Tribology and Materials
Vol. 4, No. 2, 2025, pp. 56-65

ISSN 2812-9717
https://doi.org/10.46793/tribomat.2025.010

Mechanical and tribological properties of polymer composite materials based on PTFE filled with natural mica and magnesium aluminium oxide

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Keywords
polytetrafluoroethylene
mica
magnesium aluminium oxide
composites
wear resistance

History
Received: 13-05-2025
Revised: 11-06-2025
Accepted: 19-06-2025

Abstract
In this research, the results of studies on the influence of natural mica and mixed magnesium aluminium oxide (MAO) on the structure and properties of polytetrafluoroethylene (PTFE) are presented. Mechanical and tribological tests were conducted on PTFE-based composite samples. It was found that the addition of mica increases the relative elongation by 43 % and raises the wear resistance by 277 times compared to the initial polymer. The coefficient of friction is from 0.24 to 0.29. At the supplementary addition of mixed magnesium aluminium oxide, the wear resistance increases by up to 312 times and the coefficient of friction decreases by 38 %. It is shown that, with the addition of mica alone, the relative elongation improves and that with the additional introduction of mixed magnesium aluminium oxide, the wear resistance of the material increases. The maximum wear resistance is shown by the composite containing 5 wt. % mica and 1 wt. % MAO. Investigations of the worn surface of composites have shown that the decrease in mass loss of composites is associated with the formation of a wear resistant surface formed during the asperity interaction. It is revealed that the formation of a wear resistant structure on the material's contact surface is caused by the formation of a secondary layer and chemical reactions that occur during interaction.

1. Introduction

Mica is a widespread rock-forming mineral that is mined in large volumes worldwide. Volumetric production is due to high demand and consumption in various industries (chemical and paint sectors, automotive, metallurgy and agriculture). Mica is characterised by high dielectric properties, refractoriness, resistance to sharp temperature fluctuations, and mechanical stability and is also featured by chemical persistence [1,2]. The ability to disperse into small particles and the above characteristics are of practical interest for the use of mica as fillers for polymers processed at high temperatures, such as polytetrafluoroethylene (PTFE) [3,4]. PTFE has a set of preferable properties necessary for creating parts on its base for tribological units of technical systems.

To obtain polymer composite materials (PCMs) based on PTFE, fillers such as coke, carbon fibres, molybdenum disulfide, aluminium and titanium oxide and cryptocrystalline graphite are most frequently used. The introduction of fillers leads to structural modification. For example, fibre fillers increase strength and stiffness. Dispersed fillers improve thermal conductivity and increase wear resistance. The tribological properties of composites depend on various factors. In general, fillers improve wear resistance by 10 to 10,000 times [5,6]. For example, Khedkar et al. [7] were the first to show a direct dependence of wear resistance on the thermal properties of PTFE-based composites. The dependence of the coefficient of friction of PTFE-based composites on the shape of the filler was studied by Mazur et al. [8], who demonstrated that the improvement of anti-wear properties largely depends on the filler shape. In a study of PTFE/silicon dioxide composites [9], Aderikha showed that the wear resistance of the composites largely depends on the material of the counter-body and the processes occurring on the contact surface, which depend on the composition of the tribological pair materials.

A number of works are devoted to the study of the transfer film and its effect on the wear resistance of polymer composites. The mechanisms of transfer film formation accompanying friction and their effect on wear resistance in general are studied and described in detail using the example of PTFE-based composites containing aluminium oxide [10-13]. Burris and Sawyer [10] note a sharp increase in the wear resistance of PTFE with the introduction of Al_2O_3 nanoparticles. Composites containing 1, 2, 5 and 10 wt. % of nano aluminium oxide, differing in size and modification of the crystalline phases, were studied. Al_2O_3 particles with sizes of 38 and 40 nm had a spherical shape and were a mixture of two modifications of the crystalline phases: γ (30 %) and δ (70 %) Al_2O_3. Al_2O_3 particles with sizes of 80 nm had an irregular shape and represented the α modification (99 %). It was shown that fillers with irregular particle shapes provided a significant reduction in wear rate in a steady-state mode compared to other PTFE-based nanocomposites. With the introduction of 1 wt. % of α-Al_2O_3 nanoparticles, wear resistance improved by 3000 times compared to unfilled PTFE.

