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A Study of the Wear Damage of a PTFE Coating: The Effects of Temperature and Environment on its Mechanical and Tribological Properties

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Abstract

In this study, a methodology was set up to obtain a measure of the durability of PTFE coatings. Tribological tests to study the effect of both temperature (from 25 °C to 180 °C) and environment (dry, in water, and in oil) on the mechanical and tribological properties of PTFE were carried out. Interactions between the effects of temperature and environment were also investigated. As expected, the experimental results showed that the wear resistance of PTFE significantly decreases with an increase in temperature. Measurements of the mechanical properties of the coatings by nanoindentation at different temperatures confirmed a change in the mechanical properties of the PTFE coatings with increasing temperature. The tribological properties were evaluated through the evolution of the coefficient of friction as a function of temperature and the morphological study of wear debris by scanning electron microscopy (SEM). In addition, the coefficient of friction exhibits no significant difference as a function of the environment. Examination of transfer film formed under various conditions helped us understand the various wear mechanisms occurring during these tests and indicated that the formation of an adhered and persistent transfer film was directly linked to the wear performance of the PTFE coating. In particular, higher wear observed in the oil environment could be attributed to a different tribological behavior of the transfer film on the abrasive counterface, namely poor adhesion, resulting in an ease of removal and replenishment with successive rubbing cycles.

Keywords

PTFE coatings. Temperature. Environment. Durability. Mechanical properties.

1. Introduction

Polytetrafluoroethylene (PTFE) is widely used in various industrial applications due to its self-lubricating properties, low coefficient of friction (COF), low surface energy, chemical inertness, and high temperature resistance. However, PTFE exhibits relatively weak mechanical strength and is highly susceptible to wear (particularly in abrasion conditions), which limits its use in certain applications.

Many researchers have focused on studying the tribological performance of bulk PTFE, and much progress has been made in reducing its wear rate with the use of various fillers. On the microscale, a large number of fillers (e.g., glass fibers, carbon fibers, bronze, graphite, several metals and inorganic compounds, and polymers) have been shown to reduce the wear rate of PTFE by up to two orders of magnitude [1–6]. A general hypothesized wear resistance micro-mechanism is the interruption of subsurface crack propagation, which prevents the formation of delamination debris [1–3,7–9]. Very few nanoscale fillers have shown effectiveness in reducing the wear rate of PTFE, outpacing even that provided by the same materials on the microscale. Notably, ultra-low-wear PTFE and alpha-phase alumina composites have been reported to produce wear rates of $\sim 1 \times 10^{-7} \text{ mm}^3/\text{Nm}$ with alumina filler content as low as 1–5 vol% [10–14]. Although the complete mechanism of the four orders of

magnitude wear reduction observed is still not entirely understood, it is hypothesized that this isolated behavior results from a separate nanoscale mechanism followed only by this specific nanofiller [11,14–21]. A more recent study, however, showed that the special alpha-phase alumina that leads to ultralow wear is actually composed of porous 1–10 μm agglomerates that break up during sliding into nanoscale fragments [22]. Carbon-based nanomaterials have also been shown to impart exceptional wear resistance to PTFE composites [23–27].

Nevertheless, there has been little focus on the tribological behavior of PTFE thin films [28]. In order to increase wear resistance but also preserve the desirable properties of virgin PTFE, various PTFE composite coatings have been developed incorporating epoxy [29], nanodiamond and carbon nanoparticles [30,31], colloidal SiO_2 [32], Au [33] or black phosphorus nanoparticles [34]. Furthermore, because of their low surface energy, PTFE coatings suffer from poor adhesion to substrates, which is typically the primary cause of coating failure. To increase adhesion, roughening techniques are commonly used as well as primer coats allowing PTFE to physically lock and chemically bind to the surface. Beckford and Zou first showed that by adding a polydopamine (PDA) adhesive basecoat, the wear life of PTFE coatings was extended by a factor of 500 [35]. These improvements in tribological properties are in fact the result of both the formation of a transfer film on the counterface, which allows the coating to essentially slide against itself, and improved adhesion between the PTFE topcoat and the PDA basecoat, which prevents delamination of the coating. Other studies, carried out by the same authors, aim to combine the strong adhesion between PDA and PTFE with the incorporation of several fillers (e.g., Cu nanoparticles, PDA-coated Cu nanoparticles, and graphite particles), which have been found to successfully enhance the durability of the coatings [36–38].

In all the investigations mentioned above, the number of rubbing cycles before failure (at which the film is sufficiently damaged to produce a sharp increase in the COF) is used as a measure of the durability of the film. In the present work, we set up a methodology to obtain a measure of the durability of PTFE coatings based on the presence of scratches obtained on the surface of the tested sample after each test, rather than on variation of the COF. Tribological tests to study the effect of both temperature (from 25 °C (called room temperature) to 180 °C) and environment (dry, in water, and in oil) on the mechanical and tribological properties of PTFE were carried out. Interactions between the effects of temperature and environment were also investigated. Surface characterization methods were implemented to investigate the worn surfaces and debris and thus understand the wear mechanisms under various conditions.

2. Materials and methods

2.1. Tested materials and samples preparation

The substrate was an Al 3003 alloy (Al 98%, Si 0.23%, Fe 0.56%, Cu 0.05%, Mn 1.1%, Zn 0.04%, Ti 0.02%). The PTFE coating was a **commercial** “Xylan” two-layer coating from Whitford. The substrate was thoroughly degreased and grit-blasted with 60 mesh aluminum oxide to a roughness of 5 microns. A primer coat was deposited using a spray gun and dried at 85 °C. The top layer was then applied by spray coating. Finally, the coating was cured for 15 min at 380 °C. The final coating had an average thickness of 35 μm . The thickness of the primer coat and the top layer were respectively approximately 15 μm and 20 μm .

