

A method for modifying the surface of silicon carbide with a controlled number of functional groups on surface

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Abstract: The inertia of silicon carbide (SiC) when creating polymer composite materials (PCM) often does not allow creating a high-quality final product. This is due to the weak interfacial interaction between the filler and the polymer. This paper presents a method for modifying silicon carbide with a controlled content of 3-aminopropyltriethoxysilane (APTES) on its surface. The modifier will create active functional groups on the surface of silicon carbide, and they will be the first to interact. In the study, a step-by-step assessment of the change in the surface of filler particles during the formation of reactive groups by IR spectroscopy was carried out and the effect of the proposed method on the bulk density of SiC particles was determined. The presented work contains data on the study of the adsorption kinetics of the filler after its modification, and describes the conditions under which the most complete addition of functional groups occurs during treatment with silane. The factors that can be used to vary the content of functional groups on the surface of silicon carbide, thus changing its activity, have been identified. The effectiveness of the proposed modification method is shown and samples with a reactive surface of SiC particles are obtained, which are the most promising for the creation of polymer composite materials.

Keywords: silicon carbide; filler modification method; aminopropyltriethoxysilane; APTES; polymer composite material; chemisorptions; polyimide.

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Способ модификации поверхности карбида кремния с управляемым количеством функциональных групп на его поверхности

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Аннотация: Инертность карбида кремния (SiC) при создании полимерных композиционных материалов (ПКМ) часто не позволяет создать качественное конечное изделие. Это обусловлено слабым межфазным взаимодействием между наполнителем и полимером. В данной работе представлен способ модификации карбида кремния с управляемым содержанием 3-аминопропилтриэтоксисилана (АПТЭС) на его поверхности. Модификатор создает активные функциональные группы на поверхности карбида кремния, и именно они будут первыми вступать во взаимодействие. В исследовании проведена поэтапная оценка изменения поверхности частиц наполнителя при образовании реакционноспособных групп методом ИК-спектроскопии, и определено влияние предлагаемого способа на насыпную плотность частиц SiC. Представленная работа содержит данные об исследовании кинетики адсорбции наполнителя после его модификации и описывает условия, при которых

происходит наиболее полное присоединение функциональных групп при обработке силаном. Выявлены факторы, с помощью которых можно варьировать содержание функциональных групп на поверхности карбида кремния, изменяя таким образом ее активность. Показана эффективность предложенного метода модификации и получены образцы с реакционноспособной поверхностью частиц SiC, являющиеся наиболее перспективными для создания полимерных композиционных материалов.

Ключевые слова: карбид кремния; модификация поверхности; аминопропилтриэтоксисилан; АПТЭС; полимерный композиционный материал; хемосорбция; полиимид.

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1. Introduction

The development of modern technology requires new structural materials that are superior in strength, elasticity and properties. Most of the polymer composites are developed for the aerospace industry. At the same time, these materials and technologies for their production are also promising in many other industries, such as construction, shipbuilding, mechanical engineering, energy, instrumentation, medicine, railway engineering and other industries.

The development of polymer composite materials (PCM) technology currently depends on research into interaction of polymer matrices and fillers. The introduction of the latter (mainly inorganic) into the polymer matrix makes it possible for composites to demonstrate new, often unique properties, such as their hardness, high thermal stability, and mechanical properties. In addition, varying the content of the filler in the polymer matrix allows you to change both the processing parameters and the performance characteristics of the final material. The properties of composite materials are largely determined by the parameters of interfacial interaction. The formation of this layer makes it possible to obtain composites with target characteristics: increased rigidity, wear resistance, resistance to thermal oxidation, and a controlled level of thermal conductivity. Variation of the latter is required when creating a design with a heat sink in electronic technology for a satellite system, as well as in designs with shape memory, in which the effect that occurs is initiated by heating.

However, polymer-based composites are limited by their low thermal conductivity, which for most polymers lies in the range of $0.05\text{--}0.35\text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$. The increase in the thermal conductivity of polymers increases due to the creation of heat-conducting structures based on fillers with high thermal conductivity, such as carbon nanotubes, graphene, graphite, boron nitride, aluminum nitride, aluminum oxide, silicon carbide, and others [1]. The addition of such fillers leads to the formation of continuous heat-

conducting chains in the polymer product. It is noted in [2] that at low filler concentrations, the increase in thermal conductivity has an almost linear dependence on the composition.

