

Friction and wear of PTFE composites at cryogenic temperatures¹

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Abstract

This paper presents investigations on the tribological behaviour of PTFE composites against steel at cryogenic temperatures. The results showed that the friction coefficient decreases with temperature down to 77 K, but did not follow a linear evolution further down to extreme low temperatures. It can be stated that the cryogenic environment has a significant influence on the tribological performance of the polymer composites. The effect of low temperatures was more clearly detected at low sliding speed, where friction heat is reduced. A change in wear mechanism from adhesive to abrasive was observed in this case. SEM and AFM analyses showed that the PTFE matrix composites investigated under these experimental conditions have transferred material onto the disc down to very low temperatures. Chemical analyses indicate the presence of iron fluorides. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: PTFE composites; Cryogenic tribology; Transfer film

1. Introduction

New technologies lead to a growing interest in material investigations for low temperature applications, in particular polymers and composites as an alternative to metals. Good experiences were made thereby with polymers, particularly with PTFE and PTFE filled composites [1,2]. It is well known that the material properties of polymers are strongly temperature-dependent. Young's modulus and hardness are much higher at low temperatures compared to room temperature [3,4], whereas the already low heat conductivity continues to decrease. A consequence of this is the change in wear mechanism of the polymers at low temperatures. While at ambient temperature adhesive wear dominates, abrasive wear is observed at low temperatures [1,5].

Tribological performances of polymers depend significantly on the temperature at the friction contact. That applies also to very low temperature tribosystems, especially for polymers with low heat conductivity.

More recently, some tribological studies in cryogenic environment [6–9] showed that both friction and wear are reduced due to the change in adhesion and deformation characteristics of the polymer materials. However, it was stated that this trend is not linear down to extreme low temperatures, but strongly depends on the cooling medium [9]. At $T=4.2$ K in LHe, friction and wear increased compared to the values measured at $T=77$ K in LN₂.

Concerning chemical analyses, XPS studies of filled PTFE matrix materials at room temperature [10–12] showed tribochemical reactions between the PTFE and the metal of the counterface as well as between PTFE and metallic fillers. The same results were observed with EDX (Energy Dispersive X-ray analyses) at room temperature in helium gas [13]. Polymer decompositions and triboreactions were also found for PCTFE in LN₂ [5].

The aim of this study is to investigate the tribological behaviour of polymer-composites at low temperatures, with special attention given to the elementary processes at the friction contact. This paper describes experiments carried out in parallel at room temperature and in cryogenic environments and analyses of the polymer transfer occurring during these tribological tests at different temperatures as well as in different media. Transfer films and the surface of the pins were examined, and chemical surface analyses were performed.

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Table 1
Composition of the PTFE matrix composites

PTFE matrix		wt% CF			
		5	10	15	20
wt% Ekonol®	10			X	
	20	X		X	X
wt% bronze	30		X		

2. Experiments

2.1. Materials

The materials investigated here are two types of PTFE matrix composites chosen from experiments carried out at room temperature [14]. Table 1 presents the composition of these PTFE composites. Figure 1 shows the structural formula of the polymers. The processing of these PTFE compounds takes place via a sinter process at a temperature between 360°C and 380°C. The materials were delivered as compression-moulded cylinders, which were cut into pins (surface of contact 4×4 mm). To shorten the running-in period, samples were pre-worn with grinding paper (Grid 800) placed between the pin and the disc prior to the actual tribological tests. With this method the roughness of the pins before testing was always the same, and a better parallelism between the two mating surfaces was achieved. The counterface is a disc of steel 100Cr6, that has been polished ($R_a=0.01 \mu\text{m}$) and cleaned with ethanol before use.

2.2. Tribometers

Tribological tests were carried out with a pin-on-disc configuration in the tribometers CT2 and CT3 described in [15]. Both cryotribometers are thermally insulated by vacuum superinsulation and cooled directly by a bath of liquid cryogen (CT2) or by a heat exchanger (CT3). At room temperature in air (RT), in liquid nitrogen (LN_2 , $T=77 \text{ K}$) and in liquid helium (LHe, $T=4.2 \text{ K}$) the tribometer CT2 was used. The investigations at $T=77 \text{ K}$ in helium gas (He-gas) were carried out in CT3.

The test parameters were:

Normal load: $F_N=50 \text{ N}$ (3.12 MPa)

Sliding speed: $v=1 \text{ m/s}$ and 0.2 m/s

Sliding distance: $s=2000 \text{ m}$

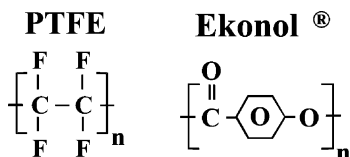


Fig. 1. Structural formula of PTFE and Ekonol®.

During the tribological test, the frictional force was measured to determine the coefficient of friction. In this experiment, the weight loss of the pin could not be measured since the wear was too small after 2000 m. An attempt was made to estimate the linear wear of the composite after this short sliding period by measuring the displacement of the pin, using an inductive sensor in CT2. Measurements were carried out before and after the tests with a load of 50 N and when the temperature of the system has returned to RT. To avoid the measurement of the creep behaviour of the composite, a static experiment was carried out with the same applied load and for the same period of time. The displacements recorded were then deduced from the total displacement measured after the wear tests. Despite all of the precautions undertaken, the linear wear results obtained here should be taken with precaution.

