wear rates lower than those reported for 316 L stainless steel substrates without coating.

## 1. Introduction

316 L stainless steels (316 Lss) are characterized by good resistance to corrosion, malleability, weldability, and biocompatibility. These properties make it a functional and versatile material in the chemical, naval, petrochemical, pharmaceutical, food and biomedical industries [1,2]. However, under certain environmental conditions, the anticorrosive properties can be reduced considerably, generating material degradation or failure [3-6]. For example, this stainless steel tends to form pitting in environments with the presence of industry, which generate chloride ions $Cl^-$ or sulfide ions $S^{2-}$ with non-constant concentrations due to different factors of contamination [7]. To improve the surface properties of 316 Lss, metal oxide coatings are applied using various deposition techniques, including the sol-gel methodology [8]. The sol-gel method is a chemical synthesis method initially used for the

preparation of inorganic materials [9,10]. This process defines conformation of ceramic materials from routes of chemical polymerization of components in liquid state, sol, at environment. The route allows the economic and efficient production of coatings. It is characterized by the use of relatively simple equipment allowing to deposit coatings with different compositions, designed chemical properties and good adhesion on metal surfaces [11-13].

Specifically, 316 Lss widely used in surgical implantology, due to its low cost compared to other materials such as titanium. Due to the corrosive susceptibility in contact with solutions containing chloride ions, it is considered a public health problem [14,15].

Although there are different ways to prevent the wear of steel, the development of material technology has allowed the manufacture of new alloys with specific properties. Replacing steels used massively in many applications with other higher cost materials is very unfeasible. For this reason, one of the alternatives of greatest interest to reduce damage by using coatings [15].

Currently, research is being done on the benefits that materials such as bismuth and its alloys can offer, because a multitude of applications has been found in the technology due to its low melting point. As their melting points are very low, they are used in special welding and melting parts of automatic sprinklers. Bismuth has been of growing commercial importance, is not considered toxic and presents a minimal threat to the environment [16].

In reducing the wear of mechanical systems, lubrication requires effective strategies to ensure that the increase in critical contact stresses does not cause material failure during operation. In addition, ecotoxicological considerations are increasingly important. Pre-conditioning involves the application of ecologically sustainable additives, based on bismuth to generate chemically reactive tribofilms by using a formulation with a high concentration of additive [17].

In the automotive and grease industries for lubrication, bismuth compounds such as bismuth octanoate or bismuth naphthenate have had great market penetration. In particular, the introduction and development of bismuth naphthenate, as a replacement for lead naphthenate as an additive in extreme pressure performance [18,19].

In this work, the results obtained from the deposition of protective films on 316Lss surfaces are presented. The films were synthesized by the sol-gel method varying concentrations of the precursors bismuth-titanium (Bi/Ti) [Bi/Ti: 20/80], [Bi/Ti: 40/60], [Bi/Ti: 50/50], [Bi/Ti: 60/40] and [Bi Ti: 80/20]. And deposited by the spin-coating technique at 1500 rpm and 4000 rpm. The films characterized by the study of their mechanical properties.

## 2. Materials and methods

The precursors used in this study were titanium (IV) butoxide - $Ti (OBu)_4$ (Aldrich, 98%) and bismuth nitrate (III) pentahydrate - $Bi (NO_3)_3 5H_2O$ (Alfa Aesar, 98%). As solvents 2-ethoxyethanol (Aldrich, 99%) and glacial acetic acid (Aldrich, 99.7%). And as a complexing ethanolamine (Aldrich, 98%), the systems studied were [Bi/Ti: 20/80], [Bi/Ti: 40/60], [Bi/Ti: 50/50], [Bi/Ti: 60/40] and [Bi/Ti: 80/20].

The methodology of conformation of stable sols is detailed in previous investigations [20,21]. The films were formed by spin-coating at a speed of 1500 rpm on substrates of 316Lss of dimensions 2 cm x 2 cm x 0.4 cm.

The sintering of the films was carried out at a heating rate of 1° C/min allowing the controlled elimination of the organic components present in the films. The thermal process is established from the initial temperature of 25 ºC to 300 ºC and is stabilized at this temperature for one hour, later, heating is resumed up to 400 ºC and for half an hour it is balanced.