Silicon carbide (SiC) is used to develop polymer composite materials operating at high temperatures, power, and high radiation conditions. It has semiconductor properties, chemical resistance and chemical inertness; therefore it has a wide range of applications: in mechanical engineering, metallurgy, nuclear, aerospace and chemical industries. However, the use of SiC as a modifying additive in polymer compositions is difficult due to insufficient chemical activity of its surface. This further leads to poor interaction of chemically inert SiC with the polymer and deterioration of the properties of the polymer composition, both at the manufacturing stage and during its operation.

When filler is added to the polymer, the first interactions occur depending on the functionality of the surface of the filler particle, i.e. on the type of molecules on the surface of the particle. The viscosity of the polymer composite material is directly related to the interactions of the filler particles with each other, as well as the particles and the polymer matrix. Taking into account the thesis "structure determines properties" accepted in materials science, a significant contribution to the properties of a composite material is made by the type, surface and composition of particles. In addition, the modification of the filler makes it possible to improve the wettability between the polymer and the filler and influence its distribution in the polymer. It is known that a good contact of the filler with the polymer always improves the properties of the resulting product [3–5]. The addition of even a small amount of SiC to polyimide compositions significantly increases its thermal stability, so when 0.5–2.0 % SiC is introduced into the polymer, the onset point of decomposition in TGA analysis shifts from 200 to 540 °C [5].

Previously, we used the plasma-chemical method of modifying SiC [6], which made it possible

to obtain a carbon shell decorated with silicon nanoparticles and nanowhiskers. At present, in order to increase the activity of the SiC surface, it is also modified by other methods: thermal oxidation [7–15] or chemical oxidation, grafting of monomers, oligomers, and polymers [16–18]. In [19–21], bifunctional compounds were used to improve the bond with the polymer, whose surface was modified with silanes. Silanization is required for the formation of a self-assembled monolayer (SAM), which was first described by Jacob Sagiv [22]. Since one of the requirements for successful silanization is activation of the substrate by producing a hydroxyl surface, silanization reactions have traditionally been applied to materials such as glass or metal oxides that can be easily hydroxylated.

The following temperature zones of heat treatment are distinguished:

- up to 1000 °C, passive oxidation occurs with carbon burnout from the SiC surface and coating with a quartz film;

- above 1000 °C, re-deposition of the quartz film occurs on the SiC surface caused by the formation of silicon monoxide at high temperature (due to this, both open and closed porosity increases).

Modifications include surface oxidation with nitrogen oxides [22] or Piranha solution [24–28].

Having treated the surface of inorganic particles, silane remains on the surface both chemisorbed and physisorbed. The chemisorbed silane is chemically bonded to the inorganic surface, while the physisorbed silane is bonded to the surface by van der Waals forces. In composites, both types of force exhibit different beneficial effects [29].

Physisorbed silanes that have diffused into the polymer matrix reduce water absorption and improve the mechanical strength of the composite, while chemisorbed silanes improve interfacial adhesion. These effects are better manifested when using compositions having related groups with a “tail” part of the silane as a polymer matrix. When modifying fillers with silanes with a “tail” amino group, it is efficient to use polyimides capable of entering into a chemical reaction with terminal anhydride groups. To obtain PCM based on polyimides, it is possible to use polyamic acids in a solvent with a low viscosity, which makes it easy to introduce fillers with subsequent polymerization of polyamic acids into polyimide. PCMs based on polyimides are thermally stable, while the application of heat-conducting filler improves their thermal stability [29].

Thus, despite the interest of researchers in the field of treatment of SiC with silanizing agents to

create reactive groups on the surface, there is little information in the literature about methods for modifying SiC. There is also no information on the amount of silane present on the surface of the filler. Therefore, in the article, a modification technology with a controlled content of silanes on the SiC surface was developed.

2. Materials and Methods

2.1. Materials

The object of the study was SiC (fraction F-1200) with a grain size of 5 µm. As a modifying agent (silane) was used 3-aminopropyltriethoxysilane (APTES). For the oxidation of SiC after calcination, H₂SO₄ (chemically pure) and H₂O (36 %) were used. Polyamic acid (PAA) (polyimide lacquer AD-9103 IS) was used as a polymer in the creation of samples of polymer composite materials, designed to obtain film materials.

