

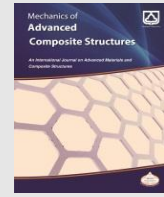


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Research Article

Climatic Testing of Polytetrafluoroethylene-Based Composites

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ABSTRACT

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This study investigates the effects of accelerated climatic cycling ($-50\text{ }^{\circ}\text{C}$ to $+100\text{ }^{\circ}\text{C}$, 10 cycles) on polytetrafluoroethylene (PTFE) composites filled with carbon fibers, carbon nanotubes (CNT), kaolinite (K), silicon dioxide SiO_2 , titanium carbide (TiC), and titanium diboride (TiB_2). Mechanical tests revealed that unfilled PTFE lost $\sim 20\%$ of its tensile strength after testing, while composites with micrometer-sized fillers (CF, TiC, TiB_2) degraded by $\sim 30\%$. In contrast, the PTFE+CNT+K composite retained its tensile strength and modulus of elasticity without significant change, demonstrating superior stability. Structural analysis showed that nanofillers (CNT, K, SiO_2) promote the formation of dense spherulite-like structures, which reduce the coefficient of linear thermal expansion (CLTE) by up to 45% compared to neat PTFE. Sealing performance tests at $-40\text{ }^{\circ}\text{C}$ confirmed that PTFE+CNT+K and PTFE+ SiO_2 composites maintained effective sealing, whereas neat PTFE and PTFE+K composites exhibited significant leakage. The results highlight the PTFE+CNT+K composite as the most promising material for sealing applications in arctic conditions due to its balanced mechanical integrity, low CLTE, and reliable performance under extreme thermal cycling.

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1. Introduction

Currently, there is a significant demand for the development of materials for Arctic conditions that provide maximum wear resistance and long service life of friction unit parts [1]. This task is particularly important for mining, road building, earthmoving, and municipal machinery, where volumetric hydraulic drives with hydraulic cylinders as actuators are widely used [2]. Usually, one of the

serious problems in the operation of hydraulic cylinders is leakage in the hydraulic system, which can be divided into external and internal leakage. External leaks can be detected by traces of hydraulic oil, while internal leaks are caused by wear of hydraulic system components and improper selection of sealing devices. Internal leaks may go undetected, causing additional operational problems. Numerous studies of the technique in real operating conditions have

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shown that the total percentage of hydraulic cylinder failures reaches 80%, and the main cause is depressurization of sealing units: 52% in rod units and 40% in piston units [3]. This problem is particularly acute in winter when the materials used fail under low temperature stress. Consequently, one of the promising directions for improving the reliability of equipment operating in cold climates is to replace traditional sealing materials with frost-resistant composites.

Polytetrafluoroethylene (PTFE) and polymer composite materials (PCMs) based on it are widely used in seals and mating parts for friction units and sealing systems in machinery and equipment due to their unique combination of antifriction and thermal properties [4]. PTFE exhibits exceptional heat and frost resistance, chemical inertness, and a low coefficient of friction. However, PTFE has several drawbacks that necessitate the development of reinforced PCMs, including poor wear resistance, a high coefficient of linear thermal expansion, and a tendency to deform under low loads even at ambient conditions. Therefore, the task of improving the mechanical properties and wear resistance of PTFE, especially under conditions of hard friction, remains a critical research challenge. The introduction of solid fillers into PTFE generally improves its thermal, tribological, and mechanical properties [5, 6]. A wide range of fillers, including fibers and dispersed particles, has been explored for this purpose. The effects of different fillers (carbon fibers [7], carbon nanotubes [8], kaolinite [9], silicon dioxide [10], titanium diboride, and titanium carbide [11]) on the structural and mechanical properties of PTFE have been extensively studied. The positive effect of the above fillers on the mechanical and tribological properties of PTFE was shown. However, these studies were carried out using standard testing methods for polymeric materials, which poorly reflect their behavior when tested at low temperatures and especially after several cycles of exposure to heat and cold. Polymers are known to be sensitive to UV radiation, microorganisms, high humidity, and temperature stress [12]. These factors can significantly degrade the performance properties of polymers [13]. Among these factors, temperature and environment are the most significant influencing factors in cold climates. Thus, in the winter period, the impact of microorganisms and high humidity is practically absent, and the parts of the friction units are usually located in the housing and are not so exposed to direct UV radiation.