2.2. Tribological testing

Tribological testing of the samples was performed using a rotating tribometer with reciprocating motion, as shown in *Figure 1*. The frequency was 0.385 Hz, and the rotation angle was 25°. Each test was carried out for 500 rubbing cycles. A load cell was used to measure normal and frictional forces during the test. The average value of the coefficient of friction was automatically calculated and recorded for each cycle using a computer data acquisition system. Tribological tests were conducted at three different temperatures (25 °C, 90 °C, and 180 °C) and for three types of environment (dry, in water, and in oil). For contact when flooded by water or oil, 80 mL of distilled water or 100 mL of a base oil without additives were used to immerse the sample during the test. A heating plate, positioned below the PTFE-coated Al sample, was used for the tests at high temperature.

A multi-asperity counterface was loaded against the flat PTFE coating sample. The counterface used was an aluminum oxide abrasive pad (Trizact 237AA, 3M™) of dimensions 20 mm × 15 mm (the dimension of 15 mm is in the direction of motion) with a micro-replicated pyramidal structure, as shown in the SEM micrographs in *Figure 2*. The three-dimensional pyramids contain micro-graded aluminum oxide particles dispersed in a synthetic resin binder matrix and are coated onto a cloth backing. They measure between 400 and 500 μm and have an 80° tilt angle relative to the sliding direction. The contact surface between the counterface and the tested sample includes approximately 1000 asperities of approximately 65 μm side length. In order to prevent fouling, the tests were interrupted periodically and the counterface was changed every 100 cycles. The center of the abrasive pad was located at 70 mm from the center of rotation, leading to an average stroke length of 30.5 mm and an average sliding speed of 23 mm/s.

Three tests were conducted for each condition of temperature and environment to determine repeatability.

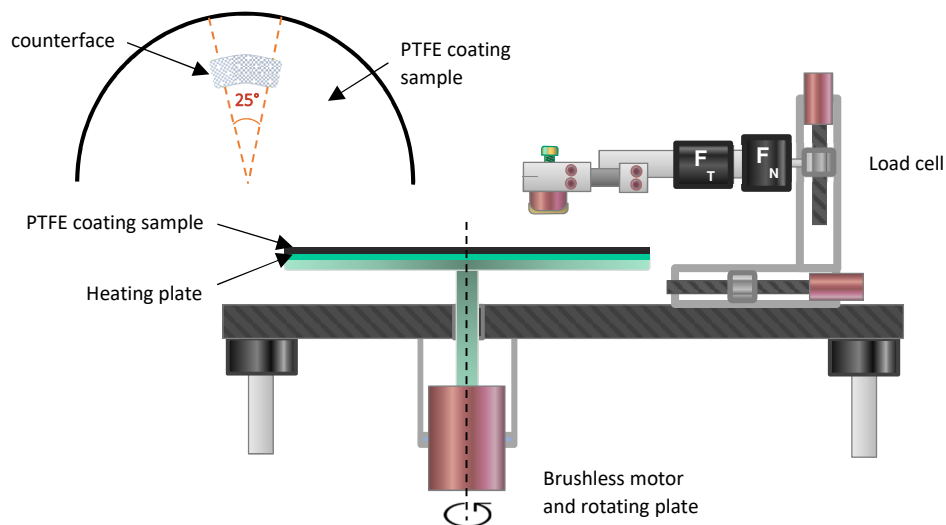


Figure 1: Rotating tribometer used for the tribological testing.

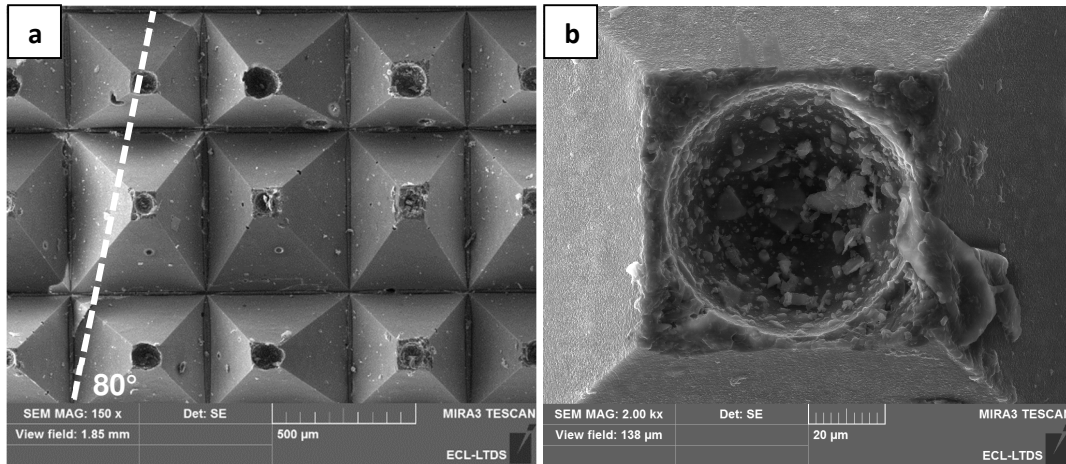


Figure 2: SEM micrographs of (a) micro-replicated pyramidal structure of the multi-asperity aluminum oxide counterface (the pyramids have an 80° tilt angle relative to the sliding direction). (b) top of each pyramid with typical defect.

2.3. Experimental methodology

Each tribological test was performed under a given value of normal load up to 40 N to identify two threshold values: a threshold value below which scratches are never observed on the surface of the sample after 500 rubbing cycles (named “the lower threshold value”) and a threshold value above which scratches are always observed on the surface of the sample after 500 rubbing cycles (named “the upper threshold value”). For each sample, tests at both threshold values were repeated three times to ensure the repeatability of the results. Between the lower and the upper values, due to the heterogeneity of the coating thickness, the behavior was not repeatable, and scratches are observed or not after 500 rubbing cycles depending on the selected location.

This threshold level (*Figure 3*) is used as a representation of the durability of the coating and thus as a means of comparing the tribological properties of PTFE coatings under different temperature and environmental conditions.