After the experiments transfer films and the surface of the pins were examined with optical and Scanning Electron Microscopy (SEM) as well as with Atom Force Microscopy (AFM). X-ray Photo Electron Spectroscopy (XPS) and Energy Dispersive X-ray analyses (EDX) were performed to examine the surface composition of the transfer film and the chemical reactions between the PTFE matrix material and the steel counterface.

3. Results

3.1. Tribological tests

3.1.1. Friction measurement

Figure 2 shows the coefficient of friction of two PTFE composites at RT in air, at $T=77 \text{ K}$ in He-gas and LN_2 and at $T=4.2 \text{ K}$ in LHe, for the two sliding speeds $v=1 \text{ m/s}$ and $v=0.2 \text{ m/s}$. The graphs clearly demonstrate the influence of temperature but also of cooling properties of the inert environment on the results.

The coefficient of friction of PTFE composites

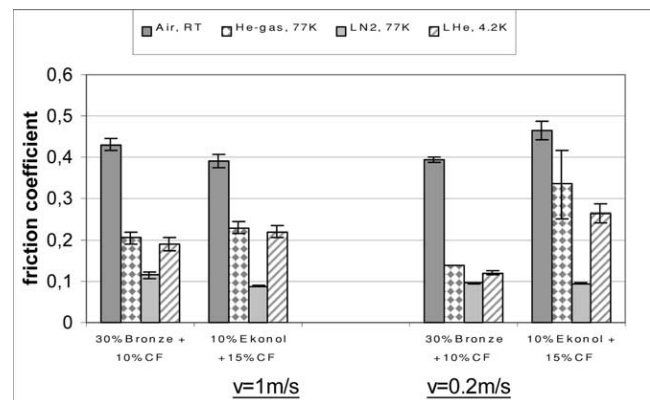


Fig. 2. Coefficient of friction of PTFE matrix composites at RT, at $T=77 \text{ K}$ in He-gas and in LN_2 , and at $T=4.2 \text{ K}$ in LHe ($F_N=50 \text{ N}$; $s=2000 \text{ m}$).

decreases from room temperature to $T=77$ K, with a minimal value in LN_2 . However, this decrease is not linear down to extreme low temperature since at 4.2 K, the friction coefficient increases. In fact, the friction values are the same at $T=77$ K (He-gas) and at $T=4.2$ K (LHe) for both materials, at $v=1$ m/s.

At a lower speed ($v=0.2$ m/s), the friction coefficient differs from the one observed at $v=1$ m/s, depending on the environment but also on the material (Fig. 2). It can be noticed that the coefficient of friction is lower at $T=4.2$ K in LHe than at $T=77$ K in He-gas.

Figure 3 shows the friction coefficient of several PTFE matrix composites at RT and in LN_2 . The parameters chosen in this experiment are $F_N=50$ N and $v=0.2$ m/s. Although the composition of the materials plays an important role at RT, the friction coefficient remains at 0.1 in LN_2 for most of the composites. Whatever the percentage of fillers and fibres, the friction coefficient decreases about 70% to 80% compared to RT values.

3.2. Wear measurement

Figure 4 shows the linear wear of these PTFE matrix composites measured at RT and in LN_2 , with $F_N=50$ N and $v=0.2$ m/s. The graph clearly illustrates the good performance of the polymer composites at low temperatures, with low linear wear. Contrary to the friction coefficient, linear wear depends on the material composition. The material with 30 wt% bronze and 10 wt% CF gives the lowest wear at RT. The materials with 20 wt% Ekonol®, however, have a very small linear wear in LN_2 , quite difficult to detect after 2000 m. (The mass loss calculated from these linear wear measurements would lie between 0.07 mg and 1.2 mg). Since the tests run only 2000 m, the wear rate in this experiment is higher than the one observed in [14] (at RT, after 72000 m).

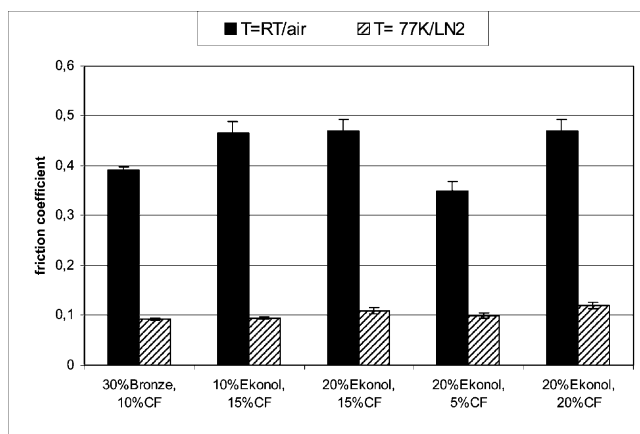


Fig. 3. Coefficient of friction of PTFE matrix composites at RT and in LN_2 ($F_N=50$ N; $v=0.2$ m/s; $s=2000$ m).

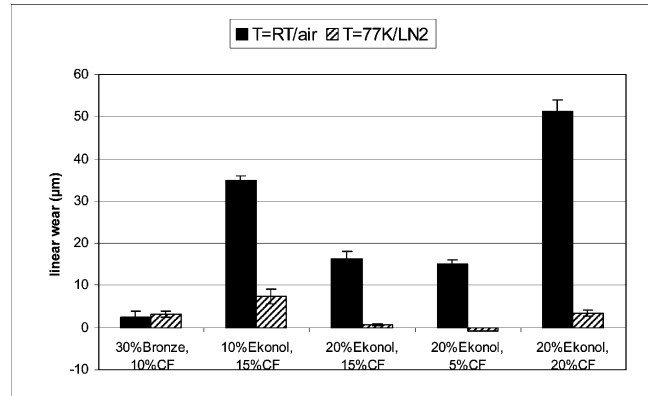


Fig. 4. Linear wear of PTFE matrix composites at RT and in LN_2 ($F_N=50$ N; $v=0.2$ m/s; $s=2000$ m).

