



Research article

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Heat-conducting and dielectric characteristics of polyorganosiloxane composites

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Abstract. The article considers the actual problem of improving the physico-mechanical, thermophysical and electrical properties of polyorganosiloxane coatings. In this article, we propose a method for obtaining a multifunctional heat-resistant composition based on oligodimethylsiloxane with terminal hydroxyl groups filled with boron nitride. The curing process of oligodimethylsiloxane and the possible interaction of boron nitride with reactive resin groups are described. The structure of the manufactured composition is investigated, it is established that the filler in the form of dispersed particles touching throughout the volume is evenly distributed in the polymer matrix. The results of experimental studies of the dependence of the tensile strength on the percentage of boron nitride, indicating the hardening of the composite, are graphically presented. Studies of the strength at separation of the cured composition from the substrate (adhesion) of various materials have shown that this value increases with the introduction of boron nitride. The dependence of the thermal conductivity coefficient on the ratio of components is established. The percolation point is determined by the Monte Carlo method. The dependences of the electrical resistivity and temperature dependence on the content of boron nitride are determined. The Poisson equation is solved in MathCad and graphical results of solving the heat transfer problem for traditional and developed composites are presented. Based on the results obtained, the areas of application of the developed composites in construction are proposed.

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1. Introduction

Currently, the compositions based on thermoplastic polymers and thermosetting polymer resins are widely used in construction for the supply and distribution cables at traditional and nuclear thermal power facilities and other construction areas. These are numerous polymer concretes, protective materials and coatings, adhesives and sealants [1–4]. Although in general, polymer compositions reveal common disadvantages such as insufficient heat resistance and a tendency to destruction under the impact of various external factors [5–6]. This facilitates the recent interest in new organoelement polymers, including organosilicon (polyorganosiloxane).

Polyorganosiloxane materials are well-known to be characterized by proper resistance towards adverse factors: UV radiation, high temperature, chemicals and microbiological impact [7–8]. However, unfilled polysiloxane resins are characterized by very mediocre physical and mechanical properties due to

the insufficient intensity of the implemented intermolecular interactions. Moreover, thermal protection causes problems with the coating stability at local overheating due to poor thermal conductivity of organosilicon resin [9–10]. To improve the heat-conducting and physico-mechanical properties, transition metal oxides (iron, zinc, titanium, etc.) are often introduced into polyorganosiloxane coatings as fillers. Nowadays the mechanisms of modifying polyorganosiloxane polymers by transition metal oxides are properly studied. A number of detailed studies resulted in the creation of Vixint protective sealing compositions [11].

The developed polymer composites are observed to find wide application not only in construction, but in other branches of science and technology, including high technologies sphere as well [12]. However, the heat-conducting properties do not correspond to the values required for the effective removal of the released heat during the heat-generating conductors operation in extreme operating mode. Therefore, polyorganosiloxanes are relatively often filled with metal powders [13]. The most common filler is aluminum powder. In this case, the composite acquires improved thermal stability not only due to the elimination of local overheating, but also because of chemical interaction of aluminum with reactive hydroxyl groups of polyorganosiloxane resin causes the formation of more stable polyaluminosiloxanes [14]. However, irreversible deterioration of dielectric characteristics makes such compositions unsuitable for the manufacturing of electrical structural elements for buildings and structures, for example, thermal power plants located near electric generators and high-voltage transformers, as well as other similar equipment. Similar restrictions concern the protective panels of high-voltage equipment. It is noteworthy that transition metal oxides additionally reduce the dielectric characteristics, in particular the composition electrical resistance [15].

The creation of new types of composites with increased heat-conducting and dielectric characteristics is an urgent task, the solution of which will increase the cooling efficiency of heat-generating conductors in extreme operating conditions. It is possible to increase the electrical resistance of compositions by using heat-conducting materials possessing high dielectric characteristics as fillers. Boron nitride represents an example of such interesting materials. This substance belongs to semiconductors exceeding the traditional aluminum nitride and silicon carbide in the energy-gap width and the breakdown electric field magnitude [16].