Despite numerous publications on the thermal properties of PTFE and PCMs, obtaining reliable data about polymer behavior at low temperatures remains challenging even for

specialists. This difficulty arises because results from individual studies are often incomparable and appear largely random, particularly for composite materials. Nevertheless, PTFE remains one of the most extensively studied crystalline polyolefins, alongside polyethylene, polypropylene, and polyalkylmethacrylates. Researchers, including Blumm J., Araki Y., Andena L., and Subrahmanyam H.N. [14-17] have investigated PTFE's thermal expansion across wide temperature ranges. Among key climatic factors, temperature exerts the most significant influence, causing substantial changes in material properties through cyclic effects. These temperature fluctuations generate periodic internal stress variations that accelerate material aging via microcrack formation in the polymer matrix [18]. The methodology for reliable assessment of such aging under extreme conditions remains a critical challenge, as highlighted in recent reviews on climatic testing of polymers for arctic applications [19]. Furthermore, while the enhancement of PTFE's thermal stability through nanofillers like boron nitride has been demonstrated [20], studies focusing on the combined effect of cyclic thermal stress and filler composition on the mechanical and sealing performance of PTFE composites in sub-zero environments are still limited. Therefore, experimental determination of how cyclic temperature variations affect mechanical properties and how filler composition influences seasonal changes in sealing performance becomes particularly important. However, most existing studies focus on standard laboratory tests, leaving a gap in understanding the behavior of PTFE composites under simulated extreme climatic cycling, particularly in the context of sealing applications for arctic conditions. This work aims to fill this gap by investigating the effects of accelerated climatic testing (from -50 °C to +100 °C) on the mechanical, structural, and sealing properties of PTFE composites filled with various micro- and nanoscale fillers, including a novel combination of carbon nanotubes and kaolinite. The correlation between filler-induced structural changes (crystallinity, supramolecular morphology) and the retention of sealing ability at subzero temperatures represents a key novelty of this study. Unlike most previous studies, which focused on standard tribological or mechanical tests at room temperature [7-11] or on thermal stability without cyclic exposure [14-17, 20], this work is the first to systematically evaluate the effect of accelerated climate cycling (10 cycles of -50 to +100 °C) on the mechanical, structural, and sealing properties of PTFE composites, including a new combination of CNT + kaolinite, in the context of Arctic conditions.

2. Materials and Methods

Polytetrafluoroethylene of grade PN90 (HaloPolymer, Russia) with an average particle size of 90 μm and density of 2.16 g/cm^3 was used as a polymer matrix. Carbon fibers, kaolinite, single-walled carbon nanotubes, silicon dioxide, titanium carbide, and diboride were used as fillers:

- Carbon fiber (CF) manufactured by R&G Faserverbundwerkstoffe GmbH, Germany. Fiber length is 200 μm , thickness is 7 μm , and density is 1.7-2.0 g/cm^3 [7].

- Kaolinite (K) of Glukhovets deposit ("AKW UkrainianKaolinCompany", Ukraine), grade KBE-1, chemical composition - $\text{Al}_4[\text{Si}_4\text{O}_{10}](\text{OH})_8$, specific surface area of 35 m^2/g , average particle size is 33 nm [9].

- Single-walled carbon nanotubes (CNT) of the TUBALL brand manufactured by LLC "PCT" (Russia). Composition: TUBALL nanotubes - 75%, metal impurities \leq 15% [8].

- Silicon dioxide (SiO_2) of "Covelos 35/5" brand (Russia). It is a white loose powder with an average particle size of \sim 7 μm , a specific surface area of 300 m^2/g , and a bulk density of 0.085 g/cm^3 [10].

- Titanium carbide (TiC, average particle size is \sim 20 μm) and titanium diboride (TiB_2 , average particle size is \sim 10 μm) produced by self-propagating high-temperature synthesis at the Institute of Structural Macrokinetics and Materials Science Problems of the Russian Academy of Sciences, Chernogolovka [11].

The filler contents were selected based on previous studies by the authors and literature data, which demonstrated optimal improvements in mechanical and tribological properties without excessive agglomeration or degradation of processability [7-11].