The wear test was developed under the ASTM G-99 standard [22], the equipment used was a CETR-UMT-2-110 tribometer, using a ball of alumina ($Al_3O_2$) of radius 3 mm, with a load of 400 g, a speed of 689 rev/s, varying the travelled distances of 1310 m in a track radius of 5.5 mm.

To test the adhesion of films deposited on 316Lss, the adhesion test was developed with the CSM revetest xpress scratch tester. The applied load increases progressively from 0.1 N to 20 N at a scratch length of 8 mm, a Rockwell indenter C 200 µm radius, the scratching speed was 10 mm/min at a load rate of 100 N/min.

## 3. Results and discussion

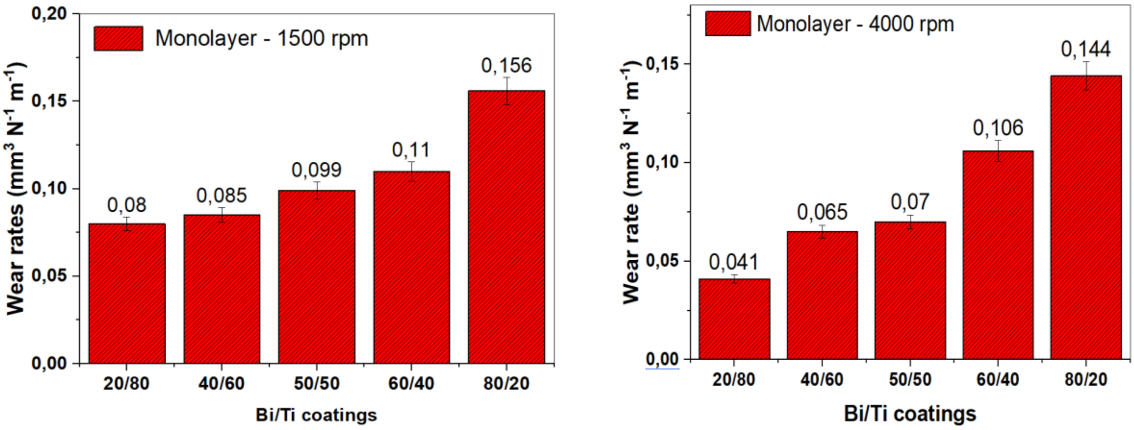
### *3.1. Wear*

Figure 1 shows the wear rates, with their standard deviation, of the coatings of the Bi/Ti system as a function of the molar concentration of the sun and the spin speeds.

The comparison between monolayers 1500 rpm according to the five concentrations studied is indicated in Figure 1(a). In this figure, it is observed for monolayer coatings that high concentrations of titanium tetra butoxide (TBT) precursor of Ti provides coatings with low wear rates.

In Figure 1(b) the values of the wear rates for coatings in monolayers formed at 4000 rpm are compared, varying the concentrations of the precursors of which the films were formed. It is evident that high concentrations of bismuth nitrate precursor of Bi provide coatings with high wear rates.

