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STUDY OF THE INTERACTION AT THE POLYCHLOROTRIFLUOROETHYLENE – CARBON FIBRE PHASE BOUNDARY

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Abstract

Aim. To investigate the chemical and physical interaction between the fluoropolymer matrix and the high-modulus Toray T700H carbon fibre to evaluate the functional characteristics of carbon plastics. Methods. We prepared carbon plastics based on polychlorotrifluoroethylene containing 5–20 wt.% Toray T700H carbon fibre by dry mixing them in an apparatus with a rotating electromagnetic field with the help of ferromagnetic particles. They were extracted later using the method of magnetic separation. We analysed the IR spectra using a Nicolet 380 (IR spectrometer from Thermo Electron Corporation, the USA). Results. We compared the spectra of powdered polychlorotrifluoroethylene and its finished product. Therefore, we determined changes in the chemical composition and molecular structure of the polymer during processing into products. The investigation shows that the carbon fibre of the studied brand exhibits low reactivity. We identified and analysed the results of IR-spectral studies of carbon plastics. Conclusions. We established the interaction between the fluoropolymer matrix and the carbon fibre. It manifests itself through the interaction between the double bonds of the terminal active groups and the oxygen-containing groups of polychlorotrifluoroethylenewith the Toray T700H carbon fibre. We can observe the strongest intercomponent interaction for the carbon fibre with a fibre content of 15–20 wt.%.

Keywords: polychlorotrifluoroethylene; carbon fibre; infrared spectral analysis; chemical bonds**.**

ДОСЛІДЖЕННЯ ВЗАЄМОДІЇ НА МЕЖІ ФАЗ ПОЛІТРИФТОРХЛОРЕТИЛЕН ТА ВУГЛЕЦЕВЕ ВОЛОКНО

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Анотація

У роботі наведені ІЧ-спектри вуглеплапластиків на основі політрифторхлоретилену армованого дискретним високомодульним вуглецевим волокном марки Toray T700 залежно від ступеню наповнення. Встановлена взаємодія між фторполімерною матрицею та вуглецевим волокном, що проявляється через взаємодію між подвійними зв’язками кінцевих активних груп та кисневмісними групами політрифторхлоретилену з вуглецевим волокном марки Toray T700H. Найбільш сильна міжкомпонентна взаємодія спостерігається для вуглепластику при вмісті волокна 15–20 мас.%.

Ключові слова: політрифторхлоретилен; вуглецеве волокно; інфрачервоний спектральний аналіз; хімічні зв’язки

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Introduction

Nowadays, materials are influenced by aggressive environments. They must withstand high force and temperature loads and work in friction modes without lubrication [1]. It is essential for the stable and efficient operation of friction assemblies of modern equipment. In addition, these materials must be manufactured using energy-efficient and ecological technologies. They must have high indicators of thermophysical and physical-mechanical characteristics. In this case, the use of pure metals and polymer materials is ineffective. The solution to this technical problem is polymer composite materials (PCMs). Using them increases the service characteristics of friction assemblies and ensures the equipment's durability and reliability [2–4].

These days, PCMs based on fluoropolymers are an adequate replacement for metals. Using these composites makes it possible to obtain tribotechnical parts with high thermal stability, excellent anti-friction properties, and inertness to many aggressive chemical environments and lubricants [5]. It makes them interesting to use as special-purpose materials. It is well-known [6] that the functional characteristics of products made of composite materials, including those based on fluoropolymers, directly depend on the intermolecular interaction between the binder and the filler. In this regard, the study of chemical and physical interaction between components using IR spectral analysis has become traditional and is a commonly used method for evaluating the properties of materials [7].

Considering the above, the work aimed to study the chemical and physical interaction between the fluoropolymer matrix and the high-modulus Toray T700H carbon fibre (CF).

Experimental

Research objects and methods

We chose polychlorotrifluoroethylene (PCTFE) [$-CF_2-CFCl-$] (Fig. 1) as a polymer matrix (manufactured by Daikin Industries, Ltd. (Japan), Neoflon® brand). PCTFE is a white solid, powdery material with high plasticity, compressive strength, and chemical resistance (close to polytetrafluoroethylene) [8]. In addition, products based on polychlorotrifluoroethylene can be used in a wide temperature range from 78 to 463 K, depending on the mode of formation of the products [9].