The dispersed PTFE powder was dried in the drying cabinet PE-0041 at 180 $^\circ\text{C}$ for 4 h before making the polymer composite blend. The dried polymer was dispersed and sieved through a 250 μm mesh sieve to obtain a homogeneous powder. PTFE and composites based on it with different content of fillers (CF - 5 wt.%, SiO_2 - 2 wt.%, kaolinite - 1 wt.%, CNT - 0.5 wt.%, TiC - 0.5 wt.%, TiB_2 - 0.5 wt.%) were processed according to the standard technology, including mixing of dry components in a paddle mixer and subsequent cold molding at a pressure of 50 MPa. After molding, the samples were sintered at 375 $^\circ\text{C}$ and cooled to room temperature.

Climatic testing of samples was carried out using a KTH-150 climatic test chamber (NPF Technologiya, Russia). The temperature chamber maintains temperature with an accuracy of \pm 0.5 $^\circ\text{C}$. The samples of polymer composites were kept in the chamber at cyclic temperature differences from -50 $^\circ\text{C}$ to +100 $^\circ\text{C}$, with a total of 10 cycles.

Climatic tests were performed without active humidity control; relative humidity corresponded to ambient laboratory conditions (typically 40-60% RH at room temperature between cycles). The number of cycles (10) was selected based on an accelerated methodology for Arctic conditions [ASTM D6944; ISO 11346], where one cycle corresponds to sharp daily/seasonal temperature fluctuations. This regime identifies initial degradation stages and relative material stability over a limited period — a standard approach for screening studies. Extrapolation to service life requires additional long-term tests (100+ cycles), which is the subject of further research. This limitation reduces applicability for predicting multi-year service but confirms the superiority of PTFE+CNT+K in the initial phase.

Before and after climatic testing, Type 1BA blade samples underwent mechanical testing on a Shimadzu Autograph AGS-J universal testing machine (Japan). Tensile strength, elongation at break, and tensile modulus of elasticity were determined in accordance with ISO 527-2:2012 (the number of samples per test is 10). The permissible error of the study is up to \pm 10% deviation. Statistical processing of experimental data based on the research results was performed using standard methods of mathematical statistics, determining the values of the sample standard deviation and the boundaries of confidence intervals using Student's criterion at a reliability level of 0.95.

The thermodynamic parameters of the composites were investigated on a DSC 204 F1 Phoenix differential scanning calorimeter (NETZSCH, Germany). To estimate the contribution due to the interaction at the interfacial boundary, the enthalpy of melting ΔH_m was recorded, and the degree of crystallinity α of the samples was determined in the temperature range from -50 to 380 $^\circ\text{C}$ in a helium atmosphere at a gas flow of 30 ml/min, with a constant heating rate of 20 K/min.

IR spectra of samples were recorded using an FT-IR 7000 step-scan spectrometer (Varian, USA) with the attenuated total reflection (ATR) method using a Pike MIRacle ATR attachment with a diamond/ZnSe prism in the frequency range 550-4000 cm^{-1} at a resolution of 2 cm^{-1} and the number of scans per spectrum of 16 scans. The IR spectra were identified and analyzed according to known methods using the spectrometer library and bibliographic data of IR spectra of polymers.

The method of thermomechanical analysis was used to study the average coefficient of linear thermal expansion of composites using a Shimadzu TMA-60 (Japan) in accordance with Russian Standard GOST 32618.2-2014 in the

temperature range from -50 to $+50$ °C. The average coefficient of linear thermal expansion of PCMs was calculated by formula (1) using the obtained TMA curves:

$$\alpha = \Delta L / \Delta T \quad (1),$$

where α is the average coefficient of linear thermal expansion, deg^{-1} ; ΔL - is the change in the length of the test specimen within the temperature range, mm; ΔT - is the temperature increment from T_1 to T_2 equal to $T_2 - T_1$, °C.

Brittle chips of PCMs were studied on a scanning electron microscope JSM-7800F (JEOL, Japan). Low-temperature brittle chips were prepared by fracturing samples at the temperature of liquid nitrogen, -196 °C.

Field tests were carried out on a test bench for testing of sealing materials (Patent for useful model of the Russian Federation №223498). The scheme and photograph of the test bench are presented in Fig. 1.

