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RESEARCH

Tribological Properties of Polytetrafluoroethylene with Mechanically Activated Carbon Fibers and Zirconium Dioxide

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ABSTRACT

This paper presents an investigation into the mechanical and tribological properties of PTFE composites reinforced with mechanically activated carbon fibers (5-15 wt.%) and nano-sized zirconium dioxide (1 wt.%) under self-mated contact conditions using a pin-on-disk tribometer. Mechanical tests reveal that incorporating mechanically activated carbon fibers, with or without zirconium dioxide, into PTFE increases compressive strength by 57-71% and Shore D hardness by 17-21% compared to the unfilled polymer. The composite density, measured using the hydrostatic weighing method, decreases with higher filler content (5 to 15 wt.% carbon fibers) regardless of zirconium dioxide inclusion. Tribological tests demonstrate that PTFE reinforced with carbon fibers and nano-sized zirconium dioxide exhibits superior wear resistance compared to composites containing only carbon fibers. However, all composites show higher coefficients of friction and surface roughness R_a than unfilled PTFE under dry sliding conditions. IR spectroscopy reveals the new peaks corresponding to oxygen-containing functional groups, suggesting tribochemical reactions occurring on the worn surfaces. Scanning electron microscopy and optical microscopy were used to analyze the friction surfaces of the polymer matrix and polymer composites and the transfer film formed on the surface of the steel counterbody. In addition, the tribological properties are compared with industrial analog.

1. INTRODUCTION

In modern applications, machine components with interacting mating surfaces in relative motion such as plain bearings, bushings, seals, or valves, collectively referred to as tribosystems are of particular importance in the Arctic region. For tribosystems, it is insufficient to consider only the temperature factor; the environmental conditions must also be taken into account. For instance, aggressive media, such as various hydrocarbons, are often present in friction units.

Polytetrafluoroethylene (PTFE) is one of the few industrial polymers capable of meeting the rigorous demands of the Arctic climate, owing to its excellent antifriction properties, chemical inertness, and resistance to both extreme cold and heat [1-3]. PTFE remains operational across a broad temperature range of -260 to +260 °C [2,4]. However, its inherently low wear resistance and high creep under stress deformation limit its use in friction units. These limitations stem from the molecular and supramolecular structure of the unfilled polymer [5]. An effective method to enhance PTFE wear resistance and reduce creep is the incorporation of solid fillers, resulting in the development of polymer composite materials (PCM). Commonly studied and widely used fillers for reducing polymer wear include aluminum oxide, glass fibers, carbon fibers, bronze, graphite, and similar materials [6-8]. Nanoscale particles, such as graphene, fullerenes, and carbon nanotubes, have also been explored as fillers [9-11]. However, for PTFE-based polymer composites designed for tribological applications, carbon fibers (CF) are particularly favored due to their chemical inertness, high strength, excellent elastic modulus, and self-lubricating properties [12,13].

Promising methods to enhance the tribological properties of PTFE involve the incorporation of complex fillers, including CF of various sizes. Ye et al. [14] investigated the tribological, mechanical, and thermal properties of PTFE composites reinforced with CFs and other carbon-based fillers, such as graphene, carbon nanotubes, and graphite. The study revealed that adding graphene to CF/PTFE significantly enhanced tribological properties by forming a smooth and continuous film on the counterbody and improving thermal conductivity. Song et al. [15] explored the tribological properties and PV factor of PTFE-based PCM filled with short CF, glass fibers, and MoS_2. The results showed that incorporating MoS_2 into fiber-filled PCM substantially improved wear resistance at low sliding speeds. However, it was observed that adding glass fibers alone to PTFE significantly deteriorated wear resistance compared to CF-filled composites. Zhang et al. [16] studied a series of PTFE composites filled with CF (sizes ranging from 7.498 μm to 53.857 μm) and MoS_2. Their findings indicated that the CF/MoS_2/PTFE composite containing fibers with a particle size of 7.498 μm exhibited optimal tensile strength and relative elongation at break. Additionally, the tribological properties of the composites improved with increasing fiber length up to 23.733 μm, resulting in a lower coefficient of friction and enhanced wear resistance. He et al. [17] examined the mechanical and tribological properties of PTFE reinforced with CF of different sizes and cubic boron nitride. Their study demonstrated that the specific wear rate of composites containing a combination of fiber types decreased by 91.1% and 93.3%, respectively, compared to composites with only one type of fiber. The results showed that larger fibers effectively transferred stress, improving the mechanical properties of the composites, while smaller fibers self-oriented and accumulated at the friction surface, collectively yielding excellent wear resistance of the polymer composites.

In previous studies [18,19], we examined polymer composites based on PTFE filled with CFs and dispersed natural fillers such as kaolin, vermiculite, and zeolite. These studies demonstrated that incorporating a mixture of CF and dispersed fillers significantly enhanced the tribological properties of PTFE compared to composites containing only fibers. Other PTFE-based composites with CFs and nanoscale particles have also been investigated. Among these, zirconium dioxide was identified as providing the most favorable tribological properties [20]. Studies by other researchers [21-23] further support this finding, showing that incorporating zirconium dioxide into the polymer matrix significantly improves mechanical and tribological performance. Specifically, zirconium dioxide enhances wear resistance while maintaining a relatively low coefficient of friction. The mechanical activation of solid particles, often performed in a planetary mill, disperses the particles and increases their surface area. This improved dispersion can enhance particle adhesion to the polymer matrix, leading to superior composite properties. Consequently, mechanically activating short CF before their incorporation into a polymeric material is a promising physical method for improving their interaction with the matrix. Petrova et al. [24,25] explored joint mechanical activation of the PTFE polymer matrix and CF of the UVIS-AK-P brand. Their results showed that joint mechanical activation significantly improved the mechanical and tribological properties of the polymer composites.

Although numerous studies have explored methods to enhance the tribological properties of PTFE filled with fibers and nanoparticles, there remains potential for further improvement through mechanical activation. In our previous work [20], we investigated the mechanical and tribological properties of polymer composites based on PTFE filled with mechanically activated carbon fibers for 5 min, including silicon dioxide and zirconium dioxide, as well as boron nitride. The results showed that studying the properties of PTFE with mechanically activated CF offers interesting possibilities for improving mechanical performance and wear resistance. Additionally, incorporating nanoscale zirconium dioxide into PTFE/CF composites is a promising strategy to further improve PTFE tribological properties. In this work, the time of mechanical activation of the carbon fibers was optimized to 2 min, and the content of CFs was varied from 5 to 15 wt.%.

2. MATERIALS AND METHODS

PTFE of PN-90 grade (HaloPolymer, Russia) was used as a polymer matrix. The average particle size of PTFE powder is ~90 µm, density 2.16 g/cm³. Short CFs with the following dimensions were used as filler: length 200 µm, diameter 7 µm, and density ~1.75 cm³ (R&G Faserverbundwerkstoffe GmbH, Germany) and zirconium dioxide (ZrO_2) powder (Plasmotherm LLC, Russia) with particle size 40-75 nm, product purity 99.75%.