A scratch was defined as a continuity of points showing a metallic substrate over $200\ \mu\text{m}$ in length. After each test, the scratches obtained on the surface of the tested sample were first observed with the naked eye and then using a digital microscope.

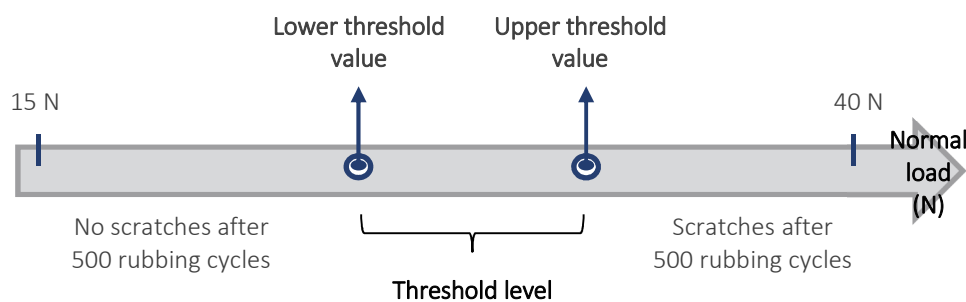


Figure 3: Schematic diagram of the experimental methodology used to compare the tribological properties of PTFE coatings under different temperature and environment conditions.

This experimental methodology allows to compare the wear damage of PTFE coatings based on the presence of scratches obtained on the surface of the tested sample after each test and thus to classify them according to their durability. In addition, under this methodology, the duration of each test is

independent of the durability of the coating tested, whereas, with the conventional method (defining the increase in the coefficient of friction as the durability of the coating), the better the resistance of the coating is, the longer the test is.

2.4. Characterization

A digital microscope (model VHX-1000, Keyence) was used to characterize each sample and counterface. Optical images were captured to qualitatively compare the worn surfaces and thus, to determine the presence or absence of scratches for the tests performed with different values of normal load as well as to identify any film transferred to the counterface after the rubbing tests.

A scanning electron microscope (model MIRA3, TESCAN) was used to characterize the surface morphology and debris on all the counterfaces. Prior to SEM observation, the specimens were Au-coated. In addition, chemical analysis of the counterfaces was performed using EDX.

Nano-indentation tests were performed on the PTFE coating surfaces at room temperature and 90 °C to determine their hardness and elastic modulus. The indentations were carried out using a Berkovich tip with a maximum load of 450 mN. The maximum indentation depth used was less than 10% of the coating thickness to eliminate the substrate effect.

3. Results and discussion

3.1. Tribological behavior at room temperature in air

3.1.1. Evolution of friction coefficient

First, tribological testing of PTFE coatings was conducted at 25 °C in air. *Figure 4* shows the typical variation of coefficient of friction (COF) as a function of rubbing cycles for each of the 100 successive rubbing cycles as well as the average data of these five plots. During the early stages of sliding, there is an obvious running-in period where the COF decreases monotonically with successive rubbing cycles. This might be explained by strong adhesion, formed across the interface, which initiates the formation of a thin and coherent transfer film of PTFE on the counterface, resulting in subsequent contact between PTFE and the transfer film of PTFE and hence easy sliding (*Figure 5*). At the end of each of the 100 rubbing cycles, the COF was approximately 0.24. Comparative plots of the average COF for the three tests are shown in *Figure 6*. All three tests exhibit similar trends in their evolution of COF, indicating that the general tribological features are characteristic of this system.

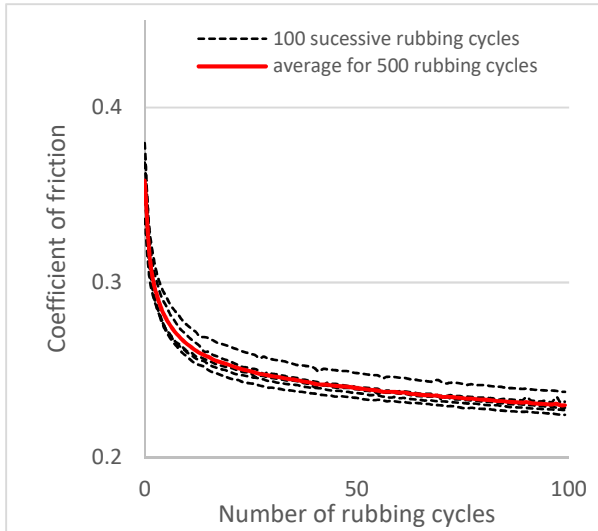


Figure 4: COF vs. rubbing cycles for a PTFE coating rubbed for 500 cycles at 25 °C in air under a 27 N normal load.

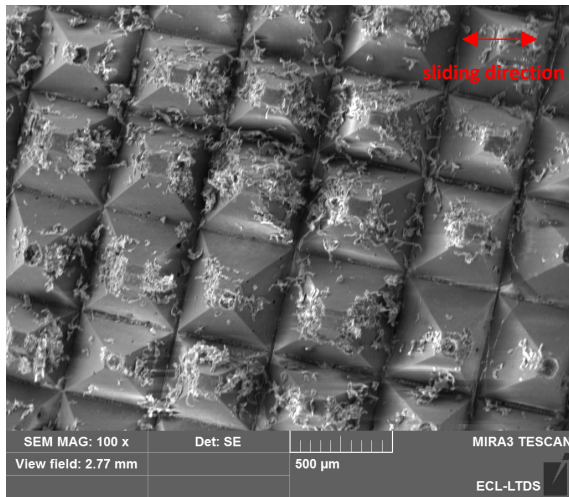


Figure 5: SEM micrograph of the counterface after 100 rubbing cycles for a test conducted at 25 °C in air.