In the work of Khedkar et al. [7], in the study of composites based on PTFE and carbon, molybdenum disulfide, glass fibre and graphite, it was shown that the composite containing 18 % carbon and 7 % graphite is characterised by wear resistance of less than 1×10^{-4} mm^3/Nm. This behaviour is explained by the presence of solid carbon particles in the volume of the composite, which impart additional strength. In the work of Lv et al. [14], the behaviour of PTFE/alumina and PTFE/silicon dioxide composites was studied. It was shown that the wear rate of the SiO_2/PTFE composite is lower than that of α-Al_2O_3/PTFE. In both cases, a tribofilm is formed, characterised by different structures and thicknesses. The difference in the formed tribofilm is explained by the tribochemistry of aluminium oxide and silicon dioxide nanoparticles. Aluminium oxide promotes the formation of a thicker and stronger tribofilm.

Layered silicates have found applications for filling polyolefins, polyamides, polyimides and other polymers. The main feature is the layered structure formed from structural units of tetrahedral and octahedral networks, which are connected to each other through oxygen ions. Depending on the ratio of these networks in the layer, minerals are designated as 1:1, 2:1, etc. Due to their structure, such fillers can delaminate within the polymer volume and improve consumer properties, even at low contents, typically less than 5 %. In addition to improving mechanical properties, improvements in thermal stability and gas permeability are noticed [15-17]. Simultaneous improvement in tensile strength and elastic modulus under flexural load is also noticed [18].

The primary purpose of filling polymers with mica is to improve the electrical properties of materials [19], to reduce the melting point of polyetherketoneketone [20] and to improve the thermal stability of polymers [21]. The effect of mica particle size on the polypropylene matrix was studied by Mihelčič et al. [22]. It was shown that mechanical properties improve with the addition of the smallest particle size of mica. For example, tensile strength increased by 27 %, flexural strength by 20 %, and the melting temperature of composites with smaller mica particles was increased by 6 °C.

The improvement of some properties invariably leads to the deterioration of others. Therefore, the search for fillers leading to optimal improvement of all characteristics remains a relevant problem. The combination of two or more fillers allows the improvement of properties in many directions. With this purpose, taking into account the previous studies [23-25], this study utilised the mixed oxide of magnesium and aluminium (hereafter referred to as MAO) as the second filler. In this context, the aim of this work is to study the effect of fillers, crushed natural mica and a mixture of magnesium and aluminium oxide, on the mechanical and tribological properties of polytetrafluoroethylene.

2. Materials and methods

Polytetrafluoroethylene (PTFE) PN-90 (HaloPolymer, Russia) was used in the manufacture of the samples. Natural mica and a mixture of magnesium and aluminium oxide were applied as fillers. The mica used in the research is potassium mica (Rikolatva deposit, Murmansk region). Hardness on the mineralogical scale is from 2.5 to 3 on the Mohs scale. Chemical analysis of mica was carried out at the Institute of Geology of Diamond and Precious Metals (Siberian Branch of the Russian Academy of Sciences, Russia). The analysis revealed the following composition, which is presented in Figure 1.

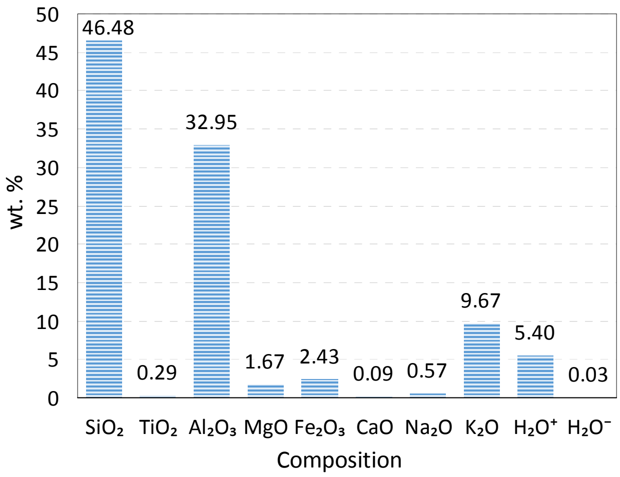


Figure 1. Chemical composition of mica

A mixture of magnesium and aluminium oxide with the general chemical formula $MgAl_2O_4$ is a dispersed powder of white colour, particle size between 60 – 80 nm, produced by mechanochemical method in the Institute of Solid State Chemistry and Mechanochemistry (Siberian Branch of the Russian Academy of Sciences, Russia).