Mechanical activation of CF was performed for 2 min on a planetary mill Activator-2S (Activator, Russia). Leave one clear line before and after a main or secondary heading and after each paragraph. Preliminary ultrasonic treatment of ZrO2 was carried out in IL100-6/4 (IN-LAB-Ultrasonics, Russia) for 5 min based on the method used in the previous study [20]. Mixing of PTFE powder with filler particles (CF and ZrO_2) was carried out in dry form in a paddle mixer for 2 min. Then the samples for testing were obtained by compression pressing in a mold on a hydraulic press at a specific load of 50 MPa with a dwell time of 2 min. Sintering of the pressed samples was performed in a programmable electric furnace SNOL 15/900 (SNOL, Lithuania) at a temperature of 375 ºC. The content of CF filler in the polymer was varied – 5, 10, and 15 wt.%, and the content of ZrO_2 was constant – 1 wt.%.

Fig. 1 shows optical microscope images of fibers before and after mechanical activation on the planetary mill, obtained using an Olympus BX41 microscope (Olympus, Japan).

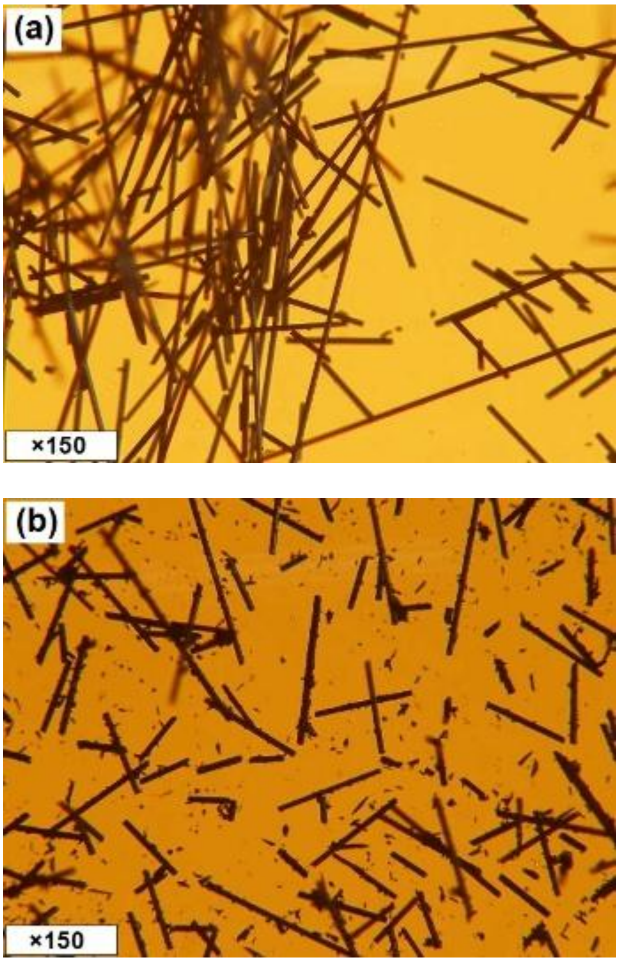


Fig. 1. Optical microscope images of carbon fibers before (a) and after (b) mechanical activation (magnification at ×150)

The mechanical properties of the neat polymer and PCM were characterized by compression testing using standard methods at room temperature on an Autograph AGS-J universal testing machine (Shimadzu, Japan). The compression test was conducted according to ISO 604:2002, obtaining the compressive stress indicator at 10% relative strain. For this technique, specimens in the form of a straight cylinder with a diameter of 10.00 ± 0.01 mm and a height of 21.00 ± 0.01 mm were used, and the test speed was 1 mm/min. The number of specimens used per test was 5. The hardness of the specimens was measured using a TBP-D hardness tester (Vostok-7, Russia) with an analog indicator for measuring hardness on the Shore hardness scale type D according to ISO 868-2003.

Measurement of the surface roughness parameter R_a – arithmetic mean deviation of the profile, was carried out in accordance with the nomenclature and ranges of values provided by the Russian standard GOST 2789-73 using a portable roughness tester TR220 (Beijing Time High Technology Co., Ltd., China). The parameters of the device correspond to the technical conditions GB/T 3505-2000. The density of unfilled polymer and PCM samples was determined based on the standard method of the Russian Standard GOST 15139-69 – hydrostatic weighing. The method involves comparing the masses of equal volumes of the test substance and a liquid of known density (e.g., distilled water), referred to as the working fluid.

Tribological characteristics all composites: the specific wear rate and coefficient of friction were investigated using a universal tribomachine CETR UMT-3 (CETR, USA) following the "pin-on-disk" friction scheme. The specimen had a cylindrical shape with a radius of 5 mm from the center, with a counterbody made of steel #45 with a hardness of 45–50 HRC and a roughness R_a = 0.06-0.08 µm. The tests were carried out at a specific pressure of 2 MPa and a linear sliding speed of 0.2 m/s at room temperature. The test time for each specimen was 3 h. The specimens were cleaned and weighed on analytical scales (0.00001 g) before testing, and the masses before and after friction were recorded. The specific wear rate was determined using equation (1):

$$k = \frac{\Delta m}{F_N \cdot d \cdot \rho}, \tag{1}$$

where F_N – normal force, N; d – friction distance, m; Δm – lost mass of the sample after friction, g; ρ – density, g/cm^3.

The IR spectra of the samples were recorded on a Varian 7000 FT-IR Fourier transform infrared spectrometer (Varian, USA). The spectra were obtained in the range of 550–4000 cm^{-1}. The samples were structurally characterized using a Jeol JSM-7800F scanning electron microscope (JEOL, Japan) with EDS detector (Oxford Instruments, UK). Secondary electrons (1-2 kV) were used to obtain micrographs of the samples. Samples for the study of the supramolecular structure of PCM were prepared by cold chipping, i.e., brittle fracture of the sample in liquid nitrogen.

3. RESULTS AND DISCUSSION

Several operating parameters are considered when selecting materials for friction units. One of the most important mechanical properties is hardness and compressive strength, which determine the reduction of wear in contact with other materials and the resistance to loads. The results of tests on Shore D hardness, compressive strength, and density of PTFE and PCM depending on the composition and filler content are shown in Fig. 2.