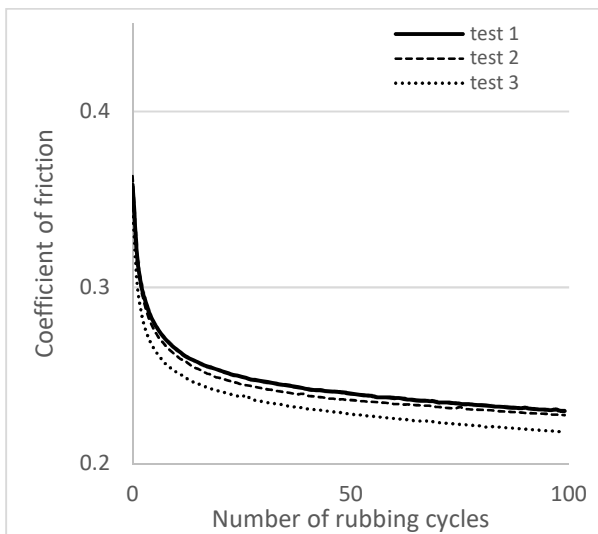


Figure 6: COF vs. rubbing cycles for primary and repeat tests.

Figure 7 shows the variation of the final COF (defined as the average value of the coefficient of friction during the last 15 cycles) with applied normal load and indicates that the COF decreases with

an increase in the normal load within the observed range. Such tribological behavior can be associated with the viscoelastic and temperature-related properties of the polymer, and notably elastic deformation of the surface asperities. A lower value of the coefficient of friction can be explained by an increased adhesion between PTFE and transferred PTFE on the counterface with an increase in the normal load. Bowden and Tabor were the first to describe the friction phenomena by taking into account mechanical contact and adhesion between microscopically rough surfaces [39]. They proposed a model where the apparent friction coefficient μ can be defined as $\mu = \mu_0 + a/F_N$ where F_N is the normal load, μ_0 is the deformation term, and a is the adhesive component of friction. The corresponding curve is shown in *Figure 7*.

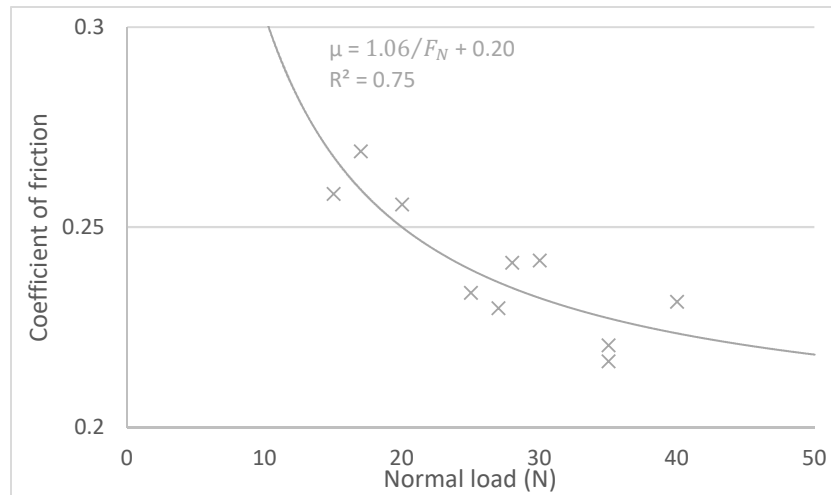


Figure 7: Variation of the final COF with normal load (dry sliding conditions in air, 25 °C).

3.1.2. Damage

For the tests carried out under dry sliding conditions in air at 25 °C, the threshold levels are [27 N; 30 N]. Indeed, as seen in *Figure 8* (a) and (b), no scratches are visible on the surface of the sample after the test was conducted at 27 N, while quite a few scratches are observed after the test was conducted at 30 N.

The variation in the coefficient of friction recorded for both threshold values (*Figure 9*) exhibits no significant difference, and thus there is no correlation between the evolution of coefficient of friction and damage. Indeed, the scratches are observed at very local levels and are only a small part of the overall sliding contact surface. Thus, considering that the counterface is still mostly sliding against the PTFE surface, there is no effect on the coefficient of friction.

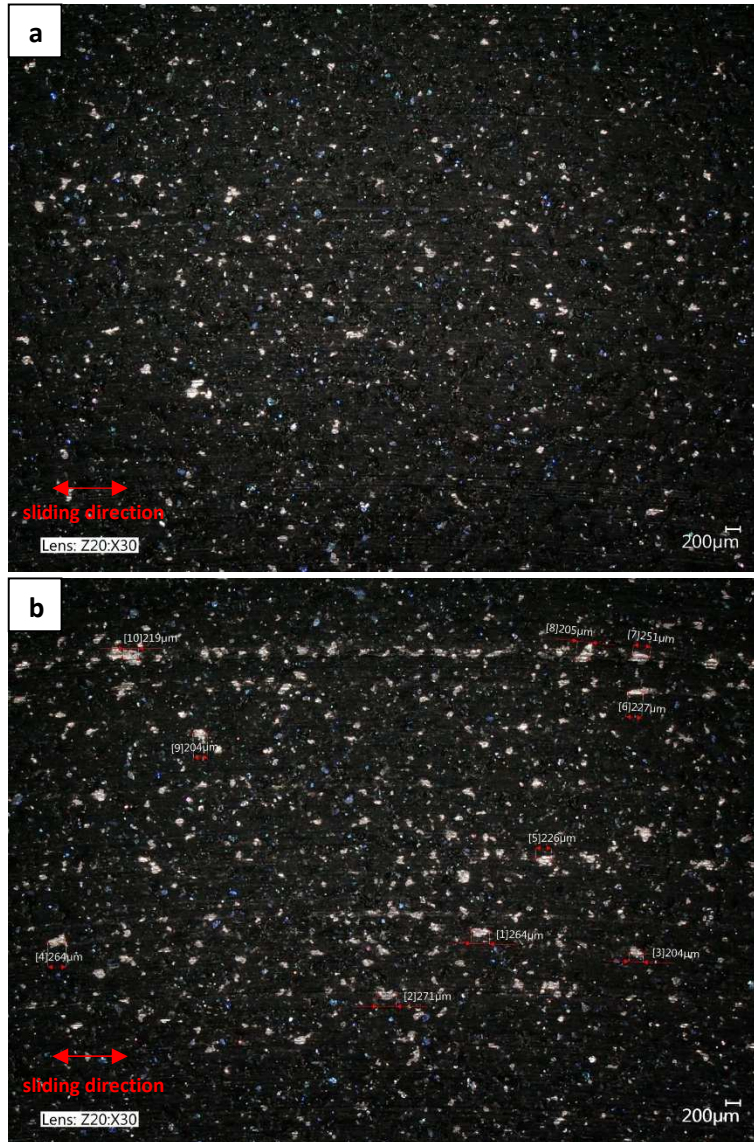


Figure 8: Optical images for a PTFE coating rubbed for 500 cycles at 25 °C in air under its (a) lower threshold value (27 N) and (b) upper threshold value (30 N).