2.2. Modification of SiC surface using APTES

The modification of SiC is reduced to the formation of organic molecules on its surface. Such molecules have specific head and end groups for specific chemical bonding to the polymer. In the presented paper, the SiC modification technology was carried out in 3 steps (Fig. 1).

Stage 1: *SiC surface oxidation*. The sample was calcined at 1100 °C for 3 hours – under these conditions, a SiO₂ surface layer is formed. The presence of this surface layer is necessary for further modification; it was also noted in [18, 30] that such treatment improves adhesion. In addition, a range of chemical treatments are available for the controlled modification of oxide surfaces to provide specific functionality.

Stage 2: *modification of the SiO₂ layer* was carried out by chemical etching with a Piranha solution (H₂SO₄/H₂O₂ in a ratio of 1 : 5), resulting in saturation with —OH groups.

Stage 3: *silanization* was carried out with APTES, due to which the polymer/filler interfacial interaction was formed. Successful silanization proceeds most efficiently in polar solvents; therefore, we used dimethylformamide (DMF) to separate the —OC₂H₅ group from APTES. The treatment of SiC with silane itself was carried out in an ultrasonic bath at 60 °C.

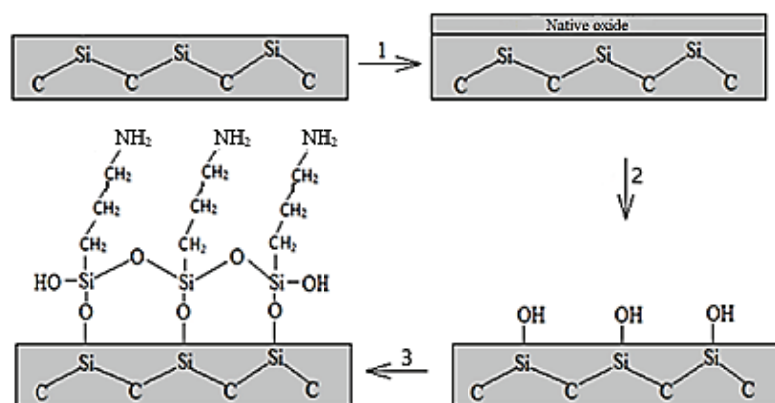


Fig. 1. SiC modification stages

2.3. Research into SiC properties at different stages of modification by an IR-spectroscopy method

SiC samples at different stages of modification were studied by FT-IR using a Nicolet iS10 spectrometer manufactured by ThermoScientific (USA).

The measurements were carried out with a spectral resolution of 4 cm^{-1} , averaging over 32 scans, in the range $4000\text{--}400\text{ cm}^{-1}$. The obtained IR-Fourier spectra were processed using the OMNIC software. Sample preparation of the studied samples included pressing tablets with potassium bromide (KBr). SiC particles were ground into powder with a mortar together with KBr (preliminarily dried in an oven at $200\text{ }^{\circ}\text{C}$ for 2 h). The optimal concentration of the test substance in KBr is 0.5 %, since this ratio gave the best structure of the spectra. The resulting powder was formed into a tablet using a PIKE Technologies hand press. The value of 12 was set on the force regulator scale of the pressure plate of the hand press, and the tablet was pressed for 15 s, the thickness of the tablets of the studied samples was in the range of $510\text{--}530\text{ }\mu\text{m}$, the diameter was 7 mm.

2.4. SiC surface adsorption kinetics

The adsorption kinetics was studied using the gravimetric method. SiCk samples (after treatment with Piranha solution) were placed in a test tube and weighed. Next, 1 mL of APTES and DMF were added to the samples, and the resulting suspension was treated in an ultrasonic bath at the following time intervals: 1, 2, 5, 10, 30, 60, 120, and 240 min. Then, the filler was washed three times with distilled water to remove DMF and calcined in an oven at $155\text{ }^{\circ}\text{C}$ to

remove residual washing water. Next, the SiC sample was weighed and the added $\text{—O—Si—C}_3\text{H}_6\text{—NH}_2$ groups were quantified by increasing the weight of the filler.