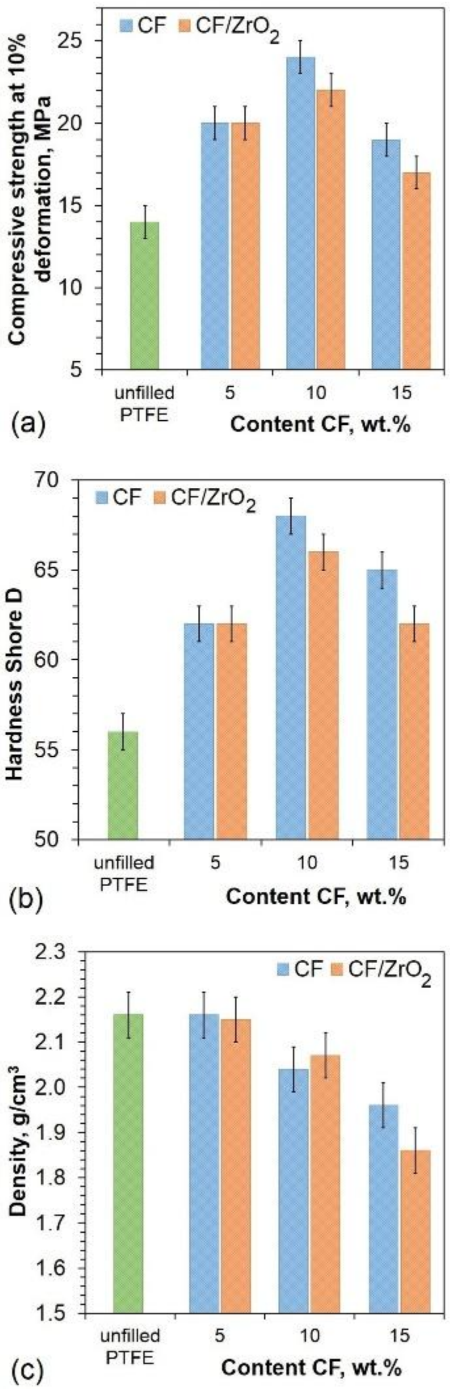


Fig. 2. Compressive strength at 10% deformation (a), hardness Shore D (b) and density (c) unfilled PTFE and PCM.

Fig. 2(a) shows that the introduction of mechanically activated CF in the amount of 5 to 10 wt.% into PTFE leads to an increase in the compressive strength by 57-71% compared to the unfilled polymer. The reinforcement of the polymer matrix upon the introduction of CF can be attributed to the reinforcing effect resulting from stress transfer from the matrix to the fibers, as previously observed by [26]. It can be reasonably assumed that the strength properties will increase in proportion to the quantity of fibers present in the polymer. However, an increase in CF content from 10 wt.% to 15 wt.%, irrespective of the ZrO_2 content, is accompanied by a reduction in compressive stress, which is only 21-35% higher than that of the polymer matrix. Such a change in compressive stress can be attributed to the formation of a more porous and defective structure when the fiber content is increased up to 15 wt.%. This is indicated by a ~14% decrease in density relative to unfilled PTFE and also compared to composites containing 5 to 10 wt.% CF and CF/ZrO_2. In other PCM samples (5-10 wt.%), a less pronounced alteration in density is discernible. The Shore D hardness of the composites exhibited an increase of 17-21% with the incorporation of CF and CF/ZrO_2 in the range of 5 to 10 wt.%. This indicates that the selected fillers effectively enhance the hardness of PTFE. However, when the filler content was further increased from 10 to 15 wt.% regardless of the ZrO_2 content, the Shore D hardness increased only by 12-18% compared with the unfilled PTFE. The observed increase in the hardness value of PCM can be attributed to the presence of hard fibers in comparison to the relatively soft polymer matrix [27]. Thus, the introduction of mechanically activated CF in the amounts of 10 wt.% and 1 wt.% ZrO_2 into PTFE leads to an improvement in the compressive strength properties and hardness of the polymer matrix, which are important for tribological materials.

Fig. 3 shows the results of the study of the supramolecular structure of the unfilled PTFE by scanning electron microscopy. It can be seen that the initial polymer matrix is characterized by a ribbon (lamellar) structure, which is typical for this polymer [28].

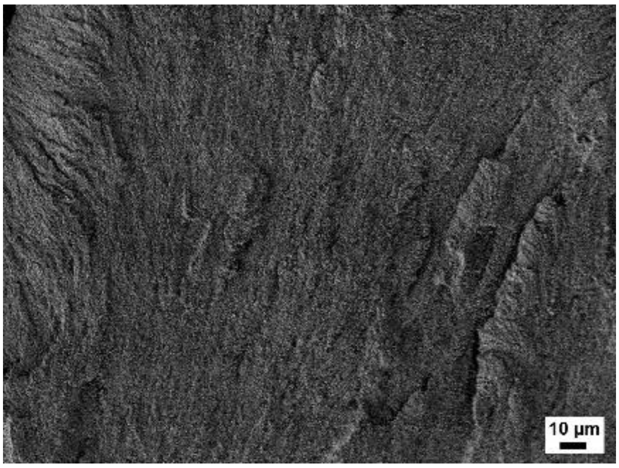
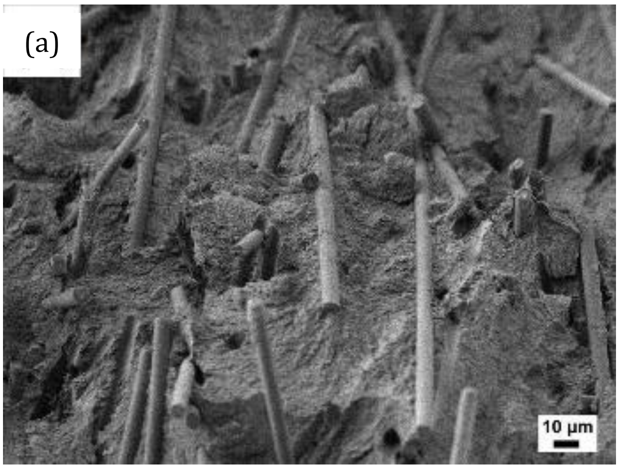
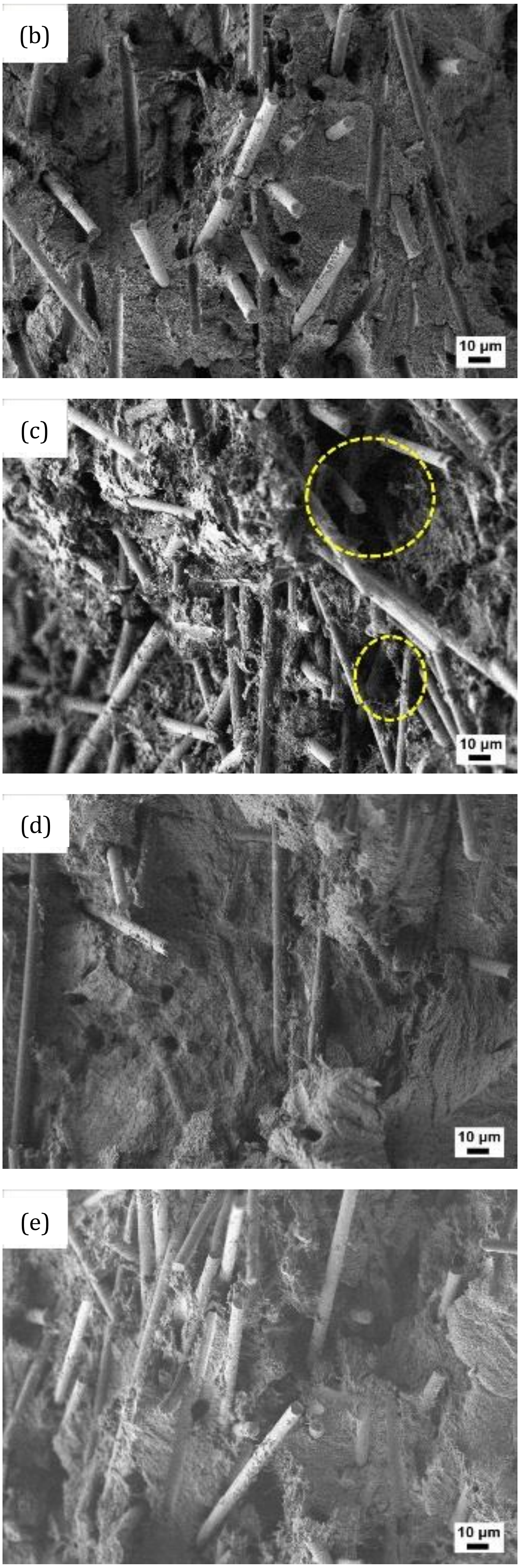


Fig. 3. SEM-image of the supramolecular structure of unfilled PTFE.