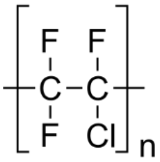


Fig. 1. Chemical formula of polychlorotrifluoroethylene

A discrete (the length was 3 mm) Toray T700H carbon fibre (manufactured by Toray Industries Inc., Japan) was chosen as a filler. We distinguished Toray T700H among others because of its increased indicators of physical and mechanical properties (modulus of elasticity and tensile strength are 230 and 4.9 GPa, respectively), which determines the use of fibres of this brand in various industries [10; 11].

Compositions based on PCTFE, containing 5–20 wt.% of Toray T700H CF, were prepared by dry mixing in an apparatus with a rotating electromagnetic field (0.12-0.15 T) with the help of ferromagnetic particles, which were subsequently extracted by magnetic separation.

We made test samples of compositions based on PCTFE in the shape of cylinders with a diameter and height of 10 mm. The finished mixture was tableted at room temperature and under a load of 40 MPa. The obtained prepregs were loaded into a mould heated to 423 K. After this, the mould temperature was raised to 510-515 K and kept at this temperature for 10 minutes. Then, a load of 40 MPa was applied. The product was cooled under pressure to a temperature of 490-495 K to fix the shape. Then, it was pushed out of the mould into water for cooling process [12].

We used a Nicolet 380 (an IR spectrometer of Thermo Electron Corporation, USA) for IR spectral analysis. We installed the spectrometer with a compressed powder containing 1 mg of the test substance and 150 mg of KBr (potassium bromide) in the form of a film [13].

Results and discussion

Research results. In the first stage of research, we compared the spectra of powdered PCTFE and the finished product made from it (a pressed sample) to determine possible changes in the chemical composition and molecular structure of the polymer with the selected processing mode, which was given in the previous section. Fig. 2 shows the IR spectra of the original (A) and pressed (B) PCTFE. Table 1 shows the distribution of absorption bands in the IR spectra of the initial PCTFE powder and the pressed sample (valence (n) and deformation (d)oscillations) [14–16].

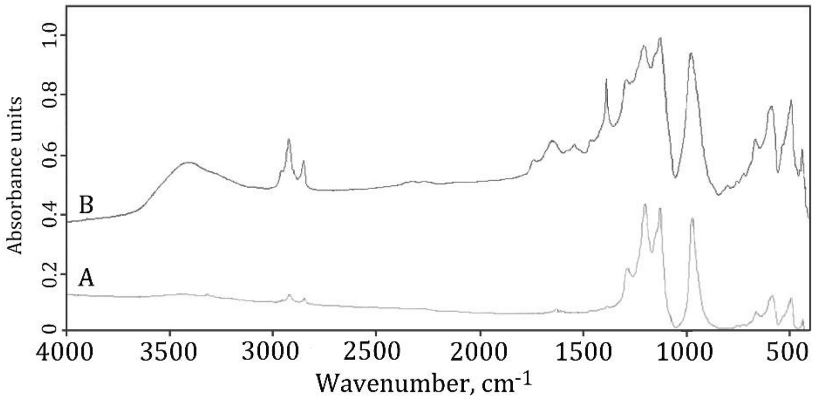


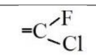
Fig. 2. IR spectra of the original (A) and pressed (B) PCTFE

We analysed and compared the obtained data with the known literature data [1]. Therefore, the original PCTFE contains bonds in the elementary chain. In addition, weak absorption bands in the region of 4000–3100 cm^{-1} are detected in the spectra. They are probably artefacts of the analysis method. They may also indicate the presence of impurities (for example, moisture or hydroxyl compounds) in the sample. Also, the appearance of two bands in the region of 2930–2850 cm^{-1} may be due to the interaction of the end groups of the polymer with residual impurities or products of thermo-oxidative destruction. They may have occurred during the synthesis, materials processsing or due to materials storage. There is a weak absorption in the 1652–1550 cm^{-1} region. It may be associated with traces of carbonyl compounds (for example, carboxylic acids or diketones) formed during polymer processes.

The presence of organofluorine groups in the structure is evidenced by absorption at 587 cm^{-1} (valence vibrations of perfluoro ketones) and a band at 937.5 cm^{-1}, characteristic of =C-F.