The purpose of this research is to demonstrate the results of a study of the physico-mechanical, thermophysical and electrical properties of a polymer composite based on oligodimethylsiloxane with reactive hydroxyl groups filled with boron nitride, which will allow us to establish the possibilities of its use in the construction industry when operating under conditions of elevated temperatures and electric fields.

2. Methods

2.1. Research objects

The following materials were used in the research: oligodimethylsiloxane (ODMS) with terminal hydroxyl groups, manufactured under the trade name of dimethylsiloxane rubber SKTN-1 GOST 13835-73 as a binder, catalyst K-18 (mixture of tin diethyldicaprilate and tetraethoxysilane) produced by Silan LLC, Moscow, and boron (III) nitride of “ultrapure” brand by Shandong Pengcheng Advanced Ceramics Co., Ltd China.

2.2. Production of samples

The experimentally specified amount of ODMS and boron nitride was added into the porcelain mortar and the mixture was thoroughly ground during four hours. Catalyst K-18 was added into the resulting mixture at the rate of catalyst 4 wt. p. per 100 wt. p. of oligomer and after five minutes of intensive mixing, the composition was poured into the molds for curing the samples during 72 hours at 25 °C.

2.3. Experimental methods

The tensile strength study was carried out in compliance with the standard tensile testing method according to Russian State Standard GOST 15873-70.

The composite adhesion to the surface was assessed using digital adhesive meter PSO-MG4 by tearing off a 20 mm diameter steel cylinder glued to the composite deposited on a metal substrate with high-strength glue. The adhesive meter PSO-MG4 is equipped with a spring device applying tensile force to the cylinder. When detached from the surface, the indicator shows the numerical value of adhesion on the scale, expressed in the force required to detach the cylinder.

The composite samples hardness by Shore A was determined using a digital hardness tester in compliance with Russian State Standard GOST 263-75 “Rubber. Method for the determination of Shore A hardness” (ASTM D2240).

The thermal conductivity coefficient was measured using the MGT-4 device. The research objects were samples 100×100×10 mm in size. A detailed description of the device operation is given in the operating manual [17].

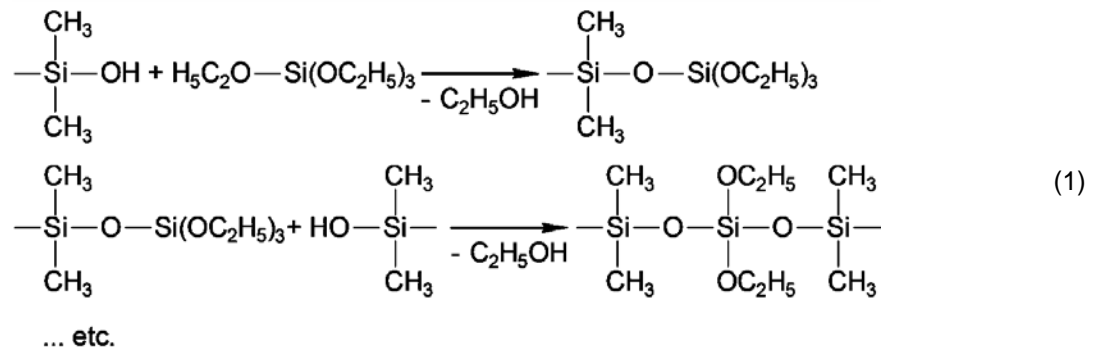
To measure the composition electrical conductivity, we used the device consisting of a measuring cell comprising two stainless steel electrodes (one movable and one stationary) and a clamping device. Prior to measuring the sample, the ends in contact with the electrodes were treated with electrically conductive paste. The sample size selection, subsequent measurements and results processing were carried out in compliance with Russian State Standard GOST6433.2-71 (ASTM D257) "Solid electrical insulating materials. Methods for evaluation of electrical resistance at d. c. voltages". The high-precision teraohmmeter UNI-T UT513 running Windows 11 OS was used as a data logger for measuring the dielectrics electrical conductivity.

Mathematical modeling and mathematical processing of the experimental results were carried out using Mathcad and OriginLab mathematical packages.