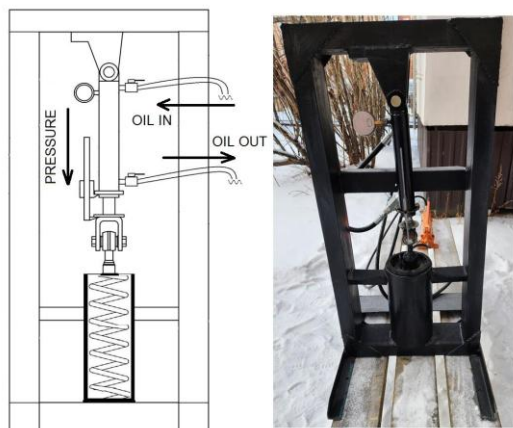


Fig. 1. Scheme and photograph of the test bench

The all-season hydraulic oil with various additives, including antifoaming and antioxidants, corresponding to class 5 (designation according to Russian Standard GOST 17479.9-85 – MG15-Vi) was used as the oil. The tests were carried out in full-scale conditions at constant pressure set on the piston, and the change in the height of the rod was measured with a caliper. In wintertime, at -40 °C, the pressure (P) on the sample was set to 300 N. The dwell time on the sample stand was 15 min. Experimental data were processed in V-t coordinates. Each point is the average value of 3 specimens. Period of testing on the test bench: winter 2023 (Yakutsk). Location: Yakutsk, territory of the North-Eastern Federal University (coordinates 62.017677° , 129.704864°).

3. Results and Discussion

The climatic studies were carried out in a heat-cold chamber, which is designed for research and testing the quality of various materials, products, components, assemblies,

structures, mechanisms, measuring devices, etc., on the impact of reduced and increased temperatures. The resistance of the material to cyclic exposure to temperatures was determined by the change in the current value of the characteristic from the initial one. Fig. 2 presents the mechanical properties of PTFE-based polymer composites before and after climatic testing in a heat-cold chamber.

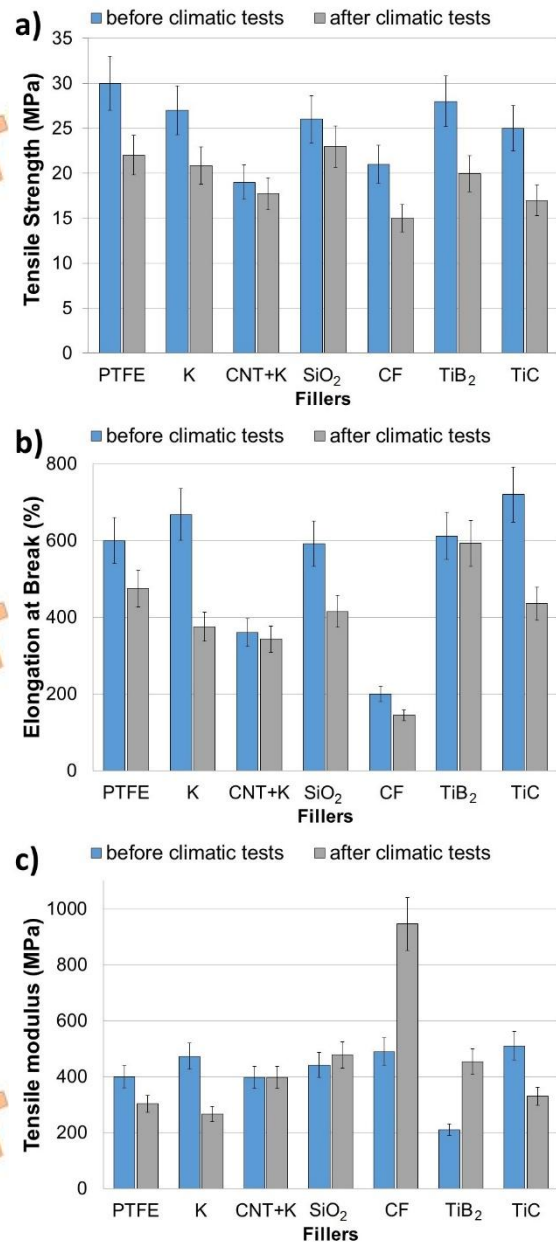


Fig. 2. Mechanical properties of PCMs before and after climatic tests: a) tensile strength; b) elongation at break; c) tensile modulus of elasticity