3.3. Analyses of the transfer film

3.3.1. Topography

Investigations of the transfer film formed at RT in air, at $T=77$ K in He-gas and in LN_2 , as well as in LHe were carried out.

For $v=1$ m/s, profile measurements and optical microscopy images show the important influence of temperature as well as environment on the topography (Fig. 5). At room temperature in air, the transfer film is clearly visible (Fig. 5(a)). It consists of a homogeneous film, 200 nm to 300 nm thick, with some 0.5 μm thick lumps. At $T=77$ K in He-gas, the transfer is inhomogeneous (Fig. 5(b)). The thin area consists of long PTFE lumps. In the centre, the transfer film is thicker, up to 1.2 μm . The polymer transfer is deformed (Fig. 6(a)). The experiment carried out in LN_2 produces a much thinner transfer (Fig. 5(c)), that consists of scattered polymer particles. The average thickness is 100 nm. The transfer film built up in LHe is thicker compared with the one formed in LN_2 (Fig. 5(d)). The film consists here of broad tracks, which are up to 1.5 μm thick. The transfer film is brittle and shows many small wear debris at the edge as well as in the centre of the film (Fig. 6(b)). Similar topography of the discs were observed for the Ekonol® filled PTFE composites (Fig. 7).

For the experiments carried out at $v=0.2$ m/s, profile measurements (Fig. 8) indicate as well a clear influence of temperature and environment. However, the topography of the disc is quite different from the one at $v=1$ m/s. Significant abrasive scarves are produced in LN_2 as well as in LHe (Fig. 9).

3.3.2. Chemical analyses of the transfer

For a better understanding of the tribological behaviour of the polymer composite, XPS analyses of the transfer film were carried out after the experiments. The objective was to examine possible chemical reactions between the PTFE matrix and the steel in the different cryogenic media. The spectra of the fluorine peak F(1s)

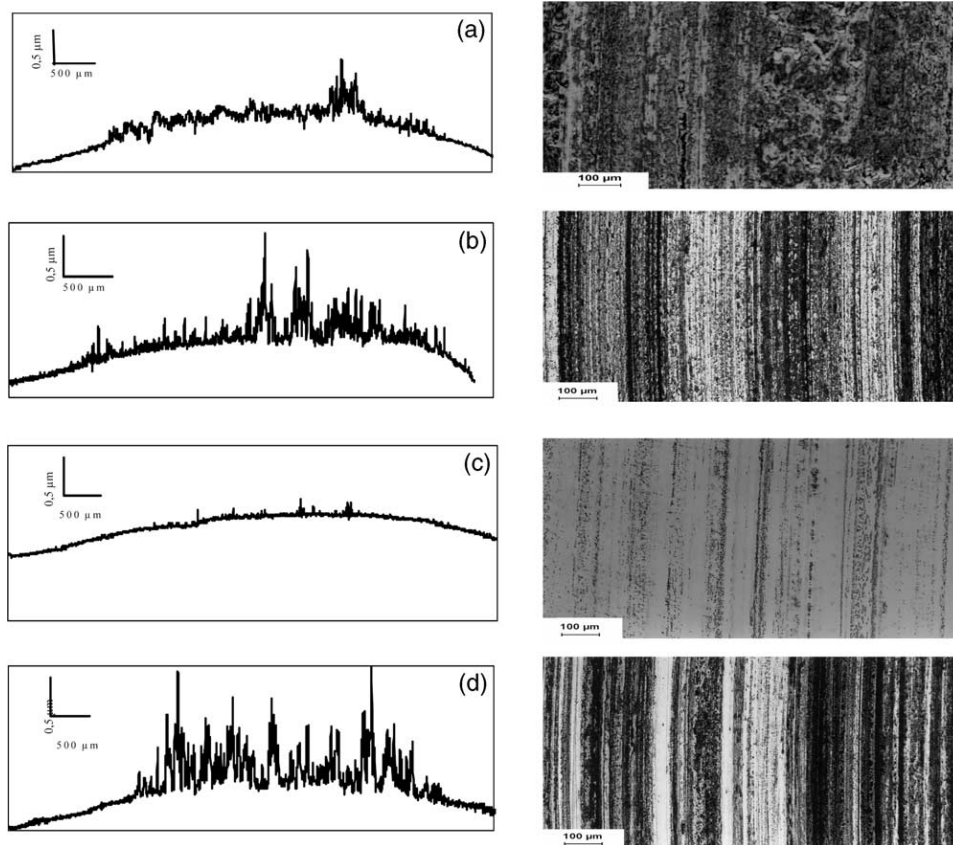


Fig. 5. Profile and optical microscopy images of polymer transfer on the disc at (a) RT in air, (b) $T=77$ K in He-gas, (c) $T=77$ K in LN_2 , (d) $T=4.2$ K in LHe (Bronze and CF filled PTFE against steel; $F_N=50$ N; $v=1.0$ m/s; $s=2000$ m).