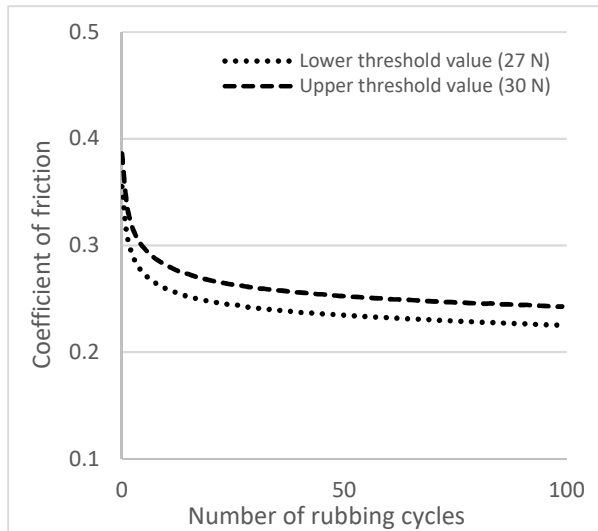


Figure 9: Average COF vs. rubbing cycles for a PTFE coating rubbed for 500 cycles at 25 °C in air under the lower and upper threshold values (27 N and 30 N respectively).

3.2. Effect of temperature

3.2.1. Tribological and mechanical properties

Figure 10 shows the normal load threshold levels determined for PTFE coatings tested at 25 °C, 90 °C, and 180 °C in air. The threshold levels were at first slightly reduced from [27 N; 30 N] to [25 N; 27 N] while increasing the temperature to 90 °C and then dropped significantly to [15 N; 17 N] at 180 °C. These results indicate that the wear resistance of PTFE decreases with an increase in temperature. It is well known that temperature strongly affects the mechanical response of polymers. In particular, the dramatic wear resistance transition observed between room temperature and 180 °C (i.e., division of the threshold level by two) could be explained by the PTFE glass transition temperature of ~120 °C [40].

Nano-indentation measurements (Table 1) confirmed a change in the mechanical properties of the PTFE coatings with increasing temperature. At 90 °C, both the average hardness and reduced elastic modulus were decreased by nearly 50% as compared to that of samples tested at 25 °C (from 30 to 15–20 MPa and from 1.5 to 0.8 GPa, respectively). This may explain the decrease in the threshold from 25 °C to 200 °C.

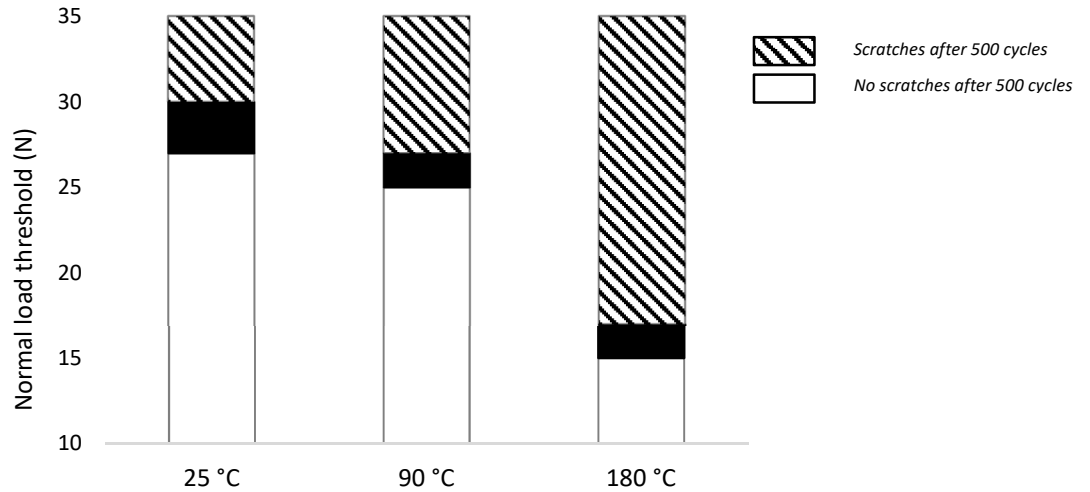


Figure 10: Normal load threshold levels for the PTFE coatings tested under dry sliding conditions in air at 25 °C, 90 °C and 180 °C.

Table 1: Average hardness and reduced Young's modulus of PTFE coatings for nano-indentation tests conducted at 25 °C and 90 °C.

Temperature	Reduced Young's modulus (GPa)	Hardness H (MPa)
25 °C	1.5 ± 0.2	30.0 ± 1.0
90 °C	0.8 ± 0.2	16.5 ± 0.5

Regarding the friction, *Figure 11* shows the variation in the final coefficient of friction as a function of temperature as well as the variation of the friction coefficient calculated according to the model proposed by Bowden and Tabor [39]. This indicates that the COF increases with an increase in temperature because of the plowing effect, which causes roughening of the tested sample surface. It is believed that the plowing effect is more significant at higher temperatures because of a decrease in the mechanical properties of PTFE and a consequent increase in the real contact area. Additionally, the variation of the final COF with applied normal load at both 90 °C and 180 °C follows a trend similar to that observed at 25 °C. As explained above, this decrease in the COF with an increase in the normal load is due to the viscoelastic deformation of PTFE under load and a consequent increase in the adhesive component of friction.