Fig. 2 shows that the tensile strength and elongation at break of the initial PTFE decrease by 20% after accelerated climatic tests. The cyclic climatic tests have different effects on the mechanical properties of PCMs, which depend on the nature of the filler used. It can be seen that in composites containing kaolin, CNT+K and SiO₂ the tensile strength is within the confidence

interval of the measurement result error, i.e. it remains at the level of the unaged material. Elongation at break of PCMs does not change significantly, so in PTFE+CNT+K it remains at the level of the unaged material, and in composites with kaolin and SiO₂ it decreases by 45%. For PTFE+CF, PTFE+TiC and PTFE+TiB₂ composites, the tensile strength decreases by 30% relative to their pre-aged state. Thus, under temperature cycling, micrometer-sized particles have a stronger effect on properties than nanosized particles in PCMs. The tensile modulus of elasticity decreases from 25 to 40% for PTFE and composites having kaolinite and titanium carbide in their composition, and increases by 10 to 115% for composite samples containing CF and titanium diboride (Fig. 2, c). In PCMs with silica, as in the sample with CNT and kaolinite, the value of elastic modulus remains unchanged. PTFE and PCMs based on it retain their performance characteristics in a wide temperature range [17]. However, the climatic testing shows that at cyclic temperature changes from minus 50 °C to plus 100 °C the mechanical properties of materials decrease. The decrease in the strength characteristics of PTFE and PCMs based on it during tensile testing may indicate the occurrence of degradation processes in the polymer and the appearance of defective areas in the material [21-23]. Comparison of the results of changes in the mechanical properties of PCM indicates that the composite containing CNT and kaolinite is the most resistant to climatic tests. Thus, the obtained data of studies of mechanical characteristics indicate that despite the high temperature resistance of PTFE, polymer composites based on it are subject to some decrease in characteristics under the influence of cyclic cooling and heating. These studies confirm the fact that depending on the type of filler there is a dependence of PCMs properties at negative temperatures. Depending on the chemical nature of the filler, its dispersibility and specific surface area of particles, distribution in the matrix, structurization processes in PTFE proceed differently.

Further, the change in the morphology of the PTFE supramolecular structure depending on the nature of the filler will be presented. SEM images of the supramolecular structure of PTFE and PCMs based on it are illustrated in Fig. 3.

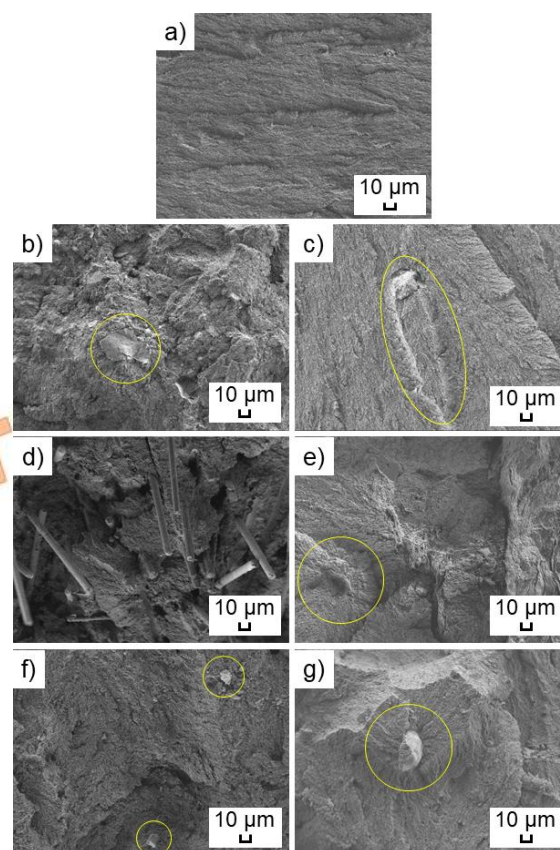


Fig. 3. Supramolecular structure of PTFE and PCMs: a) initial PTFE; b) kaolinite; c) silicon dioxide; d) CF; e) CNT and kaolinite; f) titanium diboride; g) titanium carbide