To produce the samples, the initial mica was dried in a desiccator at 180 °C for 6 hours, and the PTFE was desiccated at 180 °C for 4 hours. A planetary-type ball mill Activator – 2S was used for grinding of mica. The acceleration was 80 g, the grinding time was 10 minutes, the powder-to-grinding balls ratio was 1:40 and the diameter of the balls was 10 mm. This mill is designed for fine grinding of solid materials due to shear and compression between the grinding balls in the milling cups and the motion of the planetary disc.

The composite samples were obtained by mixing polymer powder and fillers using a laboratory mixer (HT Machinery SM-3). The prepared powder compositions were further subjected to pressing in a closed mould at a pressure of 50 MPa. The obtained billet was further sintered in a muffle furnace at 375 °C. After the sintering stage, the composite samples were cooled to room temperature.

Mechanical parameters were determined in accordance with ISO 527-2. The method is based on stretching a material sample at a given speed. The tensile tests were conducted using a universal testing machine (Shimadzu Autograph AGS-J). The speed of grip expansion was 50 mm/min. The tests were carried out at a room temperature of 23 ± 2 °C. For mechanical testing, "dog-bone" shape samples were used with a total length of 80 mm, a working part length of 40 ± 0.5 mm and a thickness of 2 mm. The number of samples for one material was 10.

Tribological tests were carried out in accordance with ASTM G99, using a universal tribometer (CETR UMT-3) in the air at room temperature, with a pin-on-disc contact interaction scheme. The coefficient of friction and mass loss were determined. The counter-body was a disc made of steel 45 with the following parameters: hardness of 40 – 50 HRC and roughness *Ra* = 0.06 – 0.08 µm. The test sample was a cylinder with a height of 20 mm and a diameter of 10 mm. The test was carried out under a load of 160 N (2.04 MPa), with a rotation speed of 0.2 m/s for the lower sample (disc) and a test time of 3 hours. The mass of the sample is recorded before and after the test using a laboratory balance. The number of samples for one material was 6.

The morphological features of PCMs were investigated using a scanning electron microscope (Jeol JSM-7800F) equipped with an attachment for elemental analysis. In addition, after tribological tests, the worn surface was investigated by infrared spectroscopy (Varian) and optical microscopy (Olympus).

The Shore A hardness of the samples was determined according to ISO 868. The depth of indentation into the material of a given indenter under the action of force under specified conditions is measured. The thickness of the test specimen was 6 mm, and the number of repetitive tests for hardness measurements was 8.

3. Results and discussion

PCMs with different filler contents were produced as part of the study. Mica content was 0.5, 1, 2 and 5 wt. % and MAO content was 0.1, 0.5 and 1 wt. %. The mechanical and tribological properties of the obtained PCMs were analysed and structural studies were carried out.

3.1 Mechanical properties

The results of the mechanical properties of PCMs (tensile strength and elongation), depending on the filler content, are presented in Figures 2 and 3. The obtained results can be described as follows: with increasing filler concentration, the tensile strength decreases, which is most likely due to an increase in local tension concentrations within the material.

