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Size dependent thermal conductivity of micron polycrystalline particles and its effect on the properties of polymer composites

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Size dependent thermal conductivity of micron polycrystalline particles and its effect on the properties of polymer composites

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Abstract. *Many micron-sized powders are obtained by crushing polycrystalline materials under intense mechanical stress. This introduces defects into the material and worsens the coefficient of thermal conductivity. The finer the particles are crushed, the higher the defect concentration and the lower the corresponding thermal conductivity coefficient. On the base of this consideration the hypothesis of the dependence of the thermal conductivity coefficient of micron-scale particles on their size and, indirectly, on the production method is formulated and quantitatively confirmed. We have demonstrated this effect based on our own results for thermal conductivity of polymer composites and on a number of experimental results from other authors. An estimate was obtained according to which the thermal conductivity of ~50 μm silicon carbide particles is around 50 W/(m K), and for a small powder of ~1 μm it decreases to 30-40 W/(m K). At the same time, the thermal conductivity of a defect-free SiC single crystal reaches 300 W/(m K) at room temperatures. In general, for particles obtained by mechanical crushing an estimated decrease in the coefficient of thermal conductivity with a decrease in size from 100 to 1 μm can be 20-40%. Moreover, we have found and described a number of examples where the thermal conductivity of the composite does not change as the powder size decreases. And in all such cases, micro-powders were obtained not by crushing, but during synthesis or solidification from the melt. Such examples correct the generally accepted opinion about the key role of interfacial thermal resistance and confirm our hypothesis. At low concentrations of 1 μm SiC particles, the effect of the thermal conductivity of the polymer-filler interfacial layer on the thermal conductivity of the composite is shown and discussed. The work provides an important theoretical basis for the creation of a new technique for indirect measurement of the thermal conductivity of micron-sized particles.*

Introduction. Polymers have a number of practically important properties, such as elasticity, machinability, chemical inertia, and dielectric properties, which are indispensable in many applications such as electrical insulation, automobile tires, packaging, etc. [1]. A significant disadvantage of polymers is their low thermal conductivity. For example, for many tasks of microelectronics and LED technology, there is a need to dissipate large heat fluxes while maintaining the electrical insulating properties of interface materials. One of the solutions for the problem is the creation of polymer composites with thermally conductive fillers. The demands of electronics manufacturers for the thermal interface materials thermal conductivity coefficient are already at the level of 50 W/(m K) [2,3]. However, such characteristics are currently achievable only at the laboratory level, as a rule, with the isotropic orientation of filler particles and at its concentration of 40-90 wt. % [4]. Materials with such a high concentration of fillers no longer look much like polymers. The pursuit of high thermal conductivity due to a high degree of filling greatly reduces elasticity and electrical insulation properties that are important for practice. Moreover, many heat-conducting fillers are expensive, and their use in large quantities on an industrial scale is not economically justified. We believe that the strategy of obtaining high thermal conductivity due to a high degree of filling does not take into account an important point. How high is the thermal conductivity of microparticles of a material that is a good conductor of heat? Is thermal conductivity preserved during the transition to micron particles? If it persists, under what conditions?

We believe that before increasing the concentration of microparticles in a polymer composite in the fight for high thermal characteristics, it is necessary to understand this issue in more detail.

There are a large number of models for describing the thermal conductivity of filled polymer composites, which are used in selecting the parameters of composite systems. The minimum required parameters for all models are the thermal conductivity of the polymer matrix and the thermal conductivity of the filler material, as well as the filler concentration. Taking into account other factors, for example, the size and morphology of particles, gives certain corrections in the description of the thermal conductivity of the composite, but does not change the magnitude and behavior of the thermal conductivity qualitatively. From the specified basic set, almost all parameters are measured fairly reliably, with errors at the level of units of percent. For example, the thermal conductivity of a polymer matrix is measured using standard laser flash techniques on macroscopic samples with a size of about 10 mm. The average particle size of the filler can be determined by microscopic methods, and the size distribution can be determined by processing arrays of microscopic images or by optical methods based on scattering optical radiation in an aqueous suspension of the measured particles. At the same time, for submicron or even micron filler particles, the thermal conductivity coefficient is almost never measured directly due to the extreme complexity of this task [5]. For example, one of the few studies where such measurements were made used a method in which a micron particle was held in an electromagnetic field and heated by a laser pulse. The thermophoretic force generated and the corresponding displacement of the particle were used to indirectly determine its thermal conductivity coefficient [6]. Therefore, in works where the thermal conductivity of filled polymer composites is calculated, it is assumed by default that the coefficient of thermal conductivity of filler particles is equal to the coefficient of thermal conductivity of a macroscopic monocrystalline or polycrystalline material (for example, see [7], [8]). In our opinion, this assumption needs to be revised. For a number of materials, but not necessarily for all, the coefficient of thermal conductivity may depend on the size of the microparticle. In this paper, we will consider this issue and, using the example of a filler made of silicon carbide powder, demonstrate qualitatively and quantitatively the existence of such an effect, discuss its physical causes and consequences.