Fig. 3 demonstrates that the supramolecular structure of the initial PTFE is characterized as lamellar. The introduction of fillers into PTFE causes a structural transformation from lamellar to spherulite-like morphology [24]. Studies of the supramolecular structure of PCMs reveal that filler particles serve as crystallization centers (Fig. 3 b-c, e-g), from which the growth of spherulite-like structures proceeds (highlighted in yellow circle). As a result of dry mixing of fillers with the polymer matrix, pressing and heat treatment, active segments of PTFE macromolecules adsorb on the filler surface, causing a certain orientation of macromolecules in the amorphous phase, which leads to a change in the ratio between the crystalline and amorphous components of the matrix [25]. From Fig. 3d, it can be seen that the carbon fibers in the supramolecular structure of PTFE are distributed and oriented chaotically. Defective areas in the supramolecular structure of PTFE+CF can be seen, consisting of the detachment of fibers from the polymer matrix due to low adhesion between them. Due to this fact, there is a decrease in the mechanical properties of PCMs with carbon fibers. It should be noted that after climatic studies of PTFE+CF, there is a decrease in strength, but the decrease is not critical, as well as virtually no change in elongation at break. It is known that after sintering of PTFE, there are no

irreversible deformations at temperatures up to 130 °C, and the thermal expansion of the material has a linear character [26]. At the same time, [27] reported that thermal exposure causes residual changes in the parameters of the crystal structure, which are retained after testing due to the memory effect of thermal expansion. Based on this, it can be assumed that exposure to temperature, including cyclic exposure, has a negative effect on the strength properties of micrometer-sized PCMs due to the accumulative effect of increasing the number of defects in the structure, but the deformation properties are preserved, possibly due to the high crystallinity of the polymer matrix.

To explain the mechanical properties of materials and to prove the changes occurring in the structure of composites under the influence of cyclic cooling and heating, we calculated the degree of crystallinity of PTFE and PCMs using DSC and XRD methods (Table 1).

Table 1. Degree of crystallinity of composites before and after accelerated climatic testing

Sample	α_{XRD} , %		α_{DSC} , %	
	before	after	before	after
neat PTFE	64 ± 1	60 ± 2	40 ± 2	52 ± 1
PTFE+CF	74 ± 2	71 ± 3	52 ± 2	59 ± 1
PTFE+K	65 ± 1	61 ± 1	45 ± 2	49 ± 2
PTFE+TiC	70 ± 2	64 ± 1	45 ± 1	48 ± 1
PTFE+TiB ₂	70 ± 1	67 ± 2	39 ± 2	69 ± 2
PTFE+CNT+K	72 ± 2	66 ± 1	51 ± 1	49 ± 2
PTFE+SiO ₂	69 ± 2	63 ± 1	50 ± 1	52 ± 1

The discrepancies in crystallinity values obtained by XRD and DSC methods can be attributed to the specific principles of each technique. XRD reflects the long-range order and regularity of the crystal lattice, whereas DSC detects thermal effects of melting, which depend on the size and perfection of crystallites. An increase in α_{DSC} accompanied by a decrease in α_{XRD} is typical for physical aging and recrystallization processes in semi-crystalline polymers under cyclic thermal exposure. DSC is more sensitive to changes in the perfection and size of lamellae/crystallites: the formation of smaller but more ordered crystallites (secondary crystallization) increases the melting enthalpy, even if the total proportion of the crystalline phase in the long order (XRD) decreases due to defects and an increase in the amorphous fraction. Similar effects have been observed previously during the thermal aging of other fluoropolymers and polyolefins [25]. Thus, DSC

and XRD data complement each other, describing different structural changes in the material.

As shown in Table 1, the degree of crystallinity calculated by the XRD method in all samples decreases after accelerated climatic tests, i.e., the proportion of amorphous phase in the samples increases. It is shown that the degree of crystallinity decreased by 14% after climatic testing of the initial PTFE. In the case of composites, the degree of crystallinity determined by XRD decreases by 3–6% relative to the initial sample not subjected to accelerated climatic tests.

At the same time, the degree of crystallinity calculated by the DSC method shows that in most composites, this crystallinity increases, except for PCMs filled with silicon dioxide and carbon nanotubes together with kaolin. Preservation of the degree of crystallinity is probably due to the fact that the tensile strength and modulus of elasticity of the two composites remain at the same level, i.e., there were no significant changes in their structure.

To evaluate the influence of climatic factors, IR spectroscopy studies of PTFE and PCMs based on it were carried out. Fig. 4 shows the IR spectra of the samples before and after the climatic testing.