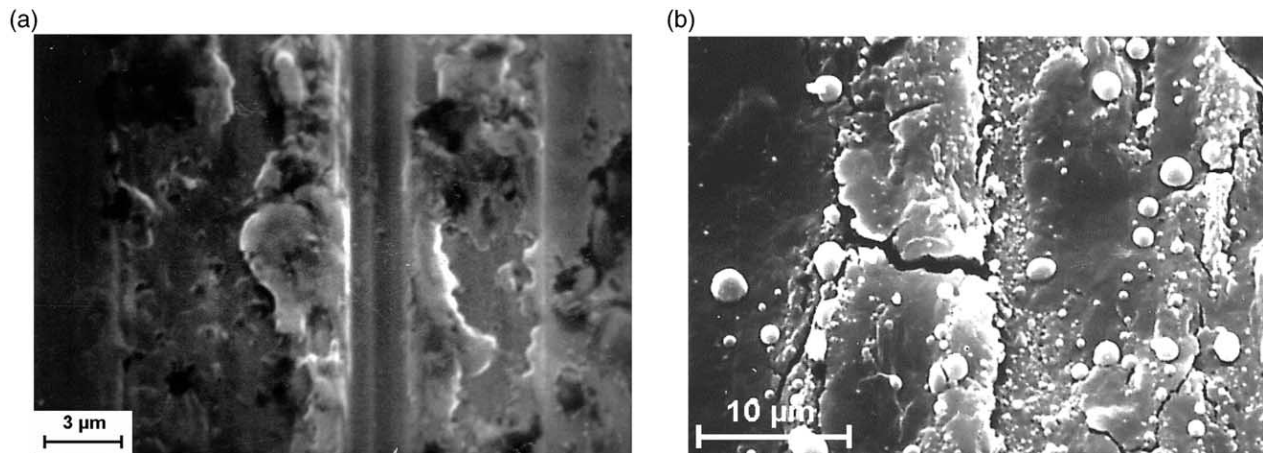


Fig. 6. SEM analysis of the polymer transfer (a) at $T=77$ K in He-gas, (b) at $T=4.2$ K in LHe (Bronze and CF filled PTFE against steel, $F_N=50$ N; $v=1.0$ m/s; $s=2000$ m).

and carbon C(1s) of the polymer material as well as iron Fe(2p) of the counterface were determined at the surface of the transfer (Fig. 10). The F(1s) spectrum of the film grown at room temperature in air, shows two peaks: one at 687.7 eV, characteristic of the fluoride in PTFE, and another at 684 eV, which indicates the presence of a metal fluoride. In the Fe(2p) spectrum two overlapping

peaks were noticed. The peak of the iron fluoride is at the same position as the one of ferric oxide (711 eV). Since iron fluoride (FeF_2) was found by other authors [10,16], it is reasonable to deduce that iron fluoride is also present in our experiment.

Similar results were obtained for the film grown at $T=77$ K in LN_2 . The F(1s) spectrum shows two peaks

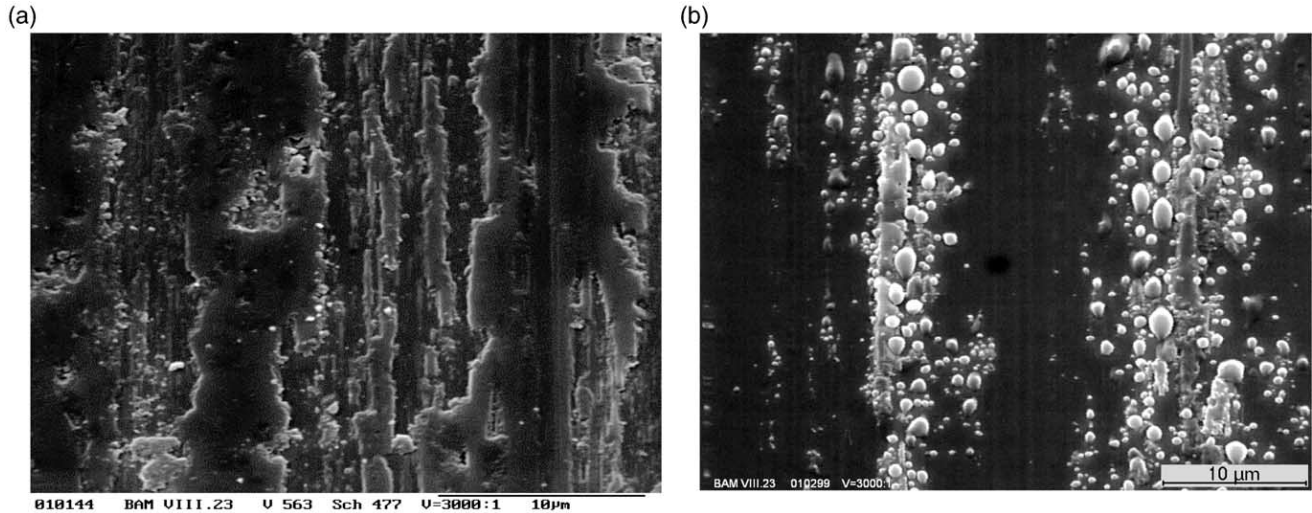


Fig. 7. SEM analysis of the polymer transfer (a) at RT in air, (b) at $T=4.2$ K in LHe (10wt% Ekonol[®] and 15wt% CF filled PTFE against steel, $F_N=50$ N; $v=1.0$ m/s; $s=2000$ m).