3. Results and Discussion

3.1. ODMS curing and possible filler interaction with reactive resin groups

The ODMS curing process is caused by the interaction of terminal functional hydroxyl groups with tetraethoxysilane being a part of catalyst K-18. Thus, the hydroxyl group interaction with the neighboring macromolecules occurs causing the formation of the cross-linked three-dimensional structure, accompanied by the ethanol molecule release:



The reaction is proceeding at room temperature during 72 hours [18].

The previously published papers [19] proved that boron introduction causes a drastic change of the composition rheological properties and its strong structuring, thus making it unacceptable to be used as a filler in potting technologies. Under the similar conditions boron nitride does not affect the composition rheology. Although in general, high probability of boron nitride interaction with the ODMS hydroxyl groups causing the formation of chemical bonds between the binder and the filler but only at high temperatures is quite obvious.

3.2. Study of physical and mechanical properties

Most polymer compositions possess the following structure: filler as dispersed particles evenly distributed in the polymer matrix touching throughout the volume.

The experiments with boron nitride used in the research were conducted using microanalyzer Horiba LB-550. They showed that the basic part of its particles is concentrated in a narrow area from 3 to 5 microns (Fig. 1). The introduction of the filler into ODMS and its distribution in the binder proceeds relatively easily without agglomeration.

Fig. 2 displays the composition structure. The geometry of boron nitride particles significantly differs from the spherical shape and largely resembles the structure of graphite. The specific distribution of particles is observed, externally resembling the conducting clusters. The percolation point can be assumed to be slightly lower than the theoretical one [20].

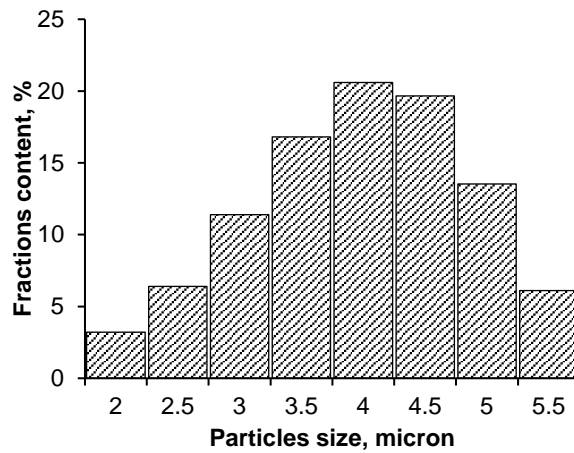


Figure 1. Fractional composition of boron nitride.

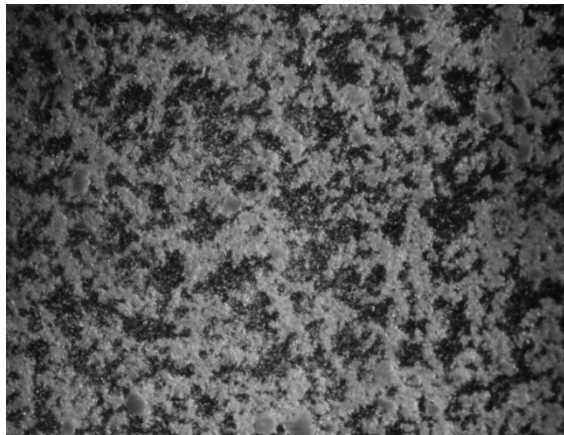


Figure 2. Boron nitride particles distribution in the composite (magnification $\times 100$).

Strength characteristics are extremely important for most composites. Based on theoretical concepts and numerous experiments dealing with the strength characteristics of organosilicon resins filled with polar fillers, we can talk about the regularity of increasing strength in the samples received in this work (see Table 1). When the filler is introduced into elastomer, the strength increase of the material is often observed. The studied systems demonstrated similar behavior (Fig. 3). The hardening can be explained using the model of elastomer molecules sliding along the filler surface (Dannenberg model [21]): if there is no filler, the short chains break first, and the filler increases the number of loaded chains, thus causing the load redistribution. If over 5% (vol.) of boron nitride is added, the composite strength increase is observed. When the filler content in the composite exceeds 20%, the increase in strength characteristics is slowing down.