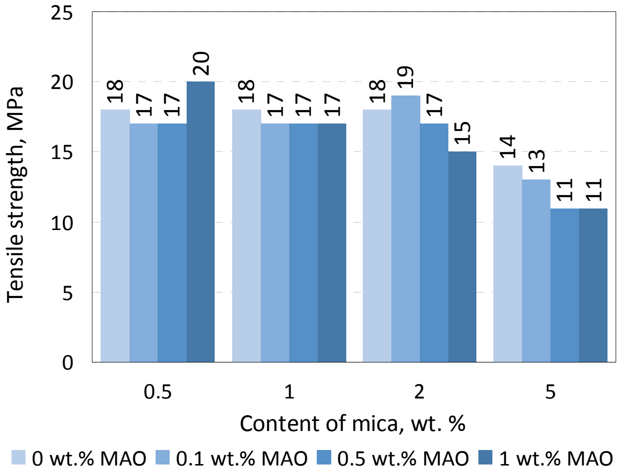


Figure 2. Tensile strength of PCMs, depending on the filler content

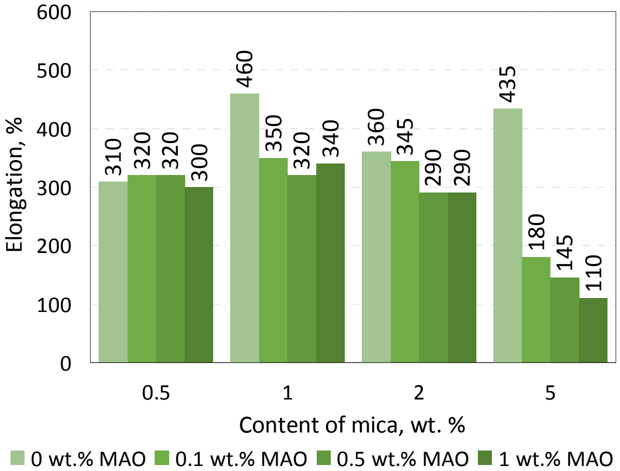


Figure 3. Elongation of PCMs, depending on the filler content

As the analysis of the graphs of changes in the tensile strength of PCMs, particularly at the content of 0.5 wt. % of mica and 1 wt. % MAO, the maximum value of the strength of the material is reached. Mica is characterised by a highly developed cleavage, which characterises the ability of the mineral to split along certain crystallographic directions, forming thin plates. Under the influence of a tensile force, mica nanosheets are displaced relative to each other with less force than is required for their delamination, which contributes to an increase in the elongation of the material. In addition, mica has a high length-to-thickness ratio (10:20) and a very high modulus of elasticity, which allows it to effectively perceive the load transmitted from the matrix through the polymer-filler interface, according to the mechanism of reinforcement of composites with short fibres. These properties contribute to the improvement of the polymer matrix. In the study by Gerasin et al. [26], it was found that the properties of composites based on layered silicates primarily depend on the position of polymer macromolecules in the interplanar space of silicates and their "retention" therein. Specifically, the properties depend on the position of the layered silicate tactoid within the material and the mobility of the polymer macromolecules relative to one another.

In view of the fact that mica has a perfectly smooth surface of layers, this property probably facilitates the mobility of PTFE macromolecules, and as a result, the values of elongation at break increase. The maximum values of elongation at break and tensile strength are achieved in PCMs with mica content from 0.5 to 2 wt. %. The elongation increased by 43 % and the wear resistance improved by 277 times compared to the original polymer. With the addition of MAO, the wear resistance is improved by 312 times, and the coefficient of friction is reduced by 38 %. Maximum values of elongation at break in PCM were with mica content of 1, 2 and 5 wt. %, without adding MAO. However, tensile strength decreases at 5 wt. % mica. Adding MAO to composites with mica results in a slight decrease in elongation at break, but a composite containing 2 wt. % mica and 0.1 wt. % MAO improves its values.

Tribological properties are improved with MAO content. PCMs with 2 and 5 wt. % mica without MAO have higher values of the coefficient of friction than with MAO. The maximum wear resistance is shown by a composite containing 5 wt. % mica and 1 wt. % MAO. This improvement is most likely due to the increased adhesion between the polymer and filler, which stimulates the processes of structure formation and leads to the formation of well-defined, densely packed structural components. The presence of these elements is confirmed with scanning electron microscopy, as shown in Figure 4.

This supramolecular structure contributes to the increase of the strength characteristics of PMCs by reducing the concentration of defective sites, pores and voids. From Figure 4, it can be seen that the supramolecular structure is characterised by homogeneity, the absence of defects, cracks and agglomerates of fillers.

Figure 5 shows the results of determining the Shore A hardness of composite samples. It is seen that the hardness of the composites increases monotonically with the increase of the filler content. This behaviour can be explained by the fact that the filler particles have a higher hardness than PTFE.