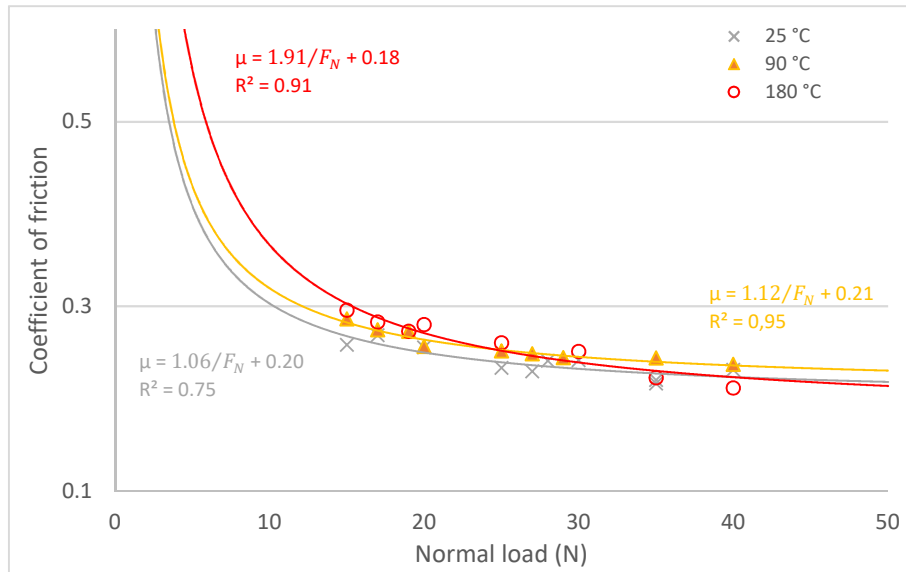


Figure 11: Variation of the final COF with the normal load under dry sliding conditions in air at 25 °C, 90 °C and 180 °C.

3.2.2. Surface morphology

The morphology of the wear debris was also investigated at different temperatures using SEM. The SEM micrographs in *Figure 12* (a) and (b) show the wear debris after 100 cycles for tests conducted at 25 °C and 180 °C, respectively. Wear debris first fills in the defects at the top of the pyramids and then spreads around them. A significant difference in morphology with temperature was observed. While the debris appears short and brittle at room temperature, it appears to be long and ductile at elevated temperatures. These observations confirm a change in the mechanical properties of PTFE with an increase in temperature. Additionally, the chemical composition of the wear debris was analyzed by EDS, and the results show little evidence of silicon only for the debris generated at 180 °C. Silicon is a characteristic component of the primer coat of the PTFE coating sample; the top layer does not contain this chemical element. Thus, it is believed that, at different temperatures, the debris is different as well as the wear mechanism of the PTFE coating. It can be assumed that at room temperature, the coating is worn by removing successive layers. At high temperatures, however, owing to a change in the mechanical properties of PTFE and the plowing effect, the different layers of the coating could be worn simultaneously. Although these complete wear mechanisms are not yet well understood, it can be inferred that they are temperature dependent.

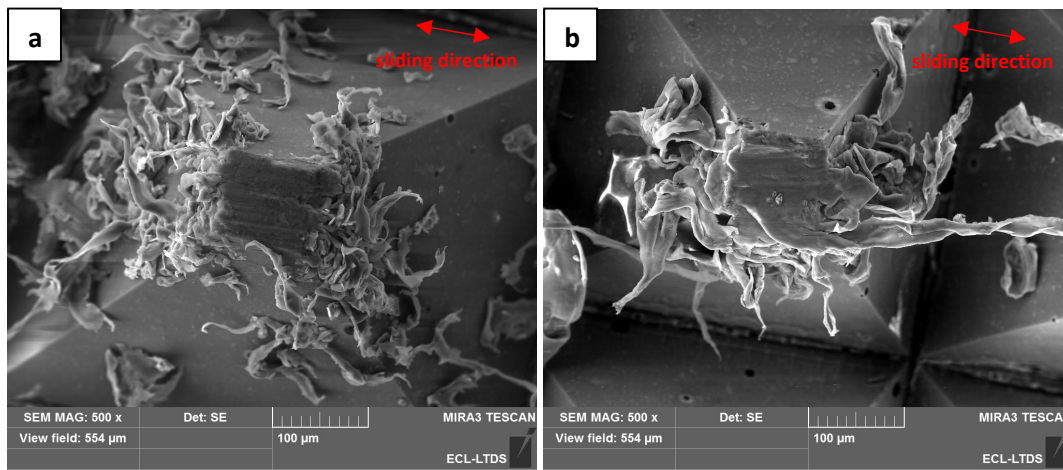


Figure 12: SEM micrographs of wear debris on the counterface for tests conducted (a) 25 °C and (b) 180 °C.

3.3. Effect of environment

3.3.1. Tribological properties

Experiments to study the effect of the environment on the properties of PTFE have also been carried out. *Figure 13* shows the normal load threshold levels determined for PTFE coating tested at 25 °C, 90 °C, and 180 °C for three types of environments (dry, in water, and in oil). For contact flooded by water, the threshold levels determined at both room temperature and 90 °C [26 N; 28 N] are comparable to those obtained under dry conditions in air at 25 °C [27 N; 30 N] and at 90 °C [25 N; 27 N]. For contact flooded by oil, on the contrary, lower threshold levels are obtained, which suggests a lower wear resistance of the PTFE coating and thus no lubricating action of the oil. In addition, contrary to what was found under dry conditions in air, no significant effect of an increase in temperature was observed, and there was very little difference in the threshold levels 25 °C [16 N; 18 N] and at both 90 °C and 180 °C [18 N; 20 N]. The slight reduction of the threshold level at 25 °C might be explained by a change in the physical properties of the oil, notably a higher viscosity. However, optical images of the worn surfaces (*Figure 14*) show a degradation of the tribological and mechanical properties of the PTFE coating with increasing temperature, and the higher the temperature, the larger the sample surface damage.