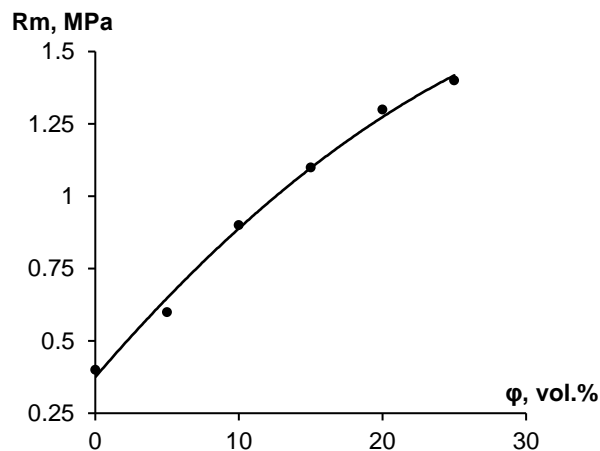


Figure 3 Tensile strength dependence (R_m) on boron nitride amount (ϕ).

The amplification effect might depend on the strong physical bonds formed between the binder and the filler, for instance, non-reacted hydroxyl groups and polar groups on boron nitride surface. Based on

theoretical concepts, the composite strength can be expected to decrease when the filler amount exceeds 25 % (vol.), associated with the lack of binder and incomplete encapsulation of boron nitride particles.

Adhesive properties are quite essential when the composite is supposed to be used as a sealing material. In general, non-modified polyorganosiloxanes are characterized by relatively low adhesion. Studies of tensile strength of the cured composition and the base from various materials demonstrate that boron nitride introduction increases this value (see Table 1). It can be explained by the absorption theory of adhesion, relating to the appearance of submicron boron nitride phase in the composition alongside the increase of intermolecular interaction between the substrate and the base. In practice, the strength increase at tearing polysiloxanes from bases made of various materials is observed at the introduction of a number of oxides, such as iron, zinc and titanium into them [22].

Undoubtedly, the adhesive characteristics of the composition largely depend on the substrate material. Thus, the strength when the discs are detached from the Steel 3 substrate is slightly higher than from the aluminum substrate. The introduction of boron nitride also contributes to the relative hardness increase of the sealing material (see Table 1), which is associated with the polymer structuring. It should be noted that the introduction of up to 25 % (vol.) boron nitride due to the structuring causes almost a double increase in Shore A hardness compared to the pure polymer.

Table 1. Composition properties

Filler content, %	Density, kg/m ³	Tensile strength (aluminum substrate), MPa	Tensile strength (steel substrate), MPa	Shore A hardness
-	980	0.08	0.11	24
10	1020	0.12	0.15	35
20	1160	0.21	0.24	44
25	1208	0.44	0.53	51

3.3. Thermophysical characteristics of the composite

As previously mentioned, studies have revealed that distribution of the filler particles in the composite is statistical. Judging by the percolation theory, the transition depends on the filler volume fraction [23]:

$$\sigma_{DC} \propto (v_f - v_{fc})^t, \quad (2)$$

where v_f is volume fraction of conductive component; v_{fc} is percolation threshold.

Monte-Carlo simulation for the composition containing spherical particles equals the value $v_{fc} = 0.16$ [24].

In addition to the particles shape, the thermo-physical characteristics of the composition largely depend on both the nature of binder and filler, and the components ratio. The thermal conductivity of composite materials consisting of several components can be determined by the following relation [29]:

$$\lambda = \lambda_{PDMS}(1 - \varphi) + \lambda_{SP}\varphi \quad (3)$$

where λ is thermal conductivity of the multicomponent material; λ_{PDMS} , λ_{SP} are the thermal conductivity of the first and second components; φ is the volume fraction of the filler.

However, the discrepancies are frequently observed between the design and experimental data, associated with certain unevenness in the filler particles arrangement in the composition, moisture absorption, insignificant amount of remaining gas phase after composition preparing due to abnormally high binder viscosity etc. Therefore, in practice, thermal conductivity coefficient, obtained by experimental methods is used.