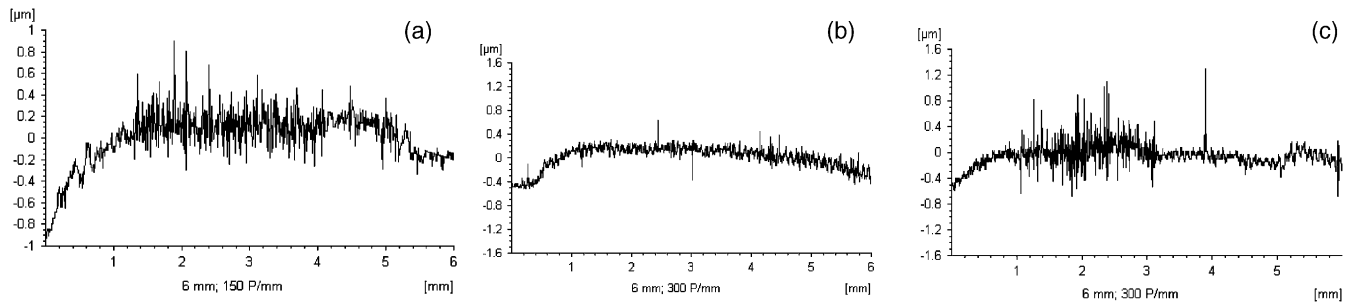


Fig. 8. Laser profilometry of the steel disc after tribological tests against bronze and CF filled PTFE at (a) $T=77$ K in He-gas, (b) $T=77$ K in LN₂ and (c) $T=4.2$ K in LHe. ($F_N=50$ N; $v=0.2$ m/s; $s=2000$ m).

at 688.6 eV (PTFE) and at 684.6 eV (metal fluoride). The Fe(2p) spectrum however shows two peaks at 711 eV and at 714.7 eV, which indicate the presence of FeF₂ or FeF₃ (714.7 eV). As for the XPS analysis of the film formed at $T=4.2$ K, the spectrum of F(1s) shows a larger percentage of the metal fluoride (peak with 684.8 eV) corresponding to the smaller C(1s) peak of PTFE (292.3 eV). The Fe(2p) spectrum has a higher peak at 714.7 eV which suggests the presence of FeF₃.

3.3.3. Surface analysis of the pins

The bronze-filled PTFE composite pin was examined after the experiments. The topography was analysed with SEM and AFM. EDX was used for the analysis of the chemical components. Similar to the transfer film analyses, a significant influence of both the temperature and the cooling properties of the medium on the morphology of the pins was observed. The surface of the pin tested at room temperature is flat, bronze particles are clearly visible (Fig. 11(a)), which corresponds to the observations of other authors [17]. The PTFE matrix, which is softer than the bronze particles, is removed from the surface and forms a smooth transfer film on the coun-

terface. Chemical analyses of the surface indicate the presence of PTFE, bronze and iron. After the experiments at $T=77$ K in He-gas, the surface of the pin is similar to the one formed at room temperature: flat, the bronze particles are uncovered. The SEM images of the sample tested in LN₂ (Fig. 11(b)) show that the bronze particles are partially covered with PTFE lumps, which are pulled out of the matrix. Since at $T=77$ K the hardness of the polymer is much higher than at room temperature, the wear process of the polymer matrix is slowed down. This corresponds with the observations described above regarding the very thin transfer film and the scattered polymer particles on the counterface produced in LN₂. EDX analyses of the pin surface revealed mainly PTFE and bronze elements. The surface of the polymer composite tested at $T=4.2$ K is characterised by some cracks and very small round particles (Fig. 11(c)). These are observed on the surface of the counterface of the bronze filled PTFE composite (Fig. 5(b)) as well as of the Ekonol[®] filled PTFE in LHe.

In the same way as for the bronze filled PTFE composite, surface analyses of the Ekonol[®] filled PTFE composites were carried out. Figure 12 shows the SEM