Table 1

The ratio of vibrational bands in the IR spectra of original (A) and pressed (B) polychlorotrifluoroethylene (Fig. 2)

Designation of the spectrum (Fig. 2)	Bands, cm^{-1}: ν – valence oscillations, δ – bond deformation							
	C – F	Poly-mers	C – Cl	F – C – F	= C – F	 $=C\genfrac{}{}{0pt}{}{F}{Cl}$	$\pi - \bar{e}$ cloud	Other bonds
	1100–1000 s.; 1300–1000 [7]	3400–3200 s.,w. [1]	$\nu_{(C-Cl)}$ = 750-695; 850-800 m. [7]	1350-1200 ver. s., as; 1200-1100 ver.s., sym; 530-510; 450-340	$\nu_{(C-F)}$ = 1250-1100 ver. s. [7]	520–420; 390–330 [7]	3600–3500 olefins [7]	1651,8; doublet 2921.3 and 2851.6
A	970 s., n.; 1280 m., n.; 1203 s., n.	4000–3200 wk.,w.	695 wk.n.	1210 s., n.; 1120 s., n.	doublet 1203 s., n. 1126 s., n.	500–490 m. n.	4000–3100 wk., w.	587; 973; doublet 2900-2850; 4000-3100 wk., w.
B	1288 m., n.; 1203.1 s.,n.; 1125.8 s.,n.; 973.5 s., n.	ν_{max} = 3414.6 wk.,w.; 3600–3160	665.7 m., n.	1384.5 wk., n.; 1288.6 m., n.; 1203.1 s., n.	doublet 1203.1 s., n. and 1125.8 s., n.	490.8 m., n.; 437.0 s., n.; 587.3 m., n.	3600–3160 wk., w.; ν_{max}= 3414.7	doublet 2921.3 wk., n. and 2851.6 ver.wk., n.

Notations: as (antisymmetric) and sym (symmetric) types of oscillations; strong (s.), medium (m.) and weak (wk.) intensity of the spectrum; wide (w.) and narrow (n.)

We can observe an increase in the intensity of the bands at 2921.3 and 2851.6 cm^{-1} in the pressed sample of PCTFE at a temperature of 515 K (Fig. 2, B). It may indicate an increase in terminal hydroxyl groups due to partial destruction of the polymer. The shift of the ν(C=O) absorption bands at 1600 cm^{-1} towards lower frequencies (1580 cm^{-1}) indicates a change in the electronic environment of these groups, in particular, due to the formation of structures characteristic of enol or carboxyl compounds. The presence of an arm of force at 1541 cm^{-1} may be due to the formation of carboxylates. After pressing, the samples show the disappearance of broad bands characteristic of possible water impurities and the appearance of bands corresponding to intermolecular bonds in the polymer (3414.6 cm^{-1}).

In addition, the intensity of vibrations characteristic of C–Cl and C–F bonds increases in the pressed sample: ν(C–Cl)=665.7 cm^{-1}, ν(=C–F)=1125.8 and 973.5 cm^{-1}, ν(CF$_2$)=1286.6 cm^{-1}. Absorption at 587.3 cm^{-1} indicates the presence of perfluoro ketones, and at 490.8 and 437.0 cm^{-1} – for CFCl.

Based on the analysis of the IR spectra of PCTFE powder and its product, we can conclude that specific changes in the chemical composition and molecular structure of the polymer occur with the selected processing mode. The changes are the partial destruction or formed during polymer processing of end groups of macromolecules, an increase in the number of hydroxyl groups, and the appearance of new bonds in the pressed sample. Additional studies, particularly with the help of mass spectrometry, are necessary to confirm these changes.

In the second stage of the research, we performed an IR spectral analysis of the Toray T700H carbon fibre. The IR spectrum of the CF is shown in Fig. 3, and the classification of its absorption bands is given in Table 2.

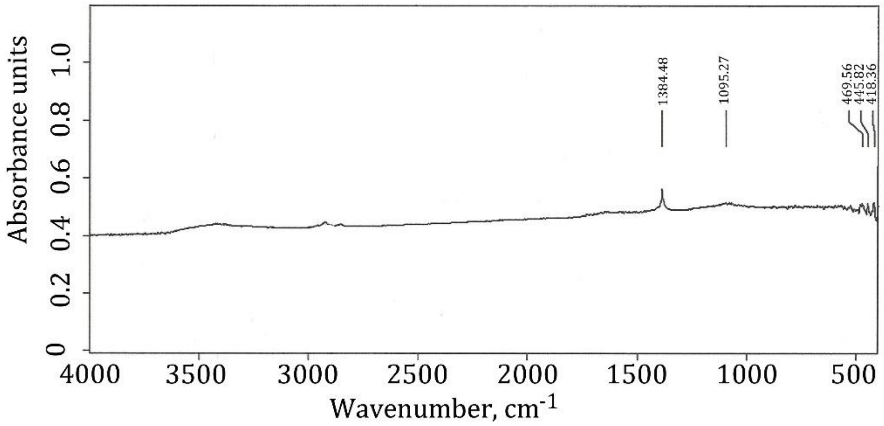
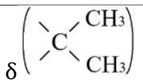