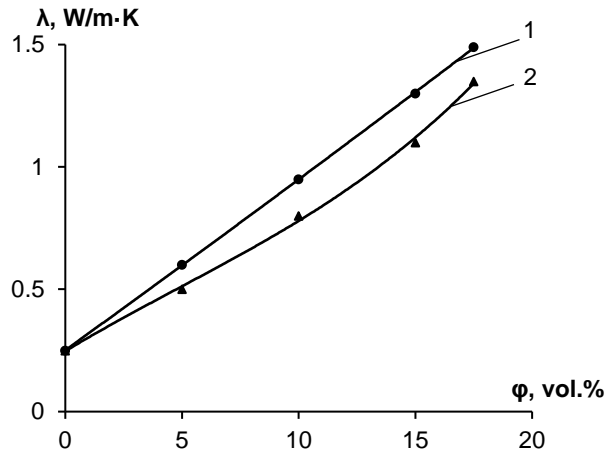


Figure 4. The dependence of thermal conductivity coefficient (λ) on filler content: 1– design; 2 – experiment.

Fig. 4 demonstrates the design and experiment values of the of thermal conductivity coefficient of SP with PDMS binder at 25 °C. In the studied max 20 % filling interval, thermal conductivity is described as follows: ascending linear dependence for design (see Fig. 4, curve 1), close to linear ascending dependence for experiment (see Fig. 4, curve 2). Thermal conductivity coefficient rises linearly as boron nitride content increases. However, the filler concentration increase up to the percolation point can be assumed to cause an abrupt increase of heat-conducting properties.

It should be noted that higher thermal conductivity coefficient of the unfilled organosilicon oligomer, compared to similar organic materials, is explained by high flexibility of polyorganosiloxane macromolecules associated with low intermolecular interaction of silicone chains.

To evaluate the effectiveness of the developed composites in MathCad environment, model calculations were performed showing the temperature distribution on the plane applying the Poisson equation. The two-dimensional Poisson equation is an example of elliptic partial differential equation including the second derivatives of the function $T(x, y)$ by two spatial variables [25]:

$$\frac{\partial^2 T(x, y)}{\partial x^2} + \frac{\partial^2 T(x, y)}{\partial y^2} = -f(x, y). \quad (4)$$

The Poisson equation frequently describes stationary distribution of temperature $T(x, y)$ on the plane with the heat sources (or absorbers) with $f(x, y)$ intensity. Poisson equation will be further considered in this very physical interpretation. Therefore, the sought-for function was denoted by T symbol. The correct formulation of the boundary value problem for Poisson equation requires four boundary conditions setting. To simplify the solution, all boundary conditions were equated to zero.

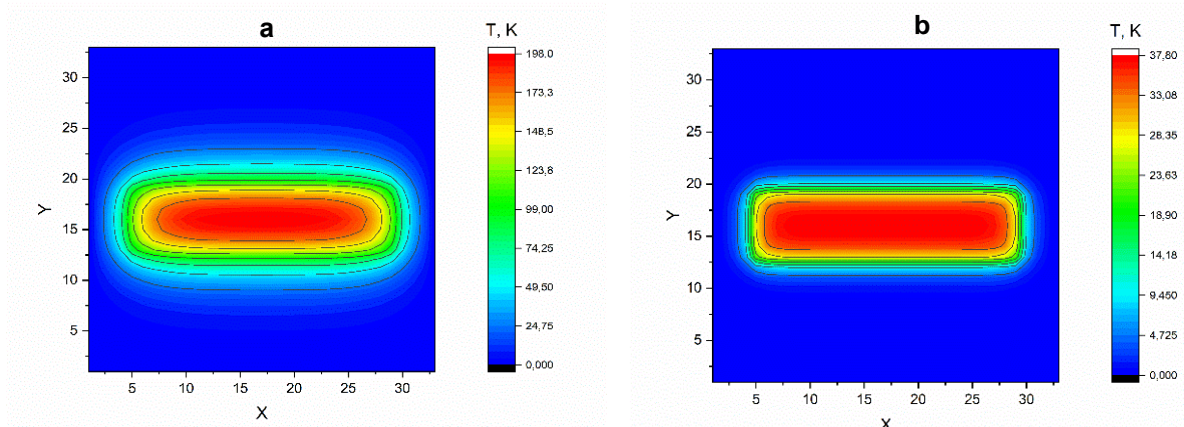


Figure 5. Lines of equal heating levels of the conductor sheath: Sheath – PVC $\lambda = 0.2$ Wt/m·K (a); Sheath – composite $\lambda = 1.3$ Wt/m·K (b).