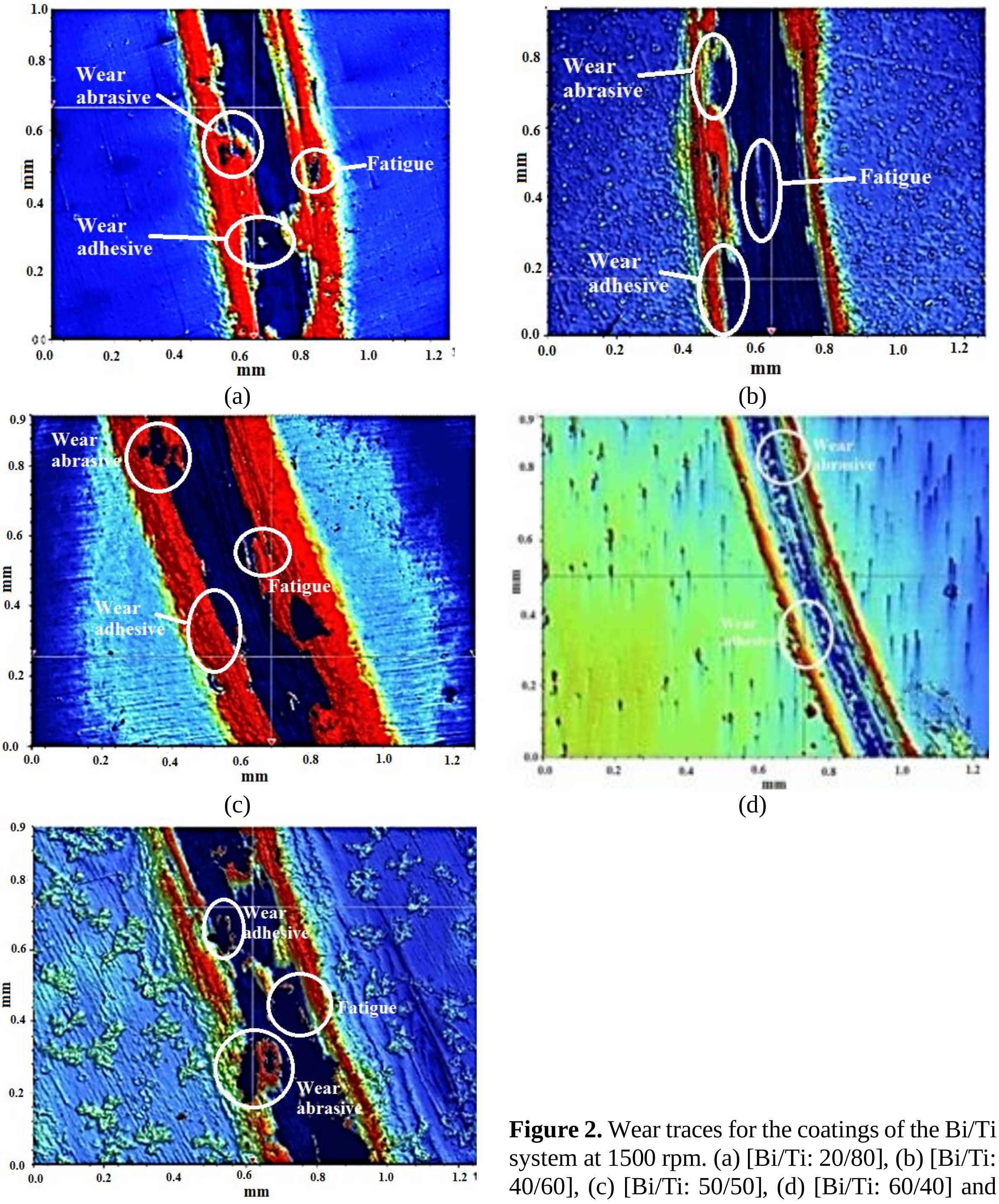
From the study, it is evident that monolayer coatings at speeds of 4000 rpm show the best results in terms of low wear rates. The wear rate for the 316 Lss substrate is 0.33 mm$^3$ N$^{-1}$ m$^{-1}$. Comparing with the results obtained for the five concentrations varying the centrifugation speeds, it is concluded that all the coatings of the Bi/Ti system offer protection to the substrate by indicating wear rates lower than that reported for 316 Lss.



(a) (b)

**Figure 1.** Wear rates for Bi/Ti system coatings. (a) 1500 rpm. (b) 4000 rpm.

Figure 2 show the two-dimensional reconstruction of the wear traces for the coatings of the Bi/Ti system as a function of the molar concentration of the sol. According to the results, wear particles distributed around the tracks are observed. The particles sometimes showed a very uniform distribution on both sides of the footprint, while at other times they did not, accumulating more to one side than to the other. In Figure 2 shows the micrographs of the wear tracks generated in the ball on disk test, for the coatings of the Bi/Ti system at 1500 rpm. Types of wear mechanisms such as abrasive, adhesive and fatigue are evident. Abrasive wear was caused by the annihilation of the rough edges by the continuous passage of the alumina ball producing abrasive particles that deform and harden plastically during the development of the tribological test. These particles adhere to the surfaces of the ball and the coating producing plow grooves on the surface of the films. The adhesive wear is possibly due to the resistance offered by certain areas of the coating to the external load exerted by the alumina ball. For all the coatings studied, abrasive wear is observed as a predominant wear mechanism because a plow groove in the coating is observed (Figure 2) due to its low hardness, high roughness and low resistance to plastic deformation. As a consequence, a large number of particles are produced that deform the surface in the form of "scratches.