2.5. Method for determining bulk density

Bulk density of samples of the original and modified SiC was found according to Russian Standard 19440–94 formula [31]:

$$r_b = \frac{m_2 - m_1}{V_b}, \quad (1)$$

where m_1 is weight of an empty measuring cylinder, kg; m_2 is weight of measuring cylinder with material, kg; V_b is measuring cylinder volume, m^3 .

2.6. Method of investigation of rheological properties of polymers filled with SiC

The efficiency of SiC surface modification upon its introduction into the polymer was evaluated by the rheological method. The flow curves obtained at a temperature of $25\text{ }^{\circ}\text{C}$ and in the range of shear rate (γ) equal to 0 to 800 s^{-1} of composite samples based on PAA and SiC particles were investigated with a Discovery HR-2 rotational rheometer manufactured by TA Instruments (USA), using the “plane-to-plane” geometry (diameter $d = 25\text{ mm}$, gap between planes $h = 1000\text{ }\mu\text{m}$). The comparison of the viscosity of the samples was carried out upon reaching some ordering of both the molecular chains of the polymer and the mutual arrangement of SiC particles [32], which is observed for all samples at $\gamma = 100\text{ s}^{-1}$.

3. Results and Discussion

3.1. Change in the reactivity of the SiC surface upon modification by FT-IR spectroscopy

The SiC reactivity was determined from the functional groups that are formed on the surface of SiC during its modification using FT-IR spectroscopy. Four samples were obtained at different stages of SiC modification: initial (SiC); after calcination (SiCt); after calcination and treatment with a Piranha solution (SiCk); after calcination and treatment with Piranha solution and APTES (SiCa).

The analysis of the functional groups formed on the surface of SiC samples at all stages of its modification is shown in Fig. 2.

Identification of the samples showed that the original SiC had Si—C peaks, while other peaks were not observed. The —OH and Si—O peaks appeared in the SiCt sample. In the graph of Fig. 2, the Si—O peaks are presented in the absorption bands 1080 and 460 cm^{-1} , and the —OH peaks are in the absorption bands from 3414 to 2849 cm^{-1} . This indicates the presence of a SiO_2 surface layer on the studied filler. After treatment with the Piranha solution, the intensity of the —OH peaks in the SiCk sample increased, which was evidenced by its saturation with the —OH groups of the SiC surface. After the 3rd stage of modification, the SiCa sample developed intermolecular bonds —N—H (3438, 3049 and 802 cm^{-1}), —C—N (1521 cm^{-1}) and —C—H (1419 cm^{-1}). This indicates the appearance of APTES on the SiC surface.

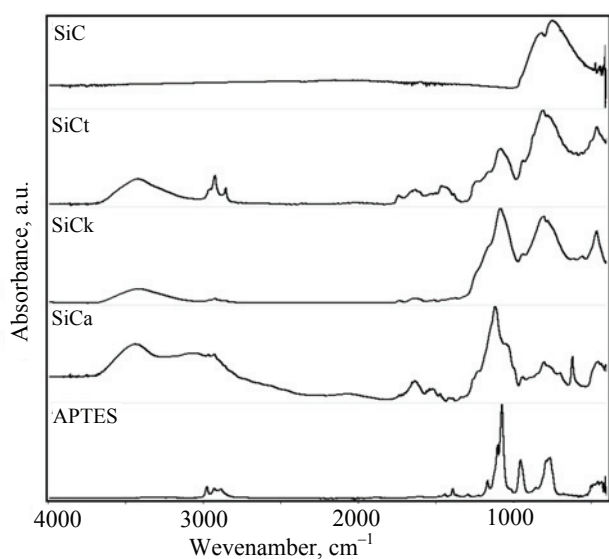


Fig. 2. IR spectra of SiC, SiCt, SiCk, SiCa, APTES samples

3.2. Study of chemisorptions at different stages of SiC modification

A study of the adsorption kinetics has shown that the APTES content on the SiC surface increases with staged modification. However, the content of APTES can be varied at different stages of modification. We have studied the influence of factors on the APTES content on the SiC surface at various stages of modification:

- at stage 2, we varied the time of treatment with the Piranha solution;
- at stage 3, we varied APTES processing time;
- the temperature of the first stage was constant.