Further, PCM were investigated by the SEM method depending on the composition and filler content, and the results are presented in Fig. 4.

Fig. 4 shows that in the supramolecular structure, CF with a content of 5-10 wt.% are visually uniformly distributed and randomly oriented, and no agglomerates of fibers were found. The randomly oriented fibers are believed to isotropically reinforce the polymer matrix, thereby improving the mechanical properties and hardness. The SEM method was unable to identify the presence of nanosized ZrO_2 particles within the supramolecular structure of PCM. A higher filler content of 15 wt.% in PTFE leads to poor compaction of polymer composites (Fig. 4 (c) and (f)). The formation of voids and fiber agglomerates (highlighted in yellow) is evident in both cases of composite composition with CF and CF/ZrO_2 at 15 wt.% filler. The formation of such a supramolecular structure explains the decrease in mechanical properties and density of PCM.





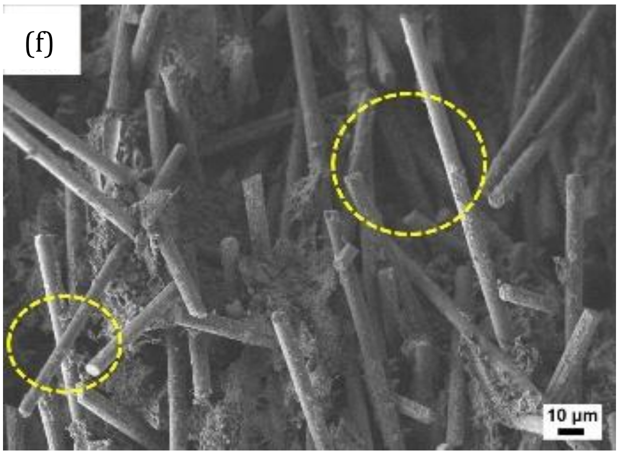


Fig. 4. SEM images of the PCM supramolecular structure: (a) PTFE/5CF; (b) PTFE/10CF; (c) PTFE/15CF; (d) PTFE/5CF/ZrO_2; (e) PTFE/10CF/ZrO_2; (f) PTFE/15CF/ZrO_2.

The tribological properties and roughness results R_a of unfilled PTFE and PCM are presented in Fig. 5. In general, it can be seen that the introduction of mechanically activated CF and CF/ZrO_2 has a positive effect on the tribological properties of PTFE under dry friction. Accordingly, the composite wear rate is significantly reduced in all composites and the coefficient of friction is slightly increased compared to unfilled PTFE.

As can be seen from Fig. 5(a), in all composites, the wear rate passes through a minimum, reaching in composites with 10 wt.% CF. It can be observed that the wear rate value is 1.38×10^{-6} mm^3/N·m in the PTFE/5CF composite and 0.86×10^{-6} mm^3/N·m in the PTFE/10CF composite. These values are 339-544 times lower than those observed in unfilled PTFE. The composites containing ZrO_2 exhibit a comparable pattern of variation in k, with values of 0.56×10^{-6} mm^3/N·m at 5 wt.% CF and 0.53×10^{-6} mm^3/N·m at 10 wt.% CF. These values indicate an 835–883 times higher wear resistance compared to the polymer matrix. Comparable results were observed between the PCM when the CF content was increased to 15 wt.%. It can be seen that the PTFE/15CF and PTFE/15CF/ZrO_2 composites exhibit a decline in wear resistance (k=1.30-1.46×10^{-6} mm^3/N·m) in comparison to the other PCM. A comparative analysis of the wear rate of PTFE/5CF and PTFE/5CF/ZrO_2 revealed that the composite with an increased ZrO_2 content exhibited a 2.4-fold enhancement in wear resistance. A comparison of the wear resistance of PTFE/10CF and PTFE/10CF/ZrO_2 revealed that the latter exhibited a 1.6 times higher wear resistance.

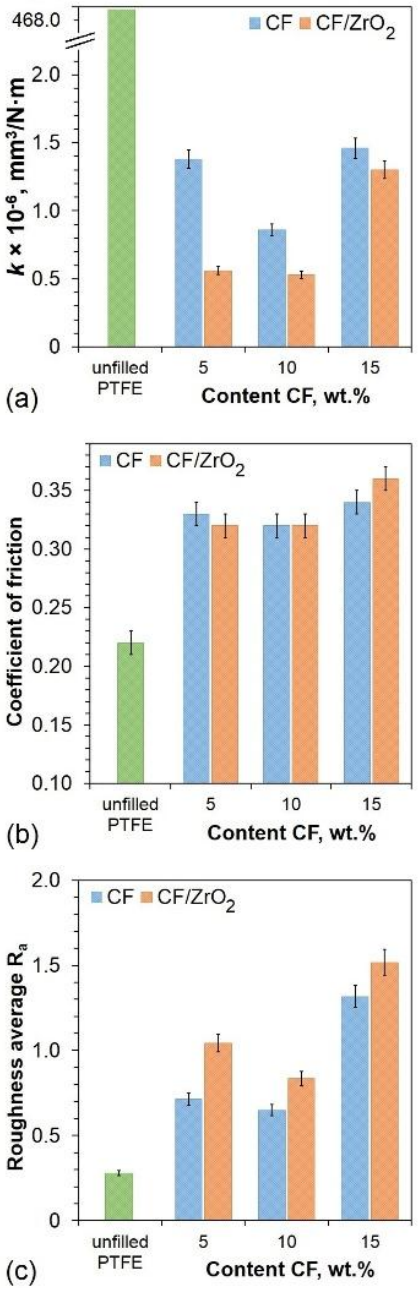


Fig. 5. Tribological properties of unfilled PTFE and PCM: a) specific wear rate; b) coefficient of friction; c) roughness R_a.

For all PCM, the coefficient of friction increased relative to the polymer matrix, regardless of the ZrO_2 content (Fig. 5(b)). The coefficient of friction exhibited an increase of 45% at a CF content of 5-10 wt.% in PTFE and a further increase of 63% at a CF content of 15 wt.% in comparison with the unfilled polymer. The roughness R_a of composites at a concentration of 5-10 wt.% CF was observed to increase in comparison to the unfilled polymer. This fact can be explained by the content of fillers in the form of fibers, which will obviously protrude on the friction surface, thereby increasing the coefficient of friction of PCM. The greatest increase in roughness R_a and the coefficient of friction was observed in PCM with a 15 wt.% content, which can be attributed to the higher fiber content (Fig. 5(c)). Thus, the best results in terms of wear resistance were obtained in the PCM of PTFE/5CF/ZrO_2 and PTFE/10CF/ZrO_2 compositions.