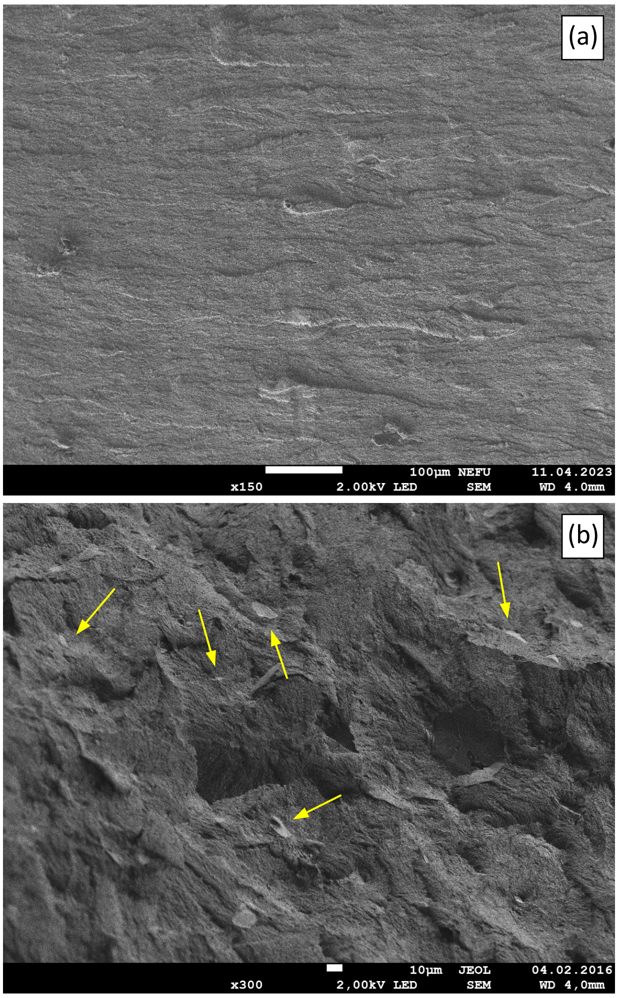


Figure 4. Micrograph of the supramolecular structure: (a) pristine PTFE and (b) composite containing 0.5 wt. % of mica and 1 wt. % of MAO; yellow arrows indicate filler particles

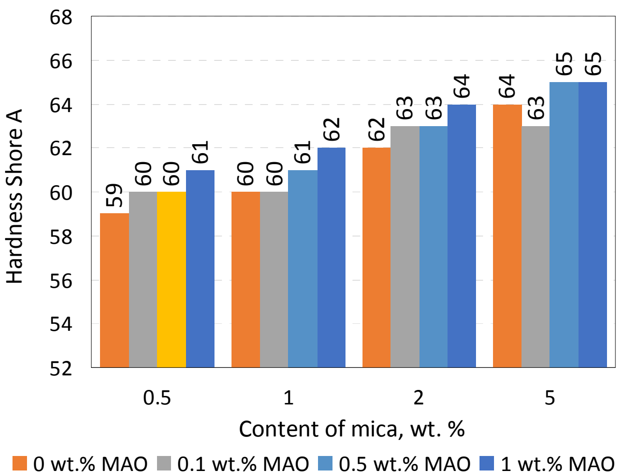


Figure 5. Shore A hardness of PCMs, depending on the filler content

3.2 Tribological properties

The results of tribological studies of PCMs (wear rate and coefficient of friction) depending on the filler content are presented in Figure 6. The wear rate largely depends on the amount of filler introduced, which means that, in this case, there is a direct interdependence between this indicator and the filler concentration. The wear rate of composites with a content of 0.5 – 1 wt. % mica and a change in the MAO content do not have a strict dependence but with a content of 2 – 5 wt. % mica, it tends to decrease. An increase in the MAO content leads to a decrease in the wear rate. Generally, an increase in filler content leads to the accumulation of filler particles on the contact surface, which participate in the formation of a durable coating that prevents destruction and wear. The coefficient of friction values of composites without MAO are higher than with the addition of MAO.

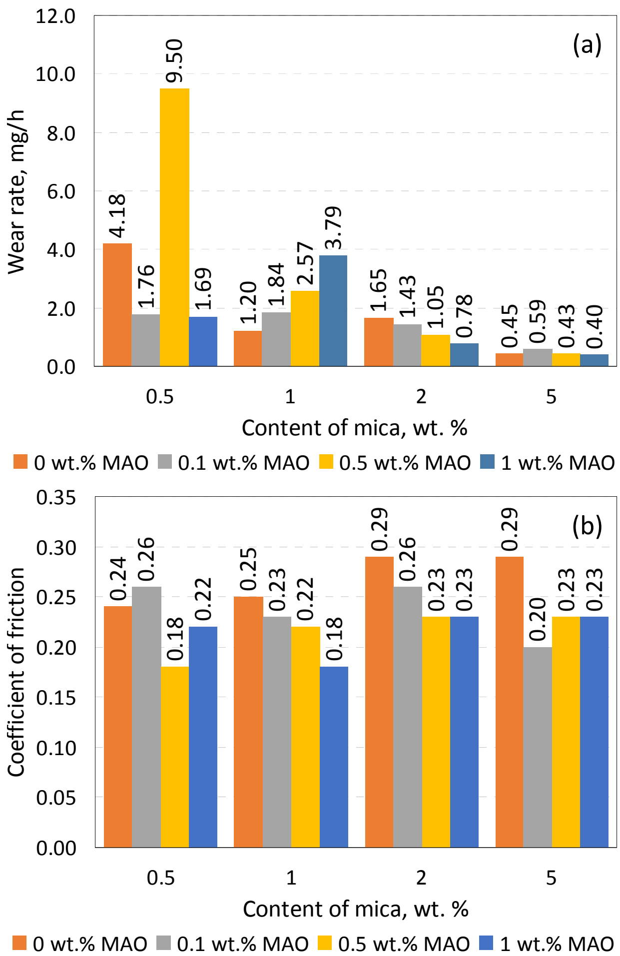


Figure 6. Tribological properties of PCMs, depending on the filler content: (a) wear rate and (b) coefficient of friction