The solution was carried out in MathCad environment applying numerical method using the built-in Relax function. The calculation results are presented in Fig. 5 as equal temperature levels on the plane.

Fig. 5 reveals that similar energy load on the conductor made from traditional polyvinyl chloride can cause the temperature rise at the metal-polymer interface exceeding 200 °C. At these temperatures, the destructive processes occur in the PVC composition, accompanied by irreversible deterioration of physical, mechanical and electrical properties [26]. But similar energy load on the heat-conducting composite does not cause the temperature rise in the system over 40 K, relative to the original.

3.4. Electrical properties of the composition

The application of hexagonal boron nitride poses the question of why boron nitride possessing high thermal conductivity, does not conduct electricity. Polar B-N bonds interfere with electron transfer, thus boron nitride in this form is not an electrical conductor, unlike graphite conducting electricity through the Pi Bonds network in the plane of its hexagonal crystals.

The available theoretical concepts do not contradict the nature of the dependence of the polymer composition electrical resistance on the boron nitride content and the fact that the percolation point is manifested at the filler content in the composition of at least 0.16 volume fractions (see Eq. 2).

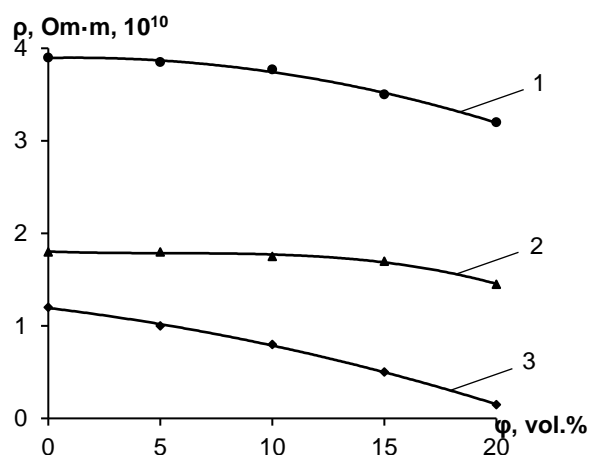


Figure 6. The electrical resistivity dependence (ρ) on boron nitride content: 1 – at 25 °C; 2 – at 50 °C; 3 – at 75 °C.

Fig. 6 shows the experimental dependences of the sealing material resistivity on the temperature and filler content in the form of specific electrical conductivity dependence on the boron nitride content at different temperatures. The measurements were carried out at 1000 V voltage at the electrodes.

In general, temperature increase causes a decrease in electrical resistance of sealing materials. At 25 °C, the dependence is downward (see Fig. 6, curve 1). When temperature reaches 50 °C, the resistivity decreases by half, but the dependence nature does not change significantly (see Fig. 6, curve 2). Further increase in temperature makes the electrical resistance decrease (see Fig. 6, curve 3). The electrical resistance decrease within the studied temperature range largely depends on ionic conductivity increase.

4. Conclusions

According to the research results, the following conclusions can be drawn:

- Within the limits of preserving rheological properties (up to 20–25 % by volume), the boron nitride introduction causes the increase in mechanical properties; similar effect is observed in the adhesive characteristics studied by the method of determining the tensile strength. The greatest tensile strength is fixed on the steel substrate, the lowest – on aluminum, depending on hydroxyl groups on the steel surface.
- The introduction of 20 % of boron nitride increases the thermal conductivity coefficient up to 1.5 W/m·K.
- The thermal conductivity problem described by the elliptic Poisson equation, was solved by numerical method in MathCad environment applying the built-in Relax function. The temperature distribution during the conductor operation in a sheath from polyvinyl chloride and an oligodimethylsiloxane based composite filled with boron nitride was shown, demonstrating more efficient operation under equal conditions.

- The dependence of electrical resistivity on temperature and filler amount was established: the temperature increase causes the electrical resistivity decrease, associated with polyorganosiloxane ionic conductivity.
- The research results can find wide application in construction industry.

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