The dimensional effect in the thermal conductivity of polymer composites. Interfacial resistance and its contribution. The existence of a dependence of the thermal conductivity coefficient of a polymer composite on the particle size of the filler has been reported in a number of papers [9]. Perhaps one of the most detailed reviews on this topic was carried out in [10]. The standard explanation for this effect is as follows. Phonon scattering can occur at the polymer-filler interfaces. All other things being equal, at the same concentration, smaller particles have a larger interface area, which leads to more intense phonon scattering and interferes with heat transfer. However, even in the mentioned review [10] it is noted that a number of reports on the effect of particle size on the thermal conductivity of a composite are often contradictory. The change in thermal conductivity is influenced by other factors, the role of which remains unclear against the background of the multiparametric nature of the problem.

Interfacial interaction and interfacial thermal resistance in polymer composites are considered in detail in the review [2]. An overview of molecular dynamics methods for calculating thermal resistance at polymer-filler boundaries is given in [8]. The commonplace of most works on this topic is the statement of the effect of interfacial resistance on the effective thermal conductivity of the composite, but the lack of quantitative estimates for the contribution of this effect. We do not dispute in any way the influence of interfacial boundaries on heat transfer in filled composites, but we believe that the question of the quantitative contribution of this interfacial thermal resistance to the value of the thermal conductivity coefficient remains open.

As another point of view on this problem, let us briefly consider several experimental examples of a different nature. Can composites with very different sizes of the same filler have very close (within the measurement error) thermal conductivity coefficients? In [11], the thermal conductivity of composites based on polyvinyldiene fluoride (PVDF) and epoxy resin with fillers of silicon carbide and aluminum

nitride was studied. In this case, silicon carbide powder was obtained in the Acheson process (first synthesis, then crushing), and aluminum nitride was obtained by direct nitriding, that is, during synthesis. For a PVDF polymer matrix for a filler made of aluminum nitride particles with a concentration of 55 vol.%, a thermal conductivity coefficient of 2.77 W/(m K) was obtained for an average particle size of 2 microns and 2.79 W/(m K) for a particle size of 56 microns (see Table 3 in [11]). The average surface area of such powders, and hence the intensity of phonon scattering, should differ by more than 700 times, and the coefficient of thermal conductivity differs by only 0.7%, which is less than the measurement error.

Polymers with fillers made of metal powders and microparticles can play an important role in this series. In most cases, such powders are also obtained either by chemical synthesis or crystallization from melts, that is, by methods that introduce a small number of defects into the structure. In [12], a thermal conductivity coefficient of 0.412 W/(m K) was obtained for a silicone rubber-aluminum composite with a filler particle size of 12 microns at a concentration of 27.5 vol%. For the same polymer matrix and for the same aluminum filler at a concentration of 27 vol.%, but with a filler particle size of 1.2 mm, the thermal conductivity coefficient is 0.406 W/(m K). There is a difference in the thermal conductivity of the composite of 1% at virtually the same concentration, with a difference in particle size of 100 times, and surface area of 10,000 times. In the same paper [12], the result for the copper particle - epoxy resin composite is given. For a copper particle size of 11 microns and a concentration of 54.6 vol. % the thermal conductivity coefficient was 1.37 W/(m K). At a close concentration of 57.7% and a particle size of 100 microns, the thermal conductivity coefficient also turned out to be 1.37 W/(m K). We believe that a sufficient number of such examples can be found.

It is important for our work that such equality of thermal conductivity coefficients does not fit into the paradigm of interfacial thermal resistance. There must be other reasons for this effect. The main idea of our work is that along with the interphase thermal resistance (phonon scattering at the boundary), there is also the effect of the dependence of the thermal conductivity coefficient of microparticles on the size. We will try to describe and justify the physics of this dependence below. It is important that there are a number of experimental facts that cannot be explained solely by the resistance of the interfacial boundaries. The use of a well-founded concept of the dependence of the coefficient of thermal conductivity on the size for certain categories of microparticles obtained by crushing allows us to consistently explain a large set of experimental facts.

Experimental results. Consider the following experimental situation. Let's say we have a set of polymer composite samples that have the same matrix and the same filler material with the same concentration. The only difference between the samples will be the size of the filler particles. And this size should differ as much as possible. If the thermal conductivity coefficient of the filler does not depend on the size, we must obtain the same value of the thermal conductivity coefficient for all samples, as described in several examples above. The difference from these examples will also be in the powder material and the method of its preparation. This is what plays a key role in understanding the discovered effect.

Sample preparation. Combinations of two polymer matrices, high-pressure polyethylene (HDPE) and polyamide (PA), with silicon carbide powders, were selected as samples. Silicon carbide powders with a grain size of F220 (according to the FEPA standard) were used to fill the composites, which corresponds to M50 (according to GOST 3647-80) and F2000 (M1). In the first case of the larger F220 powder, the size of the main fraction of silicon carbide particles is in the range of 45-63 μm (the average size is 54 μm), in the second case for F2000 – 1 μm (Figure 1). Thus, the particle sizes of the filler differed by about 50 times. Silicon carbide was chosen for the reason that the history of its production is well known. And this is important for a general understanding and explanation of the totality of experimental facts. Each sample was prepared based on the mass fraction of the components and the mass fraction of the filler μ_s. After its manufacture, the density of the sample was measured

ρ_{com}, which made it possible to recalculate the actual volume concentration ν_s filler using the formula $\nu_s = \mu_s \rho_{com}/\rho_s$. Here ρ_s is the density of the filler material. For silicon carbide, this is 3210 kg/m^3. Thermal conductivity measurements were carried out using a laser flash on an LFA device. A description of the composition of samples of filled polymer composites and the results of measuring the coefficient of thermal conductivity are presented in Table 1.