Fig. 6 shows the results of IR spectroscopy of the unfilled PTFE surface before and after friction and PCM after friction. In PCM before friction, the initial surfaces are the same and correspond to the IR spectrum of the polymer matrix surface.

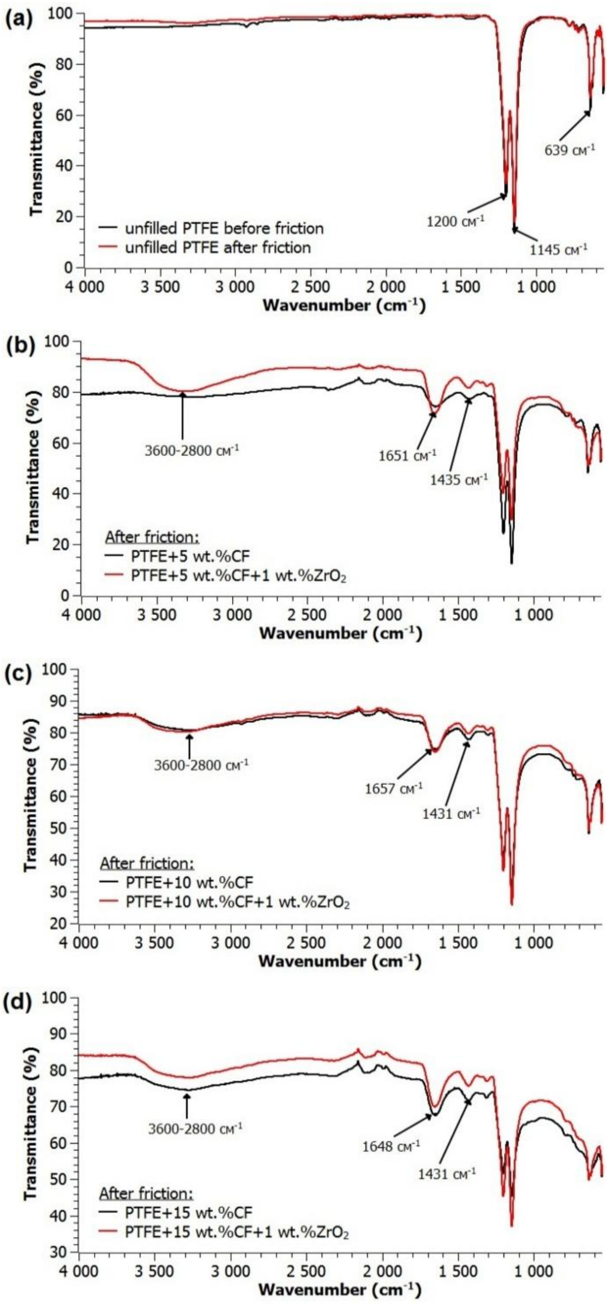


Fig. 6. IR spectra of samples as a dependence of CF content: (a) unfilled PTFE; (b) 5 wt.%CF; (c) 10 wt.%CF; (d) 15 wt.%CF.

The IR spectra of unfilled PTFE and PCM revealed strong peaks in the regions of 1200 cm^{-1}, 1145 cm^{-1}, and ~639 cm^{-1}, which are attributed to the polymer matrix of PTFE [29]. These main absorption bands of PTFE are retained after friction in all investigated PCM samples. Fig. 6(a) shows that no new absorption bands are detected in the IR spectrum of unfilled PTFE after friction. New peaks were detected on the worn PCM surfaces, where a broad band in the 3600-3200 cm^{-1} region refers to the vibrations of OH hydroxyl groups caused by adsorption during friction. The new peaks at ~1651 cm^{-1} and ~1435 cm^{-1} on the worn surfaces are attributed to metal chelates of perfluorinated carboxylic acid salts $M_2(R_f\text{-}COO^-)$ [30,31]. The registered new absorption peaks in the IR spectra of PCM after friction are associated with the polymer chain breakage by C-C bonds. Further, the broken bonds form radicals that can react with oxygen and ambient moisture, thereby forming carboxylic acid end groups [32,33]. These peaks indicate the occurrence of tribochemical reactions on the PCM friction surface. It is known that during such reactions, strong tribofilms are formed, protecting the surface layer of the material from wear [34,35]. This fact is consistent with the results of tribological studies (Fig. 5(a)), since at a content of 5 wt.% CF, the highest difference in the specific wear rate was obtained between PTFE/CF and PTFE/CF/ZrO_2.

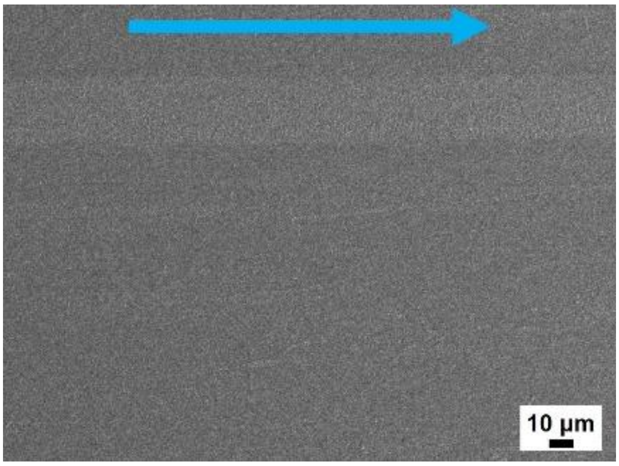
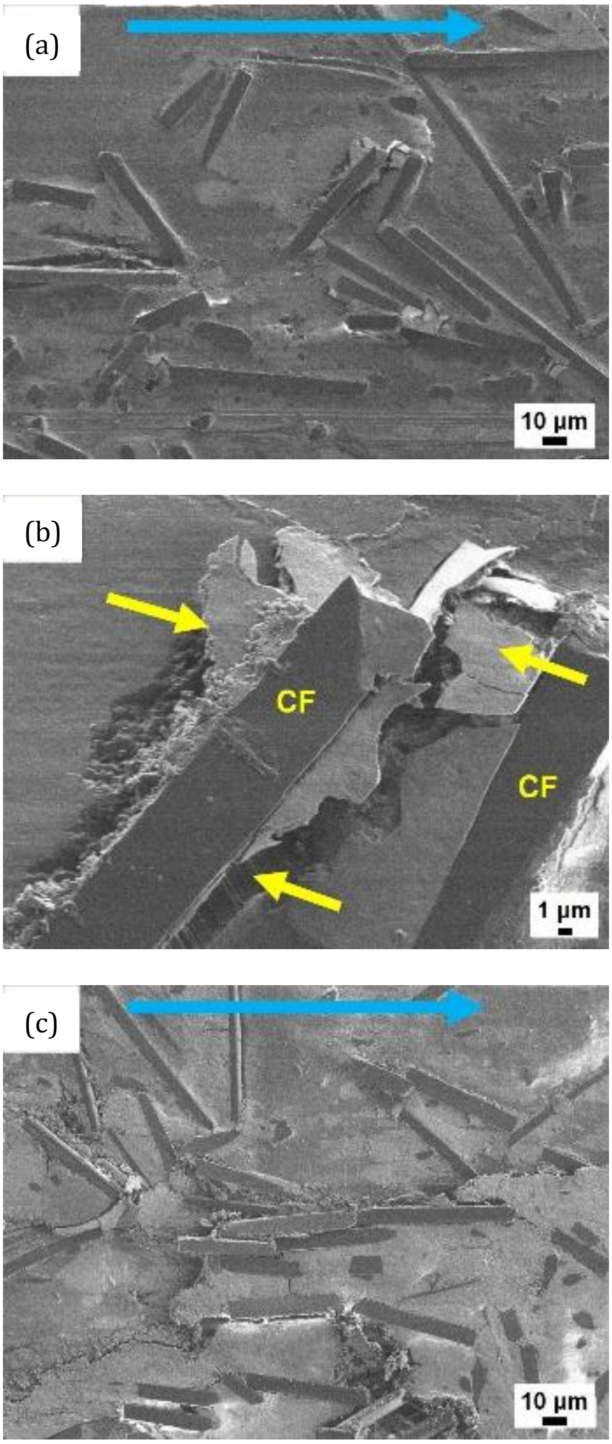