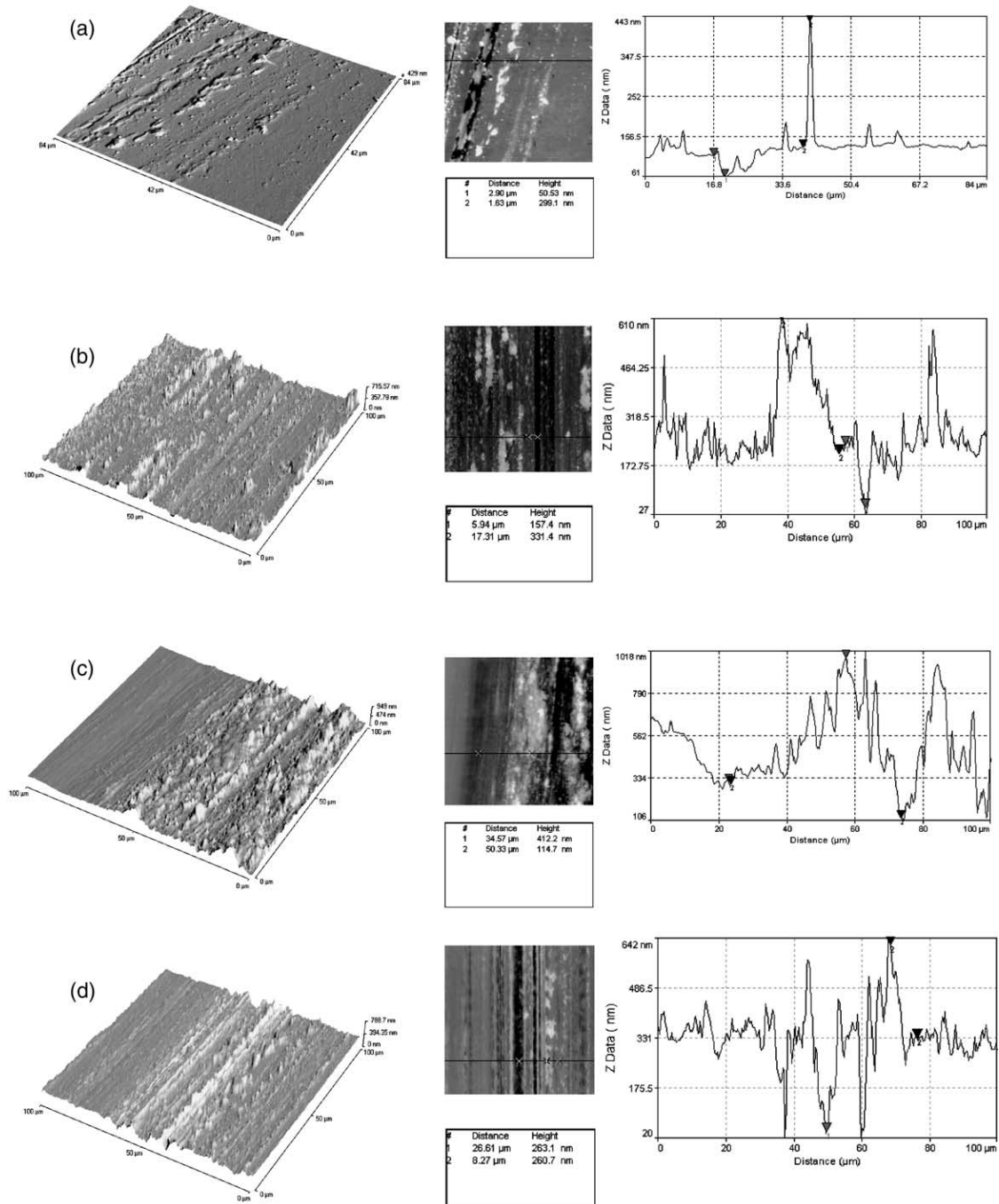


Fig. 9. AFM analysis of the steel disc after tribological tests against bronze and CF filled PTFE ($F_N=50$ N; $s=2000$ m) at: (a) $T=77$ K in LN_2 ; $v=1.0$ m/s, (b) $T=77$ K in LN_2 ; $v=0.2$ m/s; (c) $T=4.2$ K in LHe; $v=1.0$ m/s, (d) $T=4.2$ K in LHe; $v=0.2$ m/s.

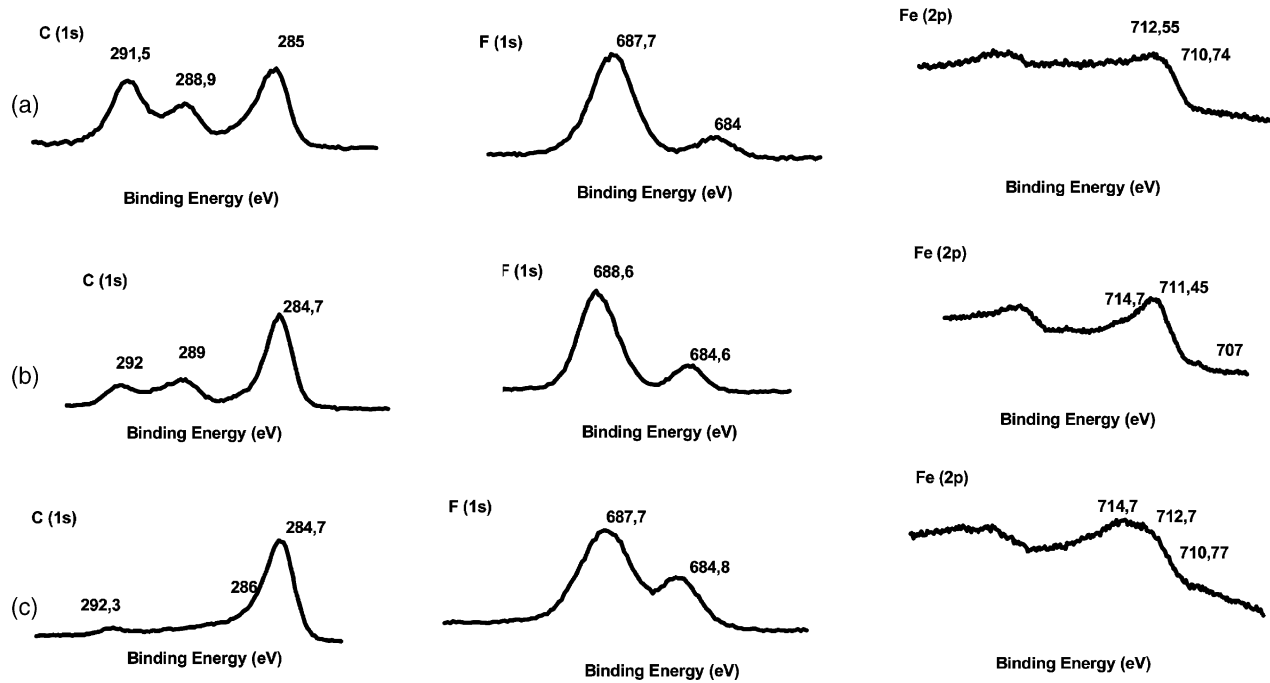


Fig. 10. XPS analysis of the transfer film grown on the disc at: (a) RT in air, (b) $T=77$ K in LN_2 , (c) $T=4.2$ K in LHe. C(1s), F(1s) and Fe(2p) spectra (Bronze and CF filled PTFE against steel disc; $F_N=50$ N; $v=1.0$ m/s; $s=2000$ m).