(a) (b)

(c) (d)

(e)

**Figure 2.** Wear traces for the coatings of the Bi/Ti system at 1500 rpm. (a) [Bi/Ti: 20/80], (b) [Bi/Ti: 40/60], (c) [Bi/Ti: 50/50], (d) [Bi/Ti: 60/40] and (e) [Bi/Ti: 80/20].

### *3.2. Coefficient of friction*

Figure 3 shows the graphs of the coefficient of friction of coatings of the Bi/Ti system in its five molar concentrations, varying the speed of production. The values of the coefficients of friction (COF) were estimated between the contact surfaces: coating - sphere of alumina. The coefficient of friction curves for the films of the concentration [Bi/Ti: 20/80] at centrifugation speeds of 1500 rpm and 4000 rpm is shown in Figure 3(a). It can be seen that the coefficient of friction of the monolayer film at 1500 rpm takes COF values between 0.13 and 0.15 and remains constant until the end of the test. For the monolayers at 4000 rpm, it is evident that the COF adopts values between 0.16 and 0.18 during the first 25 s of the test. During the last 30 seconds of the test, the COF value is increased, oscillating between 0.18 and 0.45, due to the existence of debris removed by the wear in the own processes of the adhesive wear.

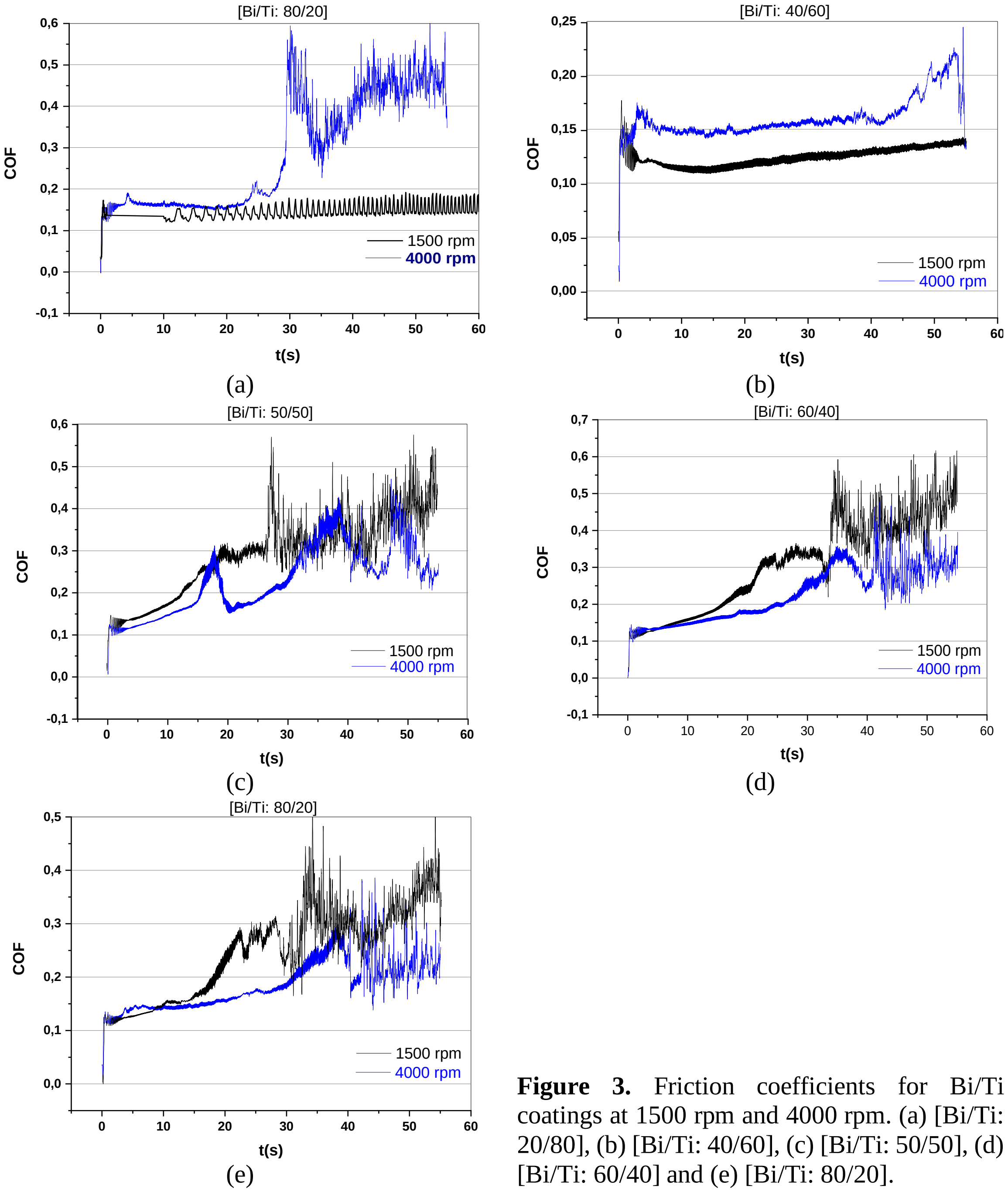


**Figure 3.** Friction coefficients for Bi/Ti coatings at 1500 rpm and 4000 rpm. (a) [Bi/Ti: 20/80], (b) [Bi/Ti: 40/60], (c) [Bi/Ti: 50/50], (d) [Bi/Ti: 60/40] and (e) [Bi/Ti: 80/20].

Figure 3(b) shows the records of the coefficients of friction for the coatings of the molar concentration [Bi/Ti: 40/60]. For the monolayer at 1500 rpm, a stable behavior is observed during the entire test time, the COF value oscillates between 0.12 and 0.14. The monolayer graph at 4000 rpm shows a fluctuation in the first seconds of the test and then stabilizes until 45 seconds. In this segment, the COF adopts values between 0.15 and 0.16. In the last 10 seconds of the test, the COF takes values of 0.2 on average.

The determination of the COF for films in concentration [Bi/Ti: 50/50] are indicated in Figure 3(c). According to the records, it can be shown that the average value of the COF is 0.15 and no effect of the centrifugation speed with which the films were formed is revealed. Regarding the monolayers, it is possible to affirm that the behaviors of the graphs, during the time that the test took, do not show greater stability. It is possible to consider a COF value of 0.13 to 2.0 for the film at 4000 rpm and 0.14 to 2.0 for the coating at 1500 rpm, during the first 15 seconds of testing. In the remaining 40 seconds, the COF values reach 0.35 in both cases, with a rather irregular graphic behavior, Figure 3(c).