The SiCt sample at the 2nd stage was treated with the Piranha solution for 1, 2, and 3 hours. It is shown that the time of exposure to the Piranha solution has a positive effect on the amount of APTES attached on the SiCk surface: an increase of 56 % was found at 3 h compared to 1 h (Fig. 3).

The APTES content on the SiCk surface was varied (stage 3) in the following time intervals: 1, 2, 5, 10, 30, 60, 120, and 240 minutes. The graphs in Fig. 3 show the kinetics of APTES adsorption on the activated SiCk surface.

A study of the kinetics of adsorption on the activated SiC surface showed that the maximum APTES content on the SiC surface is reached at 60 minutes. The duration of treatment with the Piranha solution saturates the surface with SiCt—OH groups and increases the APTES content on the SiCt surface from 2.1 % (1 h) to 3.7 % (3 h).

3.3. Change in bulk density under SiC modification

As a result of the modification, a decrease in the bulk density of SiC by 25 % is observed compared to the original sample (Fig. 4). Most likely, this is due to the presence of functional groups on the surface of

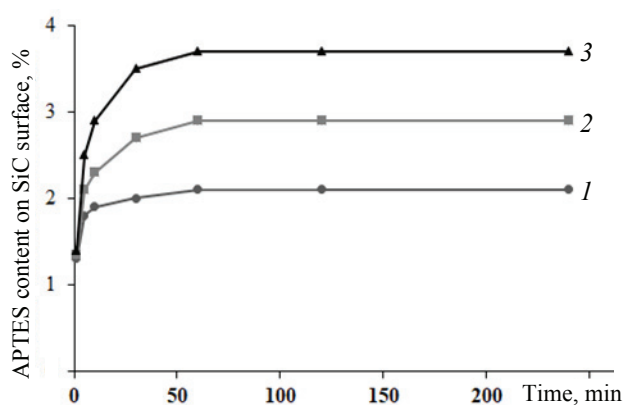


Fig. 3. Graph of APTES content on the SiCk surface versus exposure time: 1, 2, 3 – respectively 1, 2, 3 hour

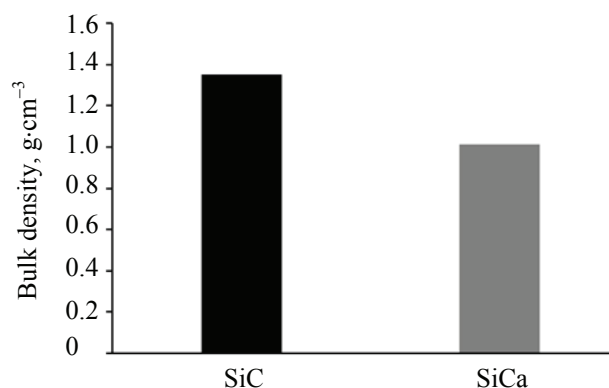


Fig. 4. Change in SiC bulk density after modification

SiC particles, which increase the specific surface area. The change in bulk density indicates that the distance between the SiC surface particles increased after the modification.

3.4. Assessment of modification efficiency of when forming interphase interaction

To form bonds with the polymer, a silane is used, which creates active functional groups on the SiC surface, and they are the first to interact. The viscosity of a polymer composite material depends on the nature of the interaction between the filler and the polymer and makes it possible to estimate the strength of the interfacial interaction. In this paper, the effectiveness of the proposed method for modifying the filler was evaluated by introducing it into the polymer and creating a polyimide-based PCM. The concentration of SiC after various stages of processing in PCM samples was 5 %. At the same time, according to the developed method, the amount of APTES on the SiC surface varied depending on the parameters: the time of treatment with the Piranha solution and the time of treatment of APTES. The viscosity of PAA filled with SiCk during treatment with Piranha solution for 3 hours is higher compared to 1 hour and 2 hours (Fig. 5), which can be explained by the formation of a larger number of filler bonds with the polymer.

With the phased modification of SiC, an increase in the viscosity of the polymer composite was observed, which was probably due to an increase in the number of functional groups on the surface of the filler particles, which can participate in the interaction with the polymer matrix (Fig. 6).