The variation in the coefficient of friction recorded for the three types of environments at their upper threshold value, as seen in *Figure 15*, exhibits no significant difference as a function of the environment and provides no understanding of the various wear mechanisms occurring during these tests. Regarding the variation of the final coefficient of friction as a function of temperature (*Figure 16*), as was the case for dry sliding conditions, the COF increased with an increase in temperature in both oil and water conditions because of the plowing effect.

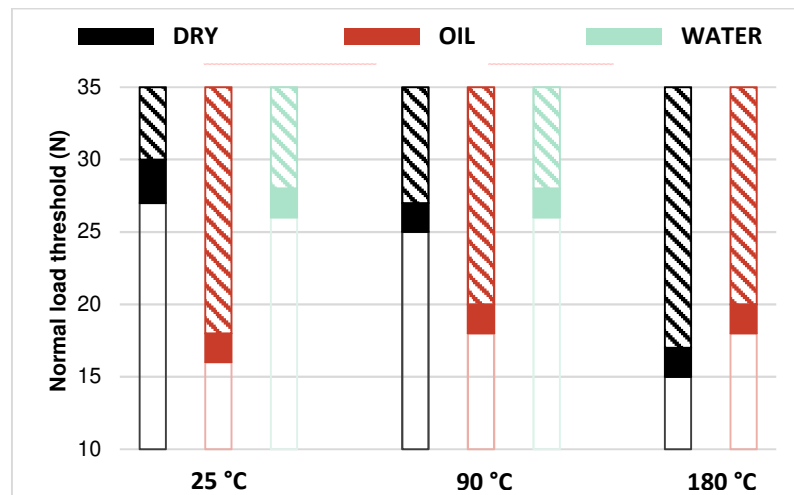


Figure 13: Normal load threshold levels for the PTFE coating tested in dry sliding conditions, in oil and water at 25 °C, 90 °C and 180 °C.

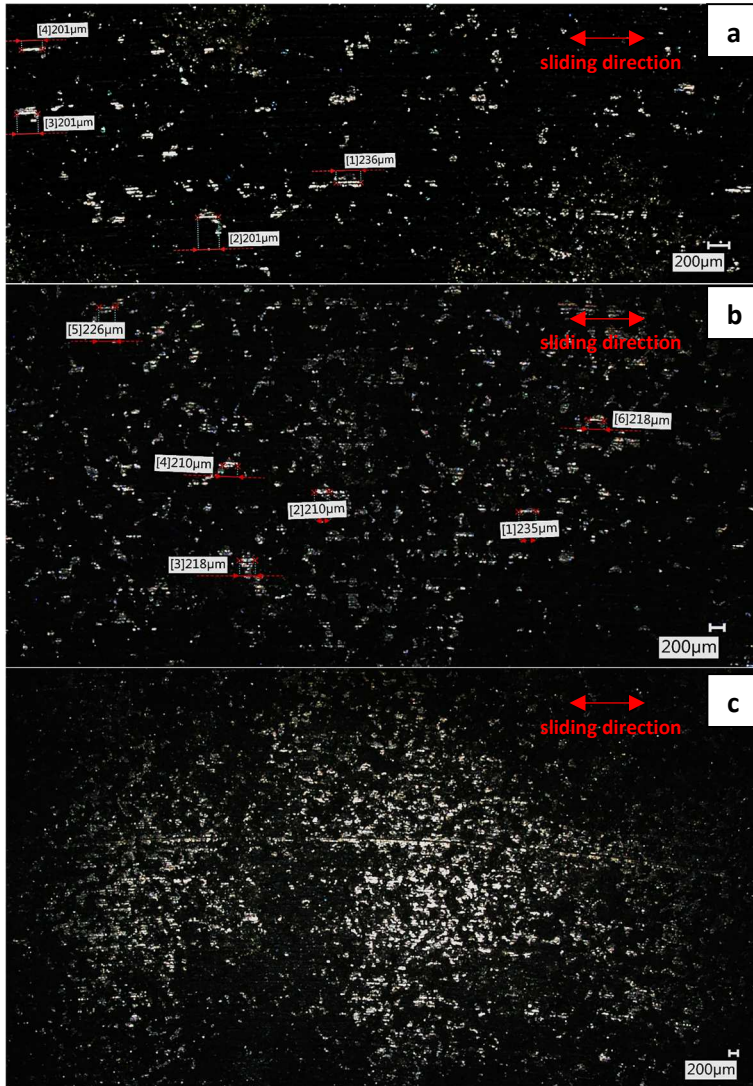


Figure 14: Optical images for a PTFE coating rubbed for 500 cycles in an oil environment at (a) 25 °C, (b) 90 °C and (c) 180 °C under their upper threshold value (18 N, 20 N and 20 N respectively).

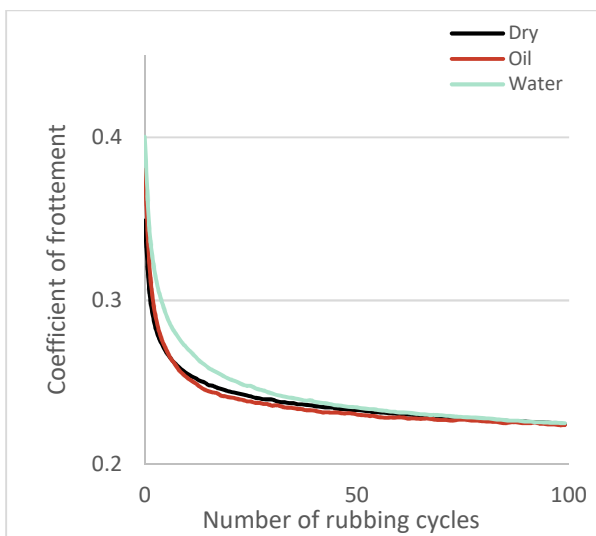


Figure 15: Average COF vs. rubbing cycles for a PTFE coating rubbed for 500 cycles at 25 °C under the upper threshold value (30 N, 18 N and 28 N in dry conditions, in oil and in water respectively).