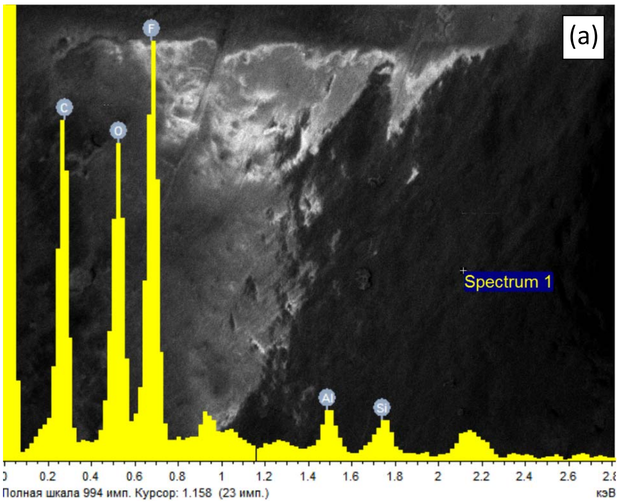
The wear resistance of composites containing only mica increases to a maximum of 277 times relative to unfilled PTFE (wear rate = 125 mg/h) with a content of 5 wt. % mica. This value is the maximum when comparing composites containing only mica (without MAO). When comparing all samples, the maximum wear resistance is achieved with a content of 5 wt. % mica and 1 wt. % MAO and increases to 312 times compared to unfilled PTFE. The highest wear resistance values are characteristic of composites containing 5 wt. % mica and different amounts of MAO. Even a content of 0.1 wt. % MAO affects the change in tribological characteristics. These data correspond to the results of Okhlopkova et al. [27], whose study demonstrated that the introduction of a small amount of ultradisperse fillers into PTFE significantly improves its wear resistance.

Changes in wear rates and coefficients of friction are correlated with each other, which can be attributed to the solid lubricating effect of fillers (mica) and the occurrence of tribochemical processes on the contact surface of materials. According to modern reasoning on polymer tribology, an increase in wear resistance is associated with the formation of secondary structures on the contact surfaces of composites. The secondary structures were formed from debris, filler particles and oxidised counter-body particles.

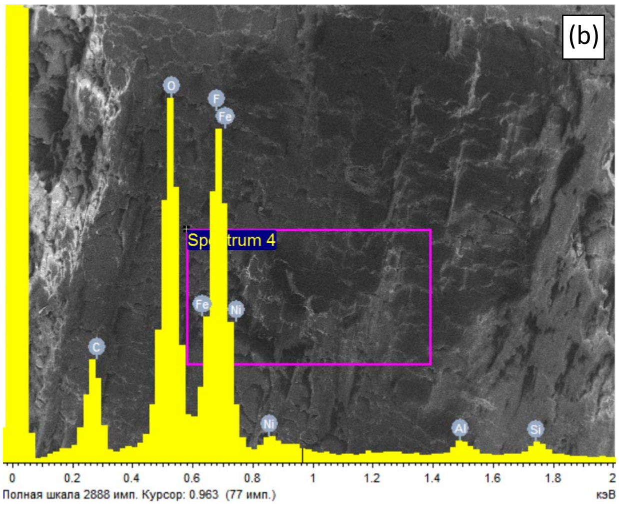
3.3 Worn surface analysis

The samples after tribological studies were further investigated by energy-dispersive analysis. The spectra obtained by this method are presented in Figure 7. The spectrum of the sample before testing is characterised by typical peaks for PTFE and fillers (presence of elements C, F, O, Si and Al). After the tribological tests, in addition to the existing peaks, the Fe and Ni peaks appear additionally, which may indicate the participation of the counter-body in the wear process. This fact may imply the wear of the counter-body. However, it is known from the literature that the metal-composite tribological pair, under the influence of specific factors (material transfer, mechanodegradation of polymers, thermo-oxidative degradation, tribochemical reactions, etc.), eventually form a wear-resistant layer that prevents deterioration of the material [28,29].