Fig. 11. SEM analysis of bronze and CF filled PTFE composites after tribological tests at (a) RT in air, (b) $T=77$ K in LN_2 , (c) $T=4.2$ K in LHe ($F_N=50$ N; $v=1.0$ m/s; $s=2000$ m).

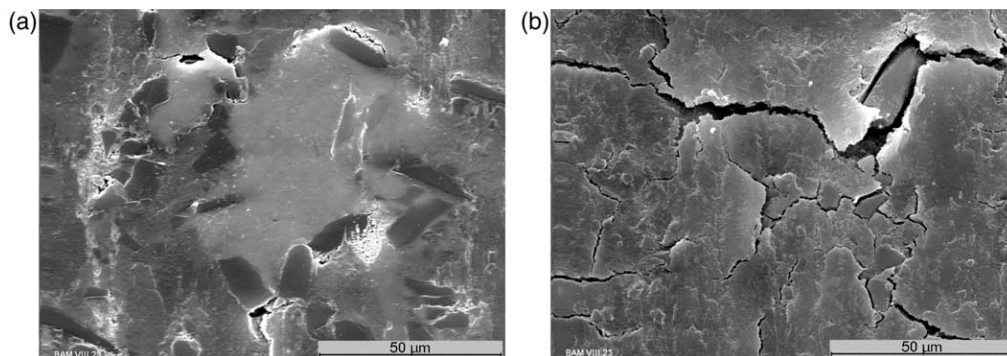


Fig. 12. SEM analysis of PTFE composites with 10 wt% Ekonol[®] and 15 wt% CF, after tribological tests at (a) RT in air and (b) $T=77$ K in LN_2 ($F=50$ N, $v=0.2$ m/s, $s=2000$ m).

analyses of a composite after room temperature and LN₂ tests at $v=0.2$ m/s. It can be seen that the surface of the composite tested in LN₂ is characterised by some cracks, which were not detected for the bronze filled composite in the same test conditions in LN₂ (Fig. 13).

4. Discussion

4.1. Influence of thermal properties of environments and materials

As described in [8,9], the coefficient of friction of PTFE composites decreases from room temperature to $T=77$ K in LN₂. This can be explained by the increased hardness of polymers at low temperature and a decrease in friction due to deformation. However, the temperature of the cryogen is not the significant parameter to understand and explain the tribological behaviour of these composites—and generally speaking of materials—at low temperature. The determinant factor is the physical—especially thermal—properties of the cryogen. A good example is given in our study with the friction results at $T=77$ K between He-gas and LN₂. Although both experiments are carried out in a medium at $T=77$ K, the coolant LN₂ has of course a better cooling efficiency than the He-gas.

Moreover, the decrease of friction with temperature of the cryogen is not observed down to extreme low temperature. Indeed, a higher coefficient of friction is measured at $T=4.2$ K compared with the one at $T=77$ K in LN₂, especially for the experiments carried out at high sliding speed.

This behaviour can not be explained with the bulk structure of PTFE, whose lowest transition temperature is at $T=160$ K, neither with the temperature dependence of its mechanical characteristics, because at $T=4.2$ K the hardness should be higher than at $T=77$ K according to [18]

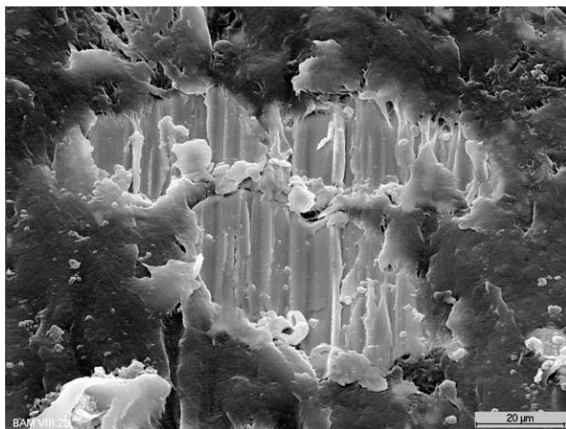


Fig. 13. SEM analysis of bronze and CF filled PTFE composites after tribological tests in LN₂ ($F=50$ N, $v=0.2$ m/s, $s=2000$ m).

Thermal properties of the cooling medium are in this case responsible for this increase. Since the frictional heat raises the temperature at the contact area, especially for polymers with low heat conductivity, the heat transfer to the environmental medium is a significant factor to consider. As explained in [19], as soon as a relative motion occurs and generates frictional heat, the liquid in the contact area begins to evaporate, and bubbles appear. Thereby, by thermal conduction, convection and evaporation, the heat is extracted from the contact. This leads to an efficient cooling effect. During this bubble formation phase (nucleate boiling), the temperature difference between the contact area and environmental medium is relatively small because the evaporation requires a large amount of heat. With increasing friction, the size and number of bubbles increases largely until finally reaching a vapour film at the contact area (film boiling). Since the heat transfer to a gaseous environment is much less efficient than the one to a boiling liquid, the transition from nucleate to film boiling is accompanied by a steep temperature rise which can amount to several hundred K. For free radiating heaters, the temperature increase as a function of the heat flux density can be taken from boiling curves [20]. The data from such curves cannot be transferred directly to the conditions in a frictional contact which represents a confined geometry and forced-flow conditions, but they may serve for a rough estimation of the temperature conditions in the contact zone. A direct measurement as well as a calculation of the actual temperature distribution in the contact zone are extremely complex and have not been carried out so far.