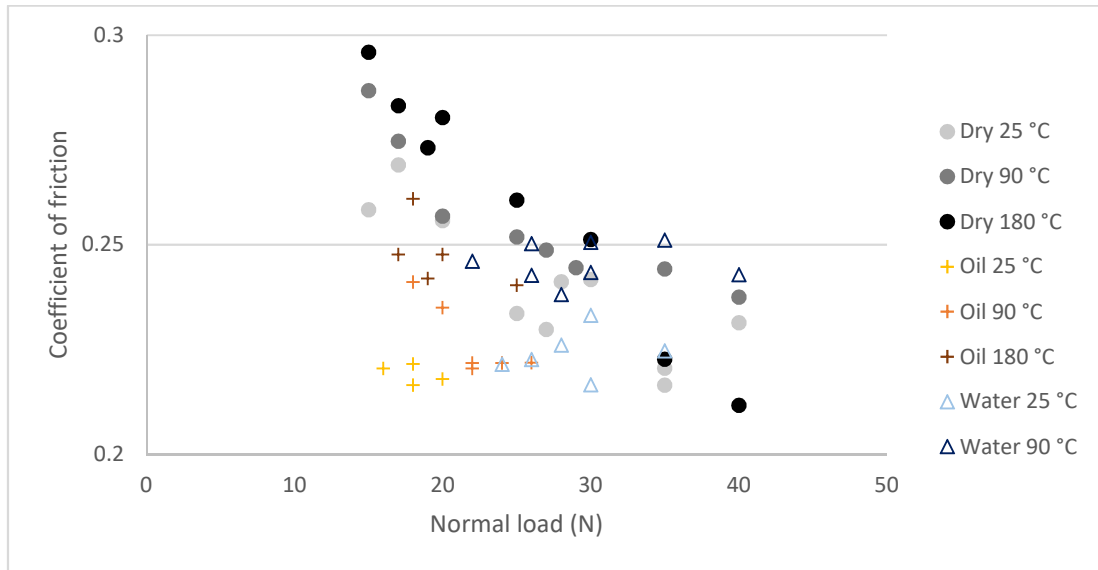


Figure 16: Variation of the final COF with the normal load under various conditions of temperature and environment.

3.3.2. Transfer film

To understand the different tribological behavior observed under the various environments considered, optical images and SEM micrographs of the counterfaces were captured to investigate the transfer film formed and thus understand the wear mechanisms occurring during these tests. On the one hand, both counterfaces obtained after tests conducted under dry conditions (*Figure 17 (a1) and (b1)*) and water (*Figure 17 (a3) and (b3)*) show a large amount of PTFE transfer film. The formation of an adhered and persistent transfer film serves to protect the PTFE sample from wear. On the other hand, the counterface obtained after the test conducted in oil does not show a significant quantity of transferred PTFE, as seen in *Figure 17 (a2) and (b2)*. It is likely that the transfer film, less well-adhered, was removed and replenished with each pass. This different tribological behavior of the PTFE debris on the abrasive counterface is assumed to be the primary cause of the higher wear observed in the oil environment (*Figure 13*).



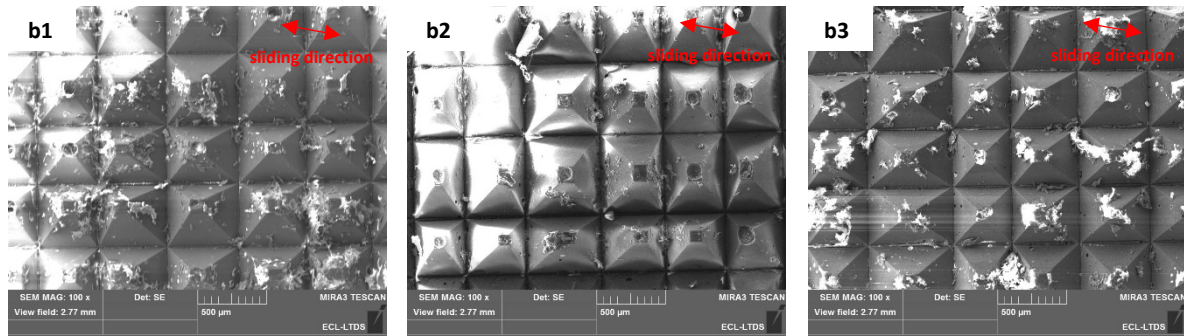


Figure 17: Optical images and SEM micrographs of transfer film for tests conducted in (a1, b1) dry sliding conditions, (a2, b2) an oil and (a3, b3) water at 25 °C under the upper threshold value (30 N, 18 N and 28 N in dry conditions, in oil and in water respectively).

4. Conclusions

In the present work, we set up an experimental methodology to obtain a measure of the durability of PTFE coatings based on the presence of scratches obtained on the surface of the tested sample after each test, rather than on variation of the COF. Under this methodology, the duration of each test is independent of the durability of the coating.

This work presents investigations of the tribological behavior of PTFE under various conditions of temperature and environment. The following conclusions were drawn from the current study:

1. The wear resistance of PTFE decreases with an increase in temperature in relation to a strong decrease in its mechanical properties at high temperatures.
2. No direct correlation between the evolution of coefficients of friction and the presence of scratches was found. No significant difference in the coefficient of friction as a function of the environment was observed.
3. Examination of the transfer film was useful to determine the role of the transfer film in the friction and wear performance of the PTFE coating under various conditions. In particular, the higher wear observed in an oil environment could be attributed to the different tribological behavior of the transfer film on the abrasive counterface, with its ease of formation and regeneration.

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