In the case of friction PCMs based on PTFE, mica and MAO, the oxidation of the counter-body metal influences the change in the structure of the worn surface of the sample. Since the filler particles increase the abrasion resistance, tribooxidative processes proceed, as well as a significant contribution is made by heat generation during friction, all of which leads to the process of cross-linking of fragments of thermo-oxidative decomposition of macromolecules. As a result, a densely packed structure characterised by high strength and high wear resistance is created.



Element	C	O	F	Al	Si
wt. %	28.22	21.73	45.17	2.58	2.30



Element	C	O	F	Al	Si	Fe	Ni
wt. %	11.66	21.24	28.09	0.79	0.76	34.38	3.07

Figure 7. Elemental analysis of surface for composite with 2 wt. % mica and 0.1 wt. % MAO: (a) before testing and (b) after testing

A microphotograph of the worn surface of a PTFE-based composite containing 2 wt. % mica and 0.5 wt. % MAO obtained by SEM is presented in Figure 8. The presence of Fe traces on the contact surface of the composite may be a consequence of metal corrosion. Contact corrosion is known to occur during metal friction [30,31]. It can be assumed that the change in the coefficient of friction and wear rate of PTFE modified with crushed mica can also be associated with the formation of iron oxides on the contacting surfaces.

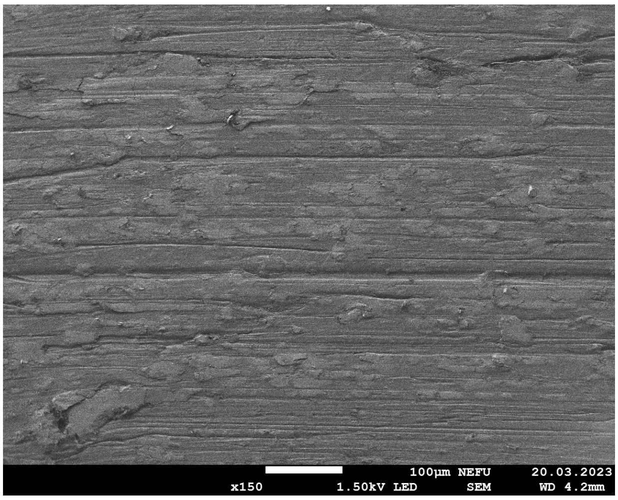


Figure 8. Worn surface of composite with 2 wt. % mica and 0.5 wt. % MAO

To confirm the occurrence of tribooxidative processes, infrared (IR) spectroscopy studies were carried out, the results of which are presented in Figure 9. The IR spectra of the samples were taken on a Fourier transform infrared stepped-scan spectrometer. For comparison, the samples were analysed before and after testing. The IR spectrum of PTFE consists of valence vibrations of CF_2 groups (1211 and 1154 cm^{-1}) and υ(CC) group vibrations. In the region below 650 cm^{-1}, wagging vibrations of $\gamma\omega(CF_2)$ are evident at 639 cm^{-1}; the 555 cm^{-1} band characterise the deformation and rocking vibrations of CF_2 groups [32].

During friction, surfaces undergo chemical activation: polymer molecules are broken along the main valence bonds and side groups are detached, forming active centres such as free microradicals, ion-radicals and other intermediate particles. This process is caused not only by mechanical degradation but also by thermal degradation caused by intensive heat generation in the contact zone. For example, the analysis shows a decrease in the molecular weight of the polymer, signs of oxidation (according to IR spectroscopy), as well as the destruction of the crystalline structure of the material, resulting in a transition to an amorphous state. The key practical consequence of these processes is the accumulation of fatigue damage in the material. Under repeated deformation, this leads to gradual wear of the surface layer, which determines the durability and reliability of polymer systems under friction [7].

The degradation of PTFE during friction can occur with the involvement of different mechanisms. The conditions of the technological process, which, as a rule, takes place in the presence of atmospheric oxygen, indicate the possibility of oxidation of the material during friction. Under such conditions, additional bands can be expected to appear in the infrared spectra: at 1710 cm^{-1} (carbonyl groups HC=O), in the interval 1880 – 1890 cm^{-1} (groups FC=O), at frequencies 1810 and 1776 cm^{-1} (corresponding to vibrations of free and bound carboxyl groups COOH), as well as in the region 3000 – 3400 cm^{-1} (vibrations of hydroxyl groups OH) [32,33]. The appearance of peaks of hydroxyl groups indicates possible oxidation of the composite surface during friction. The resulting carbonyl and carboxyl groups, due to their low stability, are further capable of forming carboxylic acid salts when interacting with metal cations of the filler or counter-body material.