Fig. 7. SEM image of the worn surface of unfilled PTFE.

Fig. 7 shows the results of the worn surface of unfilled PTFE. It can be seen that the worn surface of unfilled PTFE looks quite smooth in the sliding direction, which is also consistent with the result of the R_a roughness study. The formation of such a worn surface of unfilled PTFE is due to the micro-cutting of the steel counterbody surface and the release of a large number of wear products. This explains the low wear resistance of the PTFE and is typical of this polymer, which has also been shown in the work of other authors [36,37].

Fig. 8 illustrates the SEM images of worn surfaces PTFE/CF depending on the filler content.



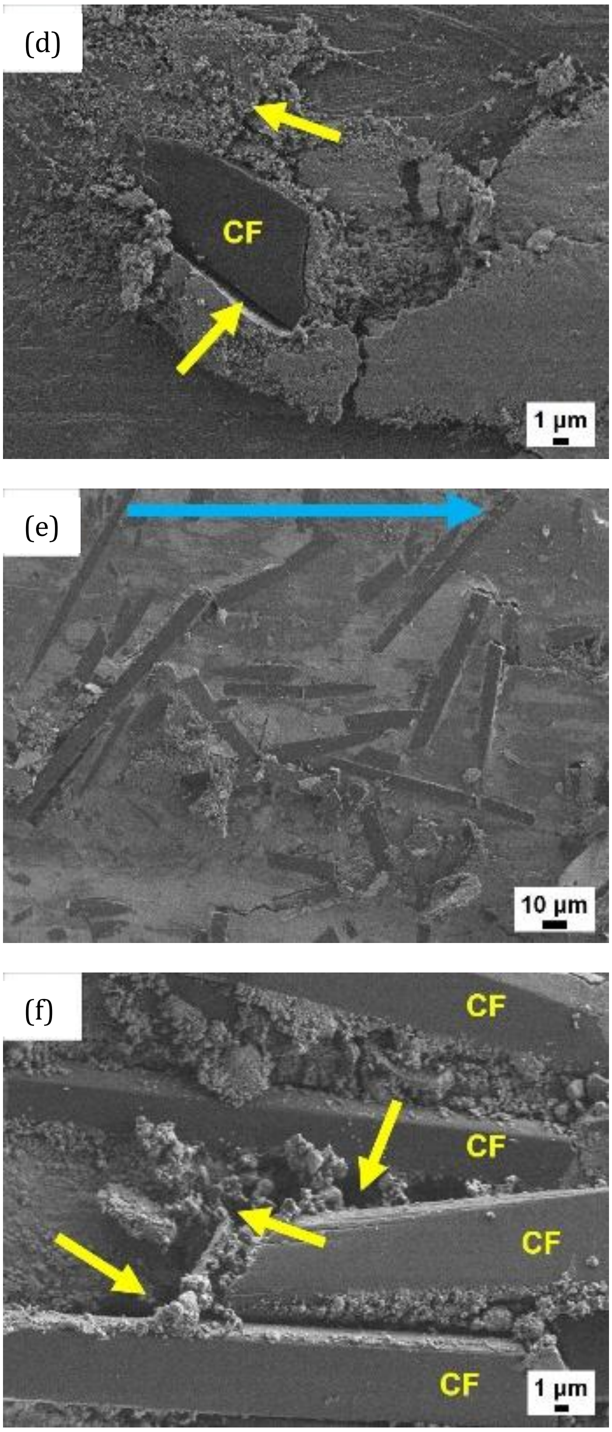
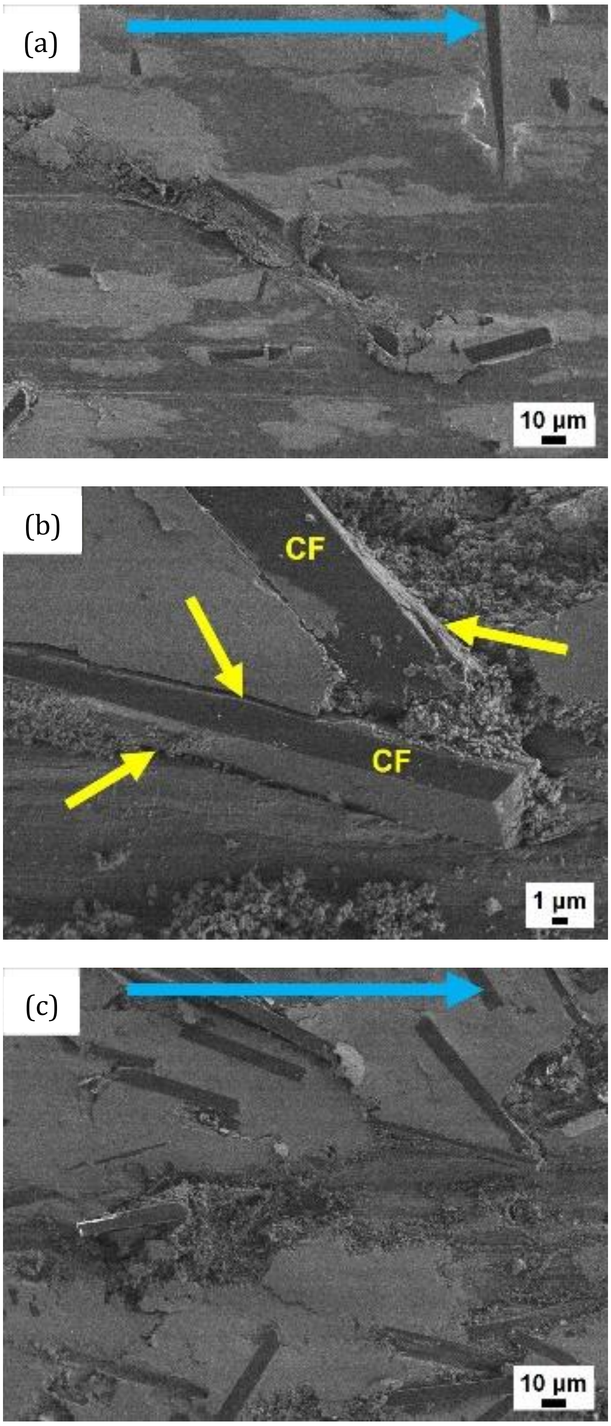


Fig. 8. SEM images of worn PCM surface at different magnifications: (a, b) PTFE/5CF; (c, d) PTFE/10CF; (e, f) PTFE/15CF.