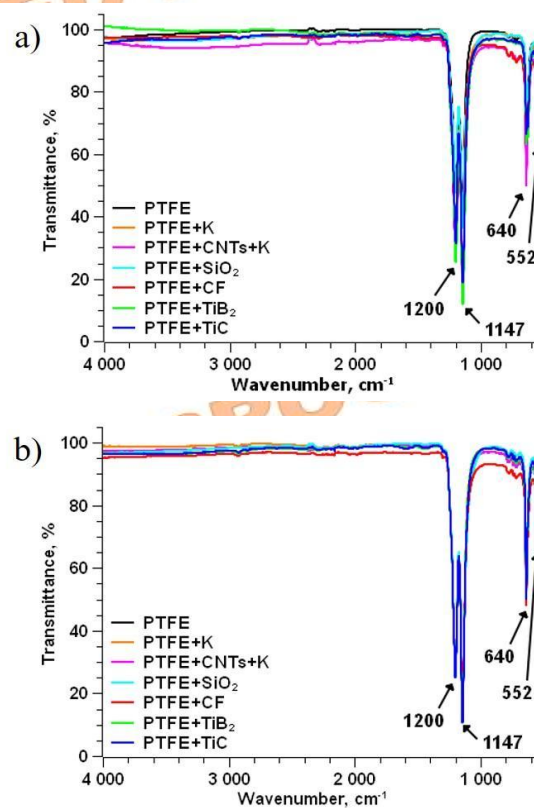


Fig. 4. IR spectra of samples (a) before and (b) after climatic testing

In all IR spectra, the most intense are those related to the stretching vibrations of CF₂-groups (1200 and 1147 cm⁻¹). In the low wavenumber

region at 650 cm^{-1} , wagging (640 cm^{-1}) and deformation (552 cm^{-1}) vibrations of CF_2 -groups were recorded. The bands observed in the $800\text{--}700\text{ cm}^{-1}$ range correspond to molecular chain vibrations in the PTFE amorphous phase [28]. In this region, there were no changes in the IR spectra, except for an increase in the intensities of these peaks after the climatic tests. After climatic tests, no new peaks appeared in the samples, and the absorption bands related to PTFE were retained. Thus, IR analysis confirmed that climatic testing did not lead to significant chemical changes in the polymer matrix, indicating that physical degradation processes (defect formation, recrystallization) prevailed over chemical destruction.

Fig. 5 presents the results of experimental tests of each sample depending on the ambient temperature in winter and spring-summer.

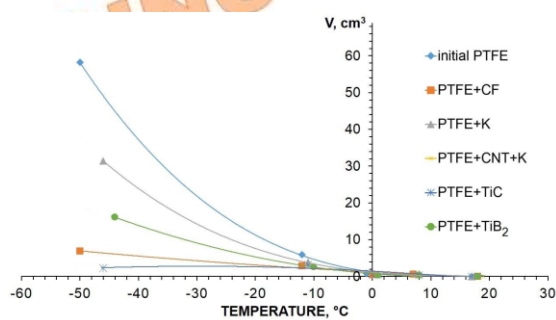


Fig. 5. Results of oil leakage measurement in hydraulic cylinder depending on temperature and PCMs composition

Fig. 5 demonstrates that at positive and around zero temperature, the samples of initial PTFE and PCMs have negligible oil leakage in the hydraulic cylinder. When the ambient temperature drops to -50°C , significant changes in this indicator are manifested. Thus, at temperatures below -40°C the results with a large scatter of values are obtained when measuring the leakage volume on the experimental bench. It was found that a large leakage was observed for the initial PTFE and composites PTFE+K and PTFE+TiB₂, while other composites maintained effective sealing. The trend lines indicate that there is a sharp decrease in leakage with increasing ambient temperature. This is because better sealing occurs at relatively high temperatures.

The sealing test results were analyzed by determining the materials' linear thermal expansion coefficients through thermomechanical analysis. It is known that structural changes cause changes in temperature characteristics [29]. Table 2 shows the results of the study of the coefficient of linear thermal expansion (CLTE) in the temperature range from -50 to $+50^\circ\text{C}$ and the density of the initial PTFE and PCMs based on it.