Fig.3. IR spectrum of Toray T700H CF

Table 2

Ratio of absorption bands in the IR spectrum of Toray T700H carbon fibre

Bands, cm^{-1}: valence oscillations		δ-deformations	Polymer H-bond
ν(CH=CH$_2$)	ν$_{as}$(CH$_3$→CH$_2$=) ν$_s$(CH$_3$→CH$_2$=)	 1385 and 1370 m.; 1145; 1170	3400-3200 s., w. polymers
412.58 [1]	ν(CH$_3$)2962 c., ν$_{as}$ [7] ν(CH$_3$)2872 s., ν$_s$ [7] or ν(CH$_2$)2925 s.; 2850 s., ν$_s$	δ($_{CH3}$)$_{as}$=1400 s.; δ($_{CH3}$)$_s$=1375 m.	π- electron cloud, 3600-3500 w., diffraction band
469.6 ver., n.; 445.8 ver., n.; 418.4 polyphenyls	2920 ver., n.; 2860 ver. wk.,n.	1384.5 m., n.; 1095.3 ver., wk., w.	4000-3200 ν$_{max}$=3400 wk., w.

The data analysis indicates a complex structure of the carbon fibre surface, which probably contains residual organic compounds or coatings. This is due to the technological processes of manufacturing and processing the material.

The absorption band at 1884 cm^{-1} may be due to functional groups that are part of the coating or treatment layer (for example, residual carbonyl groups from epoxy resins or other organic compounds). This peak is not characteristic of the carbon fibre itself. But it indicates residual substances or surface modifications.

The range 3000–2500 cm^{-1} indicates vibrations that probably belong to O–H or C–H bonds. This can be caused by:

- The presence of hydroxyl groups (O–H) due to adsorbed moisture or residues of organic substances.
- Valence fluctuations of C–H bonds in aliphatic or aromatic compounds. They could remain after the technological processing of the fiber.

The absorption band at 1384.5 cm^{-1} indicates the deformation vibrations of methyl (CH$_3$) or isopropyl groups. This band probably belongs to the remains of organic compounds or surface modifications applied to improve adhesion.

Hence, the spectral analysis indicates the possible residual chemical compounds on the surface of the carbon fibre, such as residues of epoxy resins, finishes (in particular, amide or ether coatings), or other organic substances. To determine their nature more accurately, additional research methods, such as X-ray photoelectron spectroscopy (XPS) or mass spectrometry, are necessary.

In the final stage of the research, we analysed the results of IR-spectral studies of carbon plastics based on PCTFE. This is shown in Fig. 4 and Table 3.

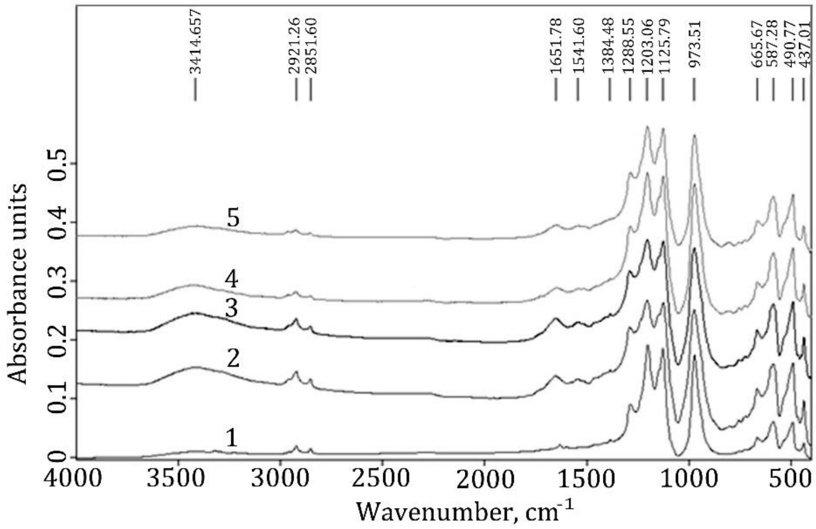


Fig. 4. IR spectra of polychlorotrifluoroethylene (1) and carbon plastics based on it, containing: 5(2); 10(3); 15(4); 20(5) wt.% fibre

The analysis of the obtained spectra indicates the interaction between the carbon fibre and the polymer matrix. This interaction consists in changing the physical and chemical properties of the composite surface layer.