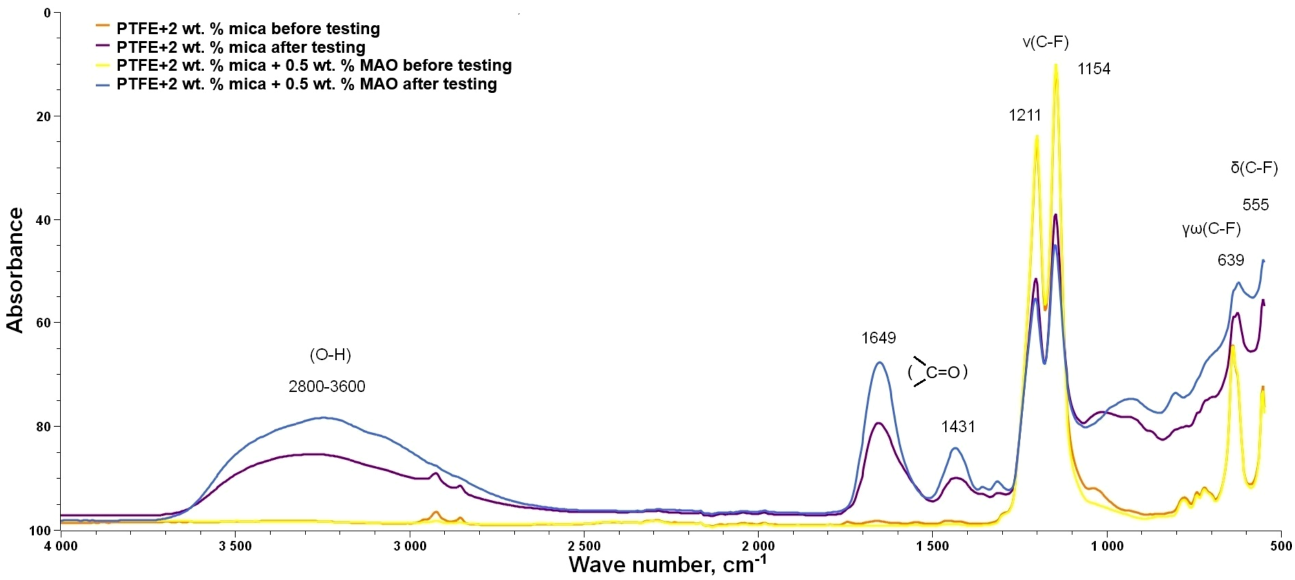


Figure 9. IR spectra of composite samples before and after testing

According to the previous study [34], metal at the initial stages of polymer material processing activates oxidative reactions that contribute to the generation of carboxylate anions. Subsequently, the metal interacts with these anions to form salt compounds that begin to function as oxidation inhibitors. During further heat treatment of the polymer, cross-linking of macromolecule fragments formed as a result of thermooxidative decomposition is noticed, which leads to the formation of a dense ordered structure with increased resistance to wear. In the IR spectra of composites in the range of 1680 – 1400 cm^{-1}, two characteristic peaks associated with fragments of carboxylic acid salts are recorded. The results of the IR spectra of PCMs confirm the formation of carboxylic acid salts and possible cross-linking of individual fragments of macromolecules through the formation of ether groups observed in the 1200 – 1300 cm^{-1} spectral region [35]. The cross-linked structure contributes to the wear resistance, which affects the durability of the material.

4. Conclusion

Composites based on PTFE filled with crushed mica and magnesium aluminium oxide have been investigated. It has been established that high wear resistance is possible with optimal complex filling of mica and MAO. For improved mechanical properties, the introduction of up to 2 wt. % of mica is preferable. The wear resistance of composites also depends on the degree of filling. The maximum wear resistance is achieved with 5 wt. % mica and 1 wt. % MAO.

Therefore, it is shown that structural changes in the material, the interaction of components and the formation of a protective layer that reduces friction and protects the material from further degradation contribute to improving the wear resistance of the material, making it more durable and resistant to mechanical impacts.

Acknowledgement

This research was funded by the Ministry of Science and Higher Education of the Russian Federation (Grant Number FSRG-2024-0004).

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