Fig. 8 shows that in all PCM samples, CF are observed on the worn surface, which are randomly oriented as in the supramolecular structure. The orientation of the fibers is not affected by the direction of friction (highlighted blue arrows), as seen on the worn surfaces of PCM. PTFE/CF composites, defective areas in the form of delamination of part of the polymer, and large gaps at the polymer-fiber interface are visible under high magnification (Fig. 8(b), (d), (f): a yellow arrow marks this region). In addition, a large amount of wear product accumulation is observed on the same worn PCM surfaces. This is usually due to the fact that the main load is on the CFs, and the actual contact area is reduced compared to the original surface [37]. Thus, the fibers will further delaminate from the polymer matrix with friction time.

Fig. 9 shows the results of the worn surface of PTFE/CF/ZrO_2 depending on the filler content.



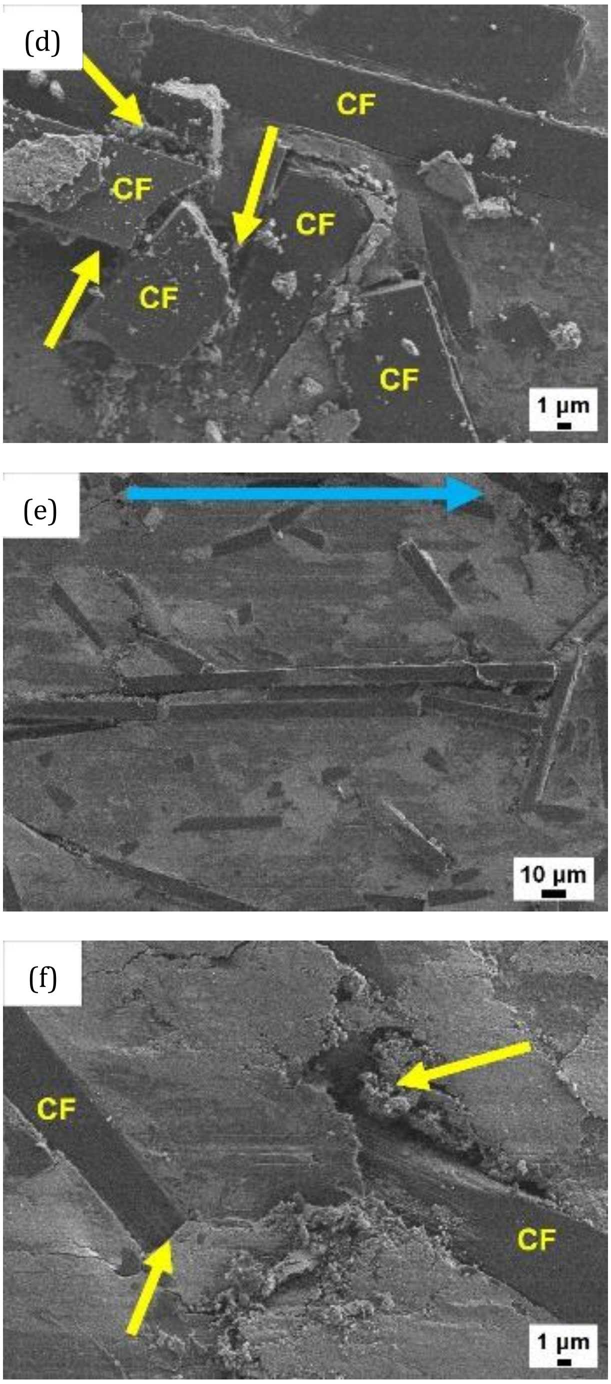


Fig. 9. SEM images of worn PCM surface at different magnifications: (a, b) PTFE/5CF/ZrO_2; (c, d) PTFE/10CF/ZrO_2; (e, f) PTFE/15CF/ZrO_2.

In the case of PTFE/CF/ZrO_2 composites, no delamination of the polymer is seen, and small gaps are observed at the fiber-polymer interface (Fig. 9(b), (d), (f)). In addition, little accumulation of wear products is observed on the worn surface.

The investigations of the worn surface (Fig. 9) are consistent with the specific wear rate results (Fig. 5 a), since more wear-resistant composites were obtained with the content of zirconium dioxide. However, the R_a roughness of PTFE/CF/ZrO_2 composites is slightly higher than that of PTFE/CF composites. Perhaps, the reason for the difference is the formation of denser secondary structures in the form of tribofilms in composites with PTFE/CF/ZrO_2, which in general increase the surface roughness compared to PTFE/CF. Similar results of roughness changes and their relationship to tribofilm formation have been shown in [38,39]. Thus, a possible mechanism for increasing wear resistance is the formation of a stable layer of secondary structure in the form of tribofilm. Tribofilms formed during friction reduce frictional loads on individual fibers and localize shear deformation, which increases the wear resistance of the composite material.

Next, the friction surface was investigated by EDS analysis method and the results are presented in Table 1.

Table 1 shows that in the case of PTFE/CF/ZrO_2 there are more intensive tribochemical reactions compared to PTFE/CF. In addition, Fe atoms were detected in the composites with PTFE/CF/ZrO_2, which indicates their abrasive effect. Thus, it is the introduction of additional ZrO_2 that contributes to the wear resistance, as indicated by the tribological test results. Thus, the wear rate of composites with 5 wt.% CF and 1 wt.% ZrO_2 is lower than in the composite with 10 wt.% CF. Probably, the introduction of zirconium dioxide promotes the formation of stable tribofilms on the friction surface of composites. This is evidenced by studies of friction surfaces of composites.

Table 1. Comparison of developed PCMs with the industrial analog.

Element	**PTFE/CF**	**PTFE/CF/ZrO_2**
	Atomic, %	**Atomic, %**
C	34.40	14.08
F	8.20	19.30
O	53.37	66.62
Fe	4.03	-

It is well known that when PTFE friction is applied to a metal counterbody, the formation of large lamellar debris, poorly adhered transfer films, and unacceptably high wear rates are observed due to easy shear between polymer lamellae or fibrils [40]. In addition, one of the criteria for increasing the wear resistance of PTFE-based PCM is related to forming a more durable transfer film [41]. The surface of the steel counterbody was evaluated using an optical microscope in Fig. 10.