The effect of the duration of treatment with the Piranha solution on the level of APTES adsorption was estimated from the change in viscosity, which is

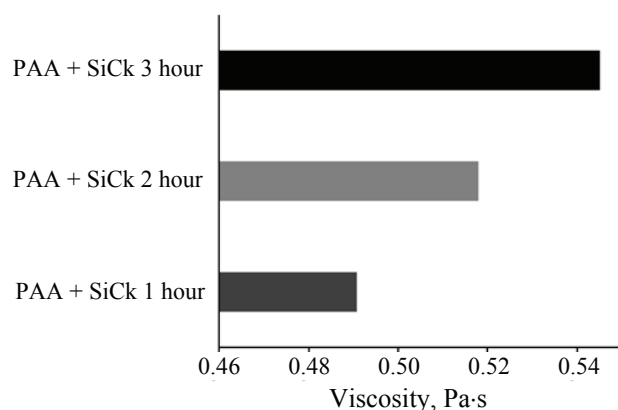


Fig. 5. Viscosity of PAA filled with SiCk depending on the treatment time with Piranha solution

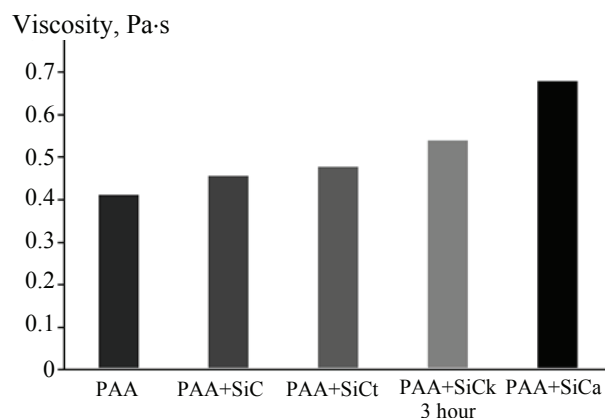


Fig. 6. PAA viscosity filled with SiC depending on its modification stages

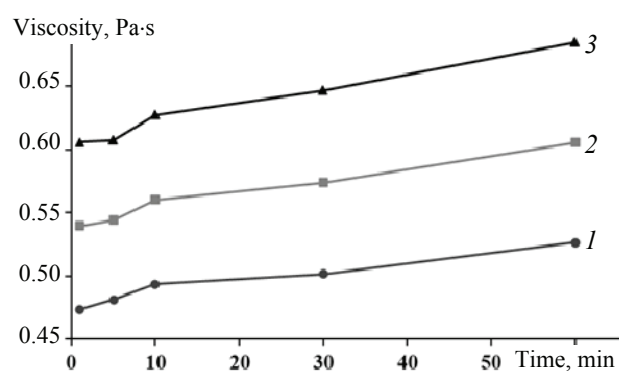


Fig. 7. Viscosity versus APTES content on the SiCk surface:
1, 2, 3 – respectively 1, 2, 3 hour

shown in Fig. 7. The duration of treatment with the Piranha solution led to an increase in the APTES content, which demonstrates an increase in the interfacial interaction between PAA and APTES on the SiCk surface.

4. Conclusion

A modification technology for the SiC surface to increase its reactivity has been proposed. The process parameters are determined by which it is possible to vary the content of APTES on the surface of SiC, thus changing its activity: the duration of treatment with acid and APTES. The content of APTES on the SiC surface at different stages of modification was established, and its highest value was recorded for the sample after the last stage of SiC modification (3.7 %). Studies have shown that the SiC sample demonstrates the highest reactivity after the third stage of surface modification (SiCa), since APTES attached to the SiC surface, as evidenced by the analysis of the IR spectra. Thus, the SiCa sample is the most promising for the creation of polymer composite materials.

The pattern of surface modification depending on the duration of treatment with the Piranha solution has been established. It saturates the surface with SiCt—OH groups and increases the APTES content on the surface from 2.1 % (at 1 h) to 3.7 % (3 h). As a result of the modification, a decrease in the bulk density of SiC by 25 % is observed compared to the original sample. The change was due to the fact that after modification, APTES was added to the surface and a more branched surface was formed due to functional groups. Regularity in the change in the polymer composite viscosity depending on the SiC modification stages has been revealed. An increase in the viscosity of the polymer composite was found depending on the time of treatment with APTES, which can be explained by an increase in the interfacial interaction between the polymer and APTES on the SiCk surface.

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7. Conflict of interests

The authors declare no conflicts of interest.

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