Continuing with the characterization, Figure 3(d) shows the graphs of the COF for the films obtained from the concentration [Bi/Ti: 60/40] as a function of the centrifugation speeds. It is evident that there is no influence marked by the centrifugation speed with which the films were made. During the first 25 seconds of the test, the monolayer at 1500 rpm varies the COF value between 0.13 to 0.25; in this time interval, the COF for the monolayer at 4000 rpm takes values between 0.13 to 0.30. In the last 30 seconds that the test takes, it is observed, for the two types of coatings, that the graphs of the coefficients of friction describe a quite erratic behavior and therefore the values of the COF increase considerably.

Finally, Figure 3(e) indicates the graphs of the coefficients of friction as a function of the test time for monolayer coatings obtained at speeds of 1500 rpm and 4000 rpm for a coating [Bi/Ti: 80/20]. These speeds were selected in the process of forming the films using the spin-coating technique. For monolayers at 4000 rpm, a constant COF value between 0.14 and 0.18 is observed for the first 30 seconds of the test. For the last 25 seconds of the test, erratic behavior of the graph is observed, the estimated value of the COF is 0.25 on average. As for the monolayer at 1500 rpm, the graph shows a COF between 0.13 to 0.20 in the initial 20 seconds of testing. In the final 35 seconds, the test takes, the graph shows that the COF value ranges between 0.20 and 0.4.

## 4. Conclusions

Comparing with the wear results obtained for the five concentrations by varying the spin speeds, it is concluded that all coatings of the Bi/Ti system offer protection to the substrate by indicating wear rates lower than that reported for 316 Lss.

From the study of the coefficient of friction for the films obtained from the Bi/Ti system by varying the spin speeds, it can be concluded that the abrupt variations of the friction coefficients may correspond to the existence of debris removed by the wear due to the processes of adhesive wear. Also, by the processes related to adhesive wear and film release. The very large fluctuations that appear in the graphs, possibly due to the appearance of wear particles that generate the abrupt increase in the friction force between the surfaces in contact and when they are expelled from the contact, the friction force decreases. In the tests, no wear of the alumina ball was observed.

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