We observe changes in the IR spectrum (particularly in the region of 1540–1660 cm^{-1}) with a carbon fibre content of 15–20 wt.%. This may be associated with the presence of carbonyl (C=O) or aromatic (C=C) groups. These changes may occur due to residual compounds on the surface of the fibres or the influence of pressing conditions.

Table 3

The ratio of vibrational bands in the IR spectra of carbon plastics (C-F) with a carbon fibre content of 5-20 wt.% (Fig. 4)

Designation of the spectrum (Fig. 4)	Content of CF, wt.%	Bands, cm^{-1}: ν – valence oscillations, δ – bond deformation							
		C – F	Poly-mers	C – Cl	F – C – F	= C – F	$=\mathrm{C}\begin{smallmatrix}/\mathrm{F}\\ \backslash\mathrm{Cl}\end{smallmatrix}$	$\pi - \bar{e}$ cloud	Other bonds
		1100-1000 s.; 1300-1000	3400-3200 s., w.	ν(C-Cl) =750-695; 850-800 m.	1350-1200 ver. s., as; 1200-1100 ver. s., sym; 530-510; 450-340	ν(C-F) 1250-1100 ver. s.	520-420; 390-330	3600-3500 olefins	1651.8; doublet 2921.3 and 2851.6
2	5	970 s.,n.; 1260 m.,n.; 1180 m.,n.	3400-3200 s., w.	695 wk.,n.	1213 s.,n.; 1100 ver. s., sym	doublet 1183 s.,n. and 1106 s.,n.	500-490 m.,n.	4000-3100 wk., w.	doublet 2921 wk., n. and 2851 ver.wk., n.
3	10	970 s.;n.; 1260 m.,n.; 1180 m.,n.	3400-3200 s., w.	695 wk.,n.	1213 s.,n.; 1100 ver. s., sym	doublet 1183 s.,n. and 1106 s.,n.	500-490 m.,n.	4000-3100 wk., w.	doublet 2921 wk., n. and 2851 ver.wk., n.
4	15	970 s.,n.; 1280 m.,n.; 1203 m.,n.	3400-3200 s., w.	695 wk.,n.	1203 s.,n.; 1120 ver. s., sym	doublet 1203 s.n. and 1125 s.n.	500-490 m.,n.	4000-3100 wk., w.	doublet 2921 wk., n. and 2851 ver.wk., n.
5	20	970 s.,n.; 1280 m.,n.; 1203 m.,n.	3400-3200 s., w.	695 wk.,n.	1203 s.,n.; 1120 ver. s., sym	doublet 1203 s.n. and 1125 s.n.	500-490 m.,n.	4000-3100 wk., w.	doublet 2921 wk., n. and 2851 ver.wk., n.

These samples also show increased intermolecular interactions, particularly π,π-interactions between the aromatic structures of the carbon fibre and the polymer matrix. These effects may result from compression moulding and tight contact between the filler and the polymer matrix.

Hence, composites with a carbon fibre content of 15–20 wt.% demonstrate increased physicochemical interaction between components, which can improve their technical characteristics. Additional studies of IR spectra and other physicochemical analysis methods should be conducted to assess these effects more accurately [16-25].

Conclusions

1. The study showed that compression moulding affects PCTFE. Specific changes occur in the chemical composition and molecular structure of the polymer. The changes include partial destruction or modification of the end groups of macromolecules, an increase in the number of hydroxyl groups, and the appearance of new bonds in the moulded sample.

2. Carbon fibre has a complex surface structure and may contain residual chemical compounds on its surface, such as residues of epoxy resins, coatings (particularly, amide or ether coatings), or other organic substances.

3. An active interaction between Toray T700H carbon fibre and PCTFE was found, which probably changes the physicochemical properties of the composite surface layer.

4. We established that the most intense changes are observed with a carbon fibre content of 15–20 wt.%, which indicates the maximum interaction between the fibre and the matrix.

5. The strengthening of the π,π-interaction between the filler and the polymer at a fibre concentration of 15–20 wt.% was noted, which will positively affect the physical and mechanical properties of carbon plastics.

6. The research showed that compression moulding promotes chemical changes in carbon plastics, such as the release of HCl and possibly the addition of water or carbon dioxide molecules, which can alter the properties of the composite.

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