Table 2. Results of the TMA study of initial PTFE and PCMs

Sample	CLTE, $\times 10^{-6}\text{ K}^{-1}$	Density, g/cm^3
neat PTFE	130	2.16
PTFE+CF	142	2.17
PTFE+K	71	2.20
PTFE+TiC	110	2.21
PTFE+TiB ₂	120	2.24
PTFE+CNT+K	96	2.16
PTFE+SiO ₂	87	2.15

From the analysis of linear thermal expansion of PCMs, it can be seen that there is a decrease in this parameter for composites except for PTFE+CF. In the case of PTFE+CF, the value of CLTE increased by $\sim 9\%$ relative to the polymer matrix, which is probably due to the weakening of intermolecular interaction and a large ratio of diameter to height of fibers, as well as chaotic orientation of fibers in the supramolecular structure of the material. In the case of PCM with micro-sized TiC and TiB₂ fillers, a decrease of $\sim 15\%$ and $\sim 7\%$ in CLTE relative to the initial PTFE is observed. The minimum values of CLTE were obtained for composites of PTFE+SiO₂, PTFE+K, and PTFE+CNT+K, which are $\sim 33\%$, $\sim 45\%$, and $\sim 26\%$ lower than the initial polymer. It can be explained by the fact that nanosized fillers structure the material with the formation of spherulite-like formations in the supramolecular structure of the polymer (Fig. 3), with denser packing of structural elements, which positively affects the values of CLTE. Another reason may be the higher adhesive interaction between the polymer and fillers, leading to high monolithicity of PCMs (Table 2), which is confirmed by the higher density and lower porosity of the samples [7]. Thus, based on the reasons described above, we can say that the addition of nanoparticles to PTFE has a positive effect on the performance characteristic - sealing ability of the material.

The data obtained allow us to establish a correlation between the microstructure of composites and their performance characteristics. Nanoscale fillers (CNT, K, SiO₂) contribute to the formation of dense spherulitic structures (Fig. 3), which leads to a decrease in CLTR (Table 2) and increases the material's resistance to thermal cycling. It is precisely the low CLTE that is the key factor in maintaining tightness at -40°C (Fig. 5), as it minimizes thermal deformation and gaps in the sealing assembly. In contrast, composites with micrometer-sized fillers (CF, TiC, TiB₂) exhibit higher CLTE and a tendency to form defects at the phase boundary, which explains their increased oil leakage at low temperatures.

Kaolinite without CNTs is prone to agglomeration, resulting in a non-uniform distribution in the matrix and the formation of weak zones. In the CNT+K combination, nanotubes prevent kaolinite agglomeration and promote the formation of a more uniform and continuous spherulitic network (Fig. 3e), ensuring a consistently low CLTE and maintaining seal contact.

It is noteworthy that at -40°C , all materials with $\text{CLTE} > 110 \times 10^{-6} \text{ K}^{-1}$ (PTFE, PTFE+CF, PTFE+TiB₂, PTFE+K) exhibit significant leakage ($>0.8 \text{ ml/min}$), while materials with $\text{CLTE} < 100 \times 10^{-6} \text{ K}^{-1}$ (PTFE+CNT+K, PTFE+SiO₂) exhibit leakage $<0.3 \text{ ml/min}$. This confirms the threshold nature of the effect of thermal expansion on seal integrity.

4. Conclusions

This study evaluated the performance of PTFE-based composites under accelerated climatic cycling (-50°C to $+100^{\circ}\text{C}$, 10 cycles) for potential use in arctic sealing applications. The key findings are as follows:

1. Mechanical stability: While unfilled PTFE lost approximately 20% of its tensile strength after testing, the PTFE+CNT+K composite retained its mechanical properties most effectively, with no statistically significant change in tensile strength or modulus of elasticity.

2. Structural influence: Nanoscale fillers (CNT, K, SiO₂) promoted the formation of dense, spherulite-like supramolecular structures, which reduced the coefficient of linear thermal expansion (CLTE) by up to 45% compared to neat PTFE. This structural reorganization contributed to enhanced dimensional stability under thermal cycling.

3. Sealing performance at low temperature: In field tests at -40°C , the PTFE+CNT+K and PTFE+SiO₂ composites maintained effective sealing, whereas neat PTFE and PTFE+K exhibited significant leakage.

4. Optimal composite: The PTFE+CNT+K composite demonstrated the best overall combination of properties: retained mechanical strength, low CLTE ($96 \times 10^{-6} \text{ K}^{-1}$), and reliable sealing ability under extreme cold. This makes it a highly promising candidate for sealing elements in hydraulic systems operating in arctic environments.

The results emphasize the role of nanofillers in climatic resistance; however, full extrapolation to long-term service requires extended tests with a greater number of cycles.

Nomenclature

α_{XRD}	degree of crystallinity by XRD
α_{DSC}	degree of crystallinity by DSC
CLTE	Coefficient of Linear Thermal Expansion
V	volume

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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