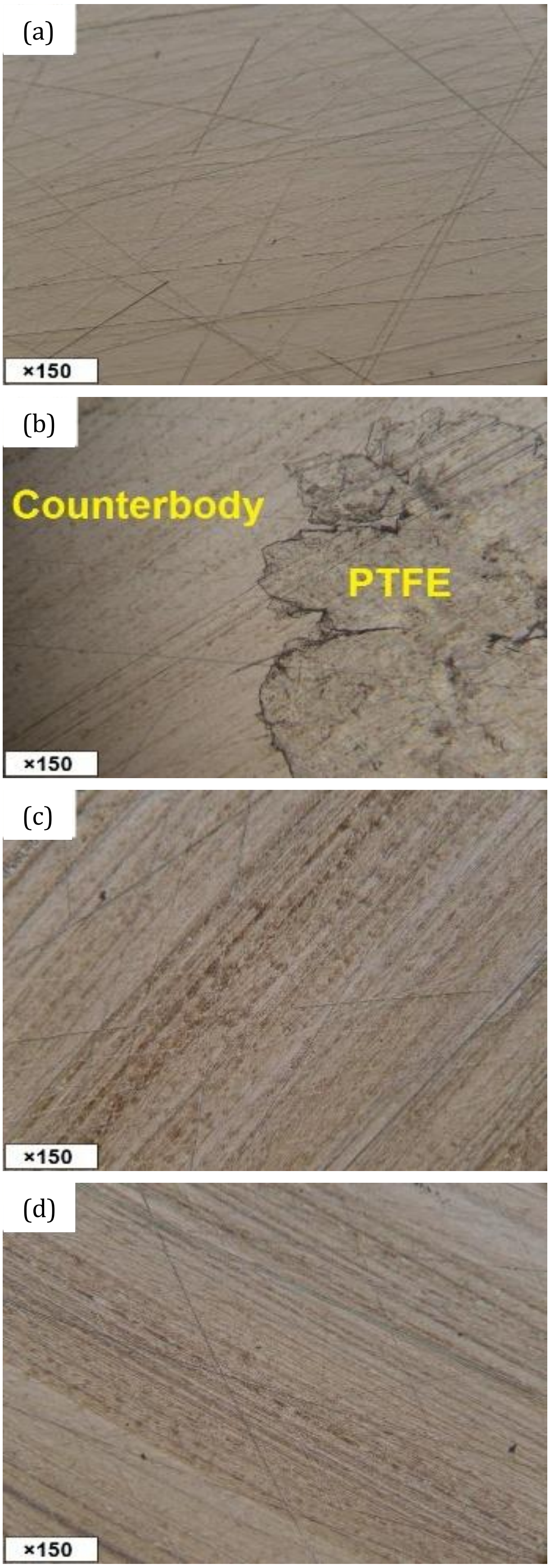


Fig. 10. Optical microscope images of the worn surfaces of the steel disc #45 (magnification at ×150): a) initial surface of steel counterbody #45; b) unfilled PTFE; c) PTFE/10CF; d) PTFE/10CF/ZrO_2.

From Fig. 10(a), it can be seen that the surface of the initial counterbody has different scratches, but after the friction test, the formation of the transfer film can be clearly distinguished. As shown in Fig. 10(b), the initial PTFE forms a visually large transfer film with very little coating on the surface of the metal counterbody. In the case of PTFE/10CF and PTFE/10CF/ZrO_2, a visually thin transfer film is formed; the difference from unfilled PTFE is a large amount of coating and a more homogeneous distribution on the surface of the counterbody (black dots). In the remaining polymer composites, at 5 wt.% CF and 15 wt.% CF, almost identical results were obtained. Such formation of the transfer film on the friction surface is due to the fact that in the case of PCM, chemical reactions occurred (IR spectra in Fig. 6) and the adhesion of the transfer film improved accordingly. On the contrary, no new peaks were detected on the friction surface for the unfilled PTFE. At the same time, there were no significant visual differences between PTFE/10CF and PTFE/10CF/ZrO_2 on the surface of the counterbody, confirming the formation of a stable tribofilm on the worn surface of the PTFE/CF/ZrO_2 composite.

A comparison of the tribological properties of PCM without mechanical activation and with the industrial analog F4K20 consisting of 80 vol. % PTFE + 20 vol. % foundry coke, has been carried out, the results are presented in Table 2. The industrial analog of F4K20 is commercially available material and is manufactured according to TU 6-05-1413-76 (Russia). Tribological tests of F4K20 were carried out under the conditions of the developed materials.

Table 2. Comparison of developed PCMs with the industrial analog.

PCM	k, 10^{-6} mm^3/N·m	CoF
PTFE/10CF without mechanical activation	0.89	0,33
PTFE/10CF	0.86	0.32
PTFE/5CF/ZrO_2	0.56	0.32
PTFE/10CF/ZrO_2	0.53	0.32
F4K20	1.75	0.32

Table 2 shows that the developed PCMs are characterized by improved wear resistance compared to PCM without mechanical activation and F4K20. In the developed composites PTFE/5CF/ZrO_2 and PTFE/10CF/ZrO_2, the wear rate decreased by 37-40% compared to PCM without mechanical activation and by 3 times compared to the industrial analog F4K20. However, the coefficient of friction in all composites is at the same level. Thus, the developed composites can replace the industrial composite F4K20 in various friction units due to the increased wear resistance.

4. CONCLUSION

This study presents the mechanical and tribological properties of PTFE composites reinforced with mechanically activated CF (5-15 wt.%) and nanosized zirconium dioxide (1 wt.%). It is revealed that the introduction of mechanically activated CF and CF/ZrO_2 into PTFE has a positive effect on its mechanical properties. It is shown that the optimum content of fillers in PTFE is 10 wt.% CF and CF/ZrO_2, at which the level of compressive strength increases by 57-71% and Shore D hardness by 17-21%. Increasing the fiber content up to 15 wt.% leads to a deterioration of these parameters due to the formation of pores and agglomerates. The study of the structure of the materials by scanning electron microscopy has shown that the fibers are visually quite uniformly distributed and chaotically oriented at the content of 5-10 wt.%. The results of tribological tests of PTFE composites showed a significant improvement with the addition of ZrO_2 nanoparticles compared to PTFE/CF. However, PCM showed higher values of the coefficient of friction and roughness R_a than unfilled PTFE. The IR spectroscopy method revealed new peaks identified as oxygen-containing functional groups, indicating the occurrence of tribochemical reactions. SEM studies showed the formation of smaller gaps in PTFE/CF/ZrO_2 than in PTFE/CF. Optical microscopy of the counterbody surface showed the difference between PCM and unfilled PTFE. However, no significant differences were found between PTFE/10CF and PTFE/10CF/ZrO_2. Thus, it is possible that the improvement in the tribological properties of PTFE/CF/ZrO_2 is associated with the formation of tribofilms on the surface of the composite that protect the material from wear. The developed materials, due to their increased wear resistance, can be used in various friction units of machines, including as a replacement for F4K20.

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