The results of the friction measurements in Fig. 2 indicated the effect of the heat transfer in different environmental media. In He-gas and liquid helium, no differences in the coefficient of friction appear at high speed ($v=1$ m/s). The heat flux density in both cases is 60 W/cm² for the bronze filled composite. Since for liquid helium the heat flux density at the onset of film boiling is only 1 W/cm², the contact area at 60 W/cm² is far into the film boiling regime with a corresponding ΔT of approximately 500 K for a static free radiating heater [20]. In this case the entire contact is certainly surrounded by gas, which separates the hot contact zone from the surrounding cryogen liquid. That means that the heat transfer at the friction contact is similar for each test in He-environment (77 K, 4.2 K), and thus the same temperatures lead naturally to the same coefficients of friction.

In liquid nitrogen a heat flux density of 30 W/cm² was calculated, which is just above the transition to film boiling for static free radiating heaters in LN₂ [20]. Because the rotating disc continuously transports liquid and provides a cold surface area into the contact zone, it can be assumed that this transition is shifted to higher heat fluxes and ΔT is still within a range of 10 K. The

low coefficient of friction and only small material transfer confirm this assumption.

At $v=0.2$ m/s, the heat flux density for LN₂ and LHe reach only 6 W/cm². The coefficient of friction in LN₂ and LHe are similar for the bronze filled composite. However, for the Ekonol[®] filled PTFE, the coefficient of friction is higher in LHe as in LN₂, but lower as in He-gas. The friction heat produced at 4.2 K is still high enough to reduce the cooling efficiency of the LHe to some extent. This can be explained by the fact that this composite has a lower thermal conductivity than the bronze filled material.

Hence, the tribological behaviour of the composite investigated in this experiment can be explained by the cooling efficiency of the cryogen. The hydrodynamic effect has not been considered since the viscosity of the cryogens are too low to establish a hydrodynamic film of sufficient thickness to separate the two surfaces [21]. However, a possible influence of the cryogenic fluid on the displacement of the wear particles from the sliding track can be considered. In this experiment, lots of small polymer debris were found on the surfaces of the pin and disc after testing in LHe, fewer after the experiment in LN₂. The exact contribution of the cryogen to remove the particles from the track is quite difficult to estimate in this experiment.

4.2. Surface analyses and wear process

The surface analyses of the discs show that PTFE matrix composites transfer down to very low temperatures. Although similar friction values were found in He-gas and LHe at $v=1$ m/s, surface analyses presented some differences (Fig. 5). In our investigations the transfer film at $T=4.2$ K is characterised by small cracks and wear particles. Obviously the transfer film formed during the test is affected by the low temperature, possibly after the test or between the passes.

At $v=0.2$ m/s, the surface of the disc tested in LN₂ and in LHe presented strong abrasive wear scarves. The fact that there are no or fewer deep scarves at $v=1$ m/s as at $v=0.2$ m/s at the surface of the disc, points out that the wear process is mainly adhesive in the first case and abrasive in the latter case. Low speed reduces the heat generated at the friction contact. Due to the resulting lower temperature, the polymer hardness increases, which causes more abrasive wear. That corresponds with previous studies where adhesive wear was observed in [4] whereas abrasive wear was found in [1] for experiments running at very low speeds (0.006 m/s), in order to keep the friction temperature low, to make the very low-temperature characteristics of the material visible.

4.3. Chemical surface analyses

A first attempt to understand the tribological behaviour of PTFE composites in a cryogenic environment by

means of chemical analyses was presented in this study. The XPS results correspond with previous works carried out with PTFE composites at RT [8,16] which described chemical reactions between the fluorine atom and the metal counterface. In this study, the presence of iron fluoride was detected down to LHe. It was also observed that the percentage of the fluor from PTFE decreases at the surface of the transfer in liquid helium and, respectively, the percentage of the metal fluoride increases. This could be due to the fact that iron fluorides build up at the steel surface within the first few runs of the test. Subsequently PTFE transfers to the top of this triboreaction layer. Similar structured layers of the transfer film were observed at room temperature by other workers [16,22]. At $T=4.2$ K the transfer film is characterised by small cracks and wear debris, which could belong to the upper PTFE layer and to the iron fluoride layer. In this study the relation between the presence of metal fluorides and the tribological performance was not obvious. However, fluoridation of metal surfaces with formation of metal fluorides layers is used in recent space applications studies to improve the tribological performance of cryogenic turbo pump bearings [23].

5. Conclusion

This work presents investigations on the tribological behaviour of PTFE composites against steel at cryogenic temperatures. It can be stated that thermal properties of the cryogenic medium have a significant influence on the tribological performance of the polymer composites. The generation of a gaseous film around the friction contact decreases significantly the cooling ability of the environment. Therefore, the effect of the low temperatures on the material properties was more clearly detected at low sliding speed, with a change in wear mechanism from adhesive to abrasive. Chemical analyses show the presence of iron fluorides down to 4.2 K. The XPS results suggested that these fluorides lay directly at the surface of the disc and are covered by a layer of PTFE. No influence of the metal fluorides on the tribological performance could be determined here, but results from other works suggest we should pursue these investigations.

Further studies will be carried out in particular at low speeds to evaluate the influence of the cryogenic environment on the tribological behaviour of polymer composites. The complex contact conditions encountered during sliding in cryogen, due to the presence of liquid and gas, will receive special attention, and temperature measurement will be further investigated.

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