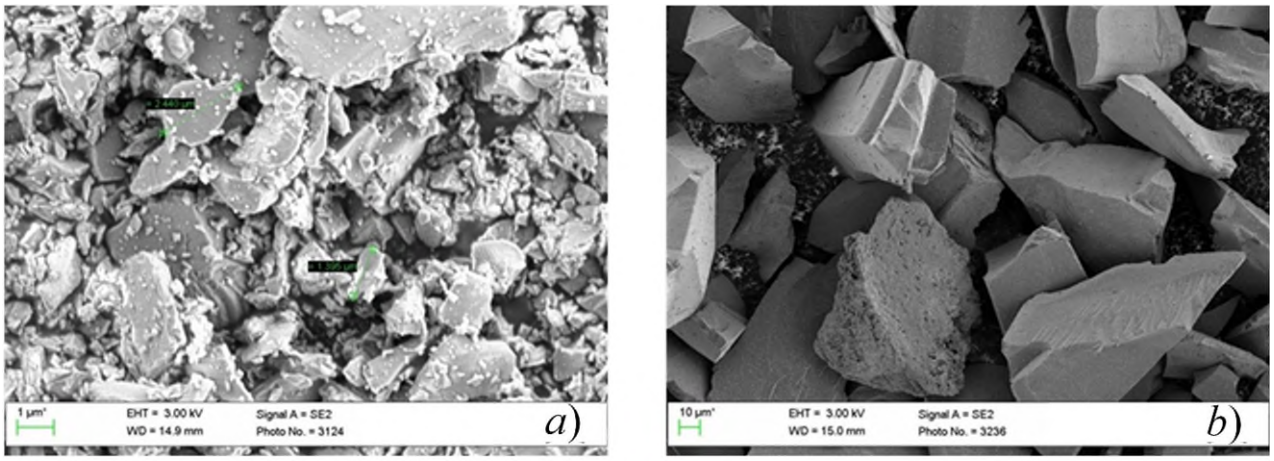


Figure 1. SEM image of silicon carbide powders of fractions F2000 (M1) a) and F220 (M50) b) used as a filler for a polymer composite.

Table 1. Composition of samples of filled polymer composites and measurement results of their thermal conductivity coefficient.

#	Polymer matrix	Filler	Filler concentration		λ_{com}, W/(m·K)	ρ_{com}, kg/m^3
			mas. %	vol. %		
1	HDPE	SiC, F220, ~54 µm	0,5	0,14(2)	0,269 ± 0,013	911
2	HDPE	SiC, F2000, ~1 µm	0,5	0,14(2)	0,275 ± 0,014	910
3	HDPE	SiC, F220, ~54 µm	20	6,44	0,405 ± 0,02	1034
4	HDPE	SiC, F2000, ~1 µm	20	6,44	0,292 ± 0,015	1033
5	HDPE	SiC, F220, ~54 µm	60	27,12	1,046 ± 0,05	1451
6	HDPE	SiC, F2000, ~1 µm	60	26,92	0,851 ± 0,04	1440
7	PA	SiC, F220, ~54 µm	0,5	0,17(3)	0,270 ± 0,014	1110
8	PA	SiC, F2000, ~1 µm	0,5	0,17(8)	0,287 ± 0,014	1140
9	PA	SiC, F220, ~54 µm	20	7,85	0,416 ± 0,02	1260
10	PA	SiC, F2000, ~1 µm	20	8,10	0,367 ± 0,018	1300
11	PA	SiC, F220, ~54 µm	60	36,82	1,600 ± 0,08	1970
12	PA	SiC, F2000, ~1 µm	60	34,54	1,245 ± 0,06	1847

Analyzing the results obtained, we note the following. For high filler concentrations of 20 and 60 vol.% we observe a difference in the coefficient of thermal conductivity for samples at the same concentrations and polymer matrices. All the studied combinations of samples showed the same behavior – a significant difference in the coefficient of thermal conductivity when using a small and large silicon carbide filler at the same concentration and for the same polymer matrices. The increase in the thermal conductivity coefficient ranged from 13% for a pair of samples 9, 10 to 38% for samples 3,4 (Table 1). On average, it was 26% for the studied combinations. Such an increase in thermal conductivity cannot be explained by measurement error. We believe that the most likely possible reason explaining this effect is related to the dependence of the thermal conductivity coefficient of filler microparticles on their size. It is important to note that from the analysis of experimental data based on this hypothesis, it follows that the coefficient of thermal conductivity of particles should decrease with a decrease in their size.

Below we will provide arguments in support of this hypothesis. As noted above, another possible reason for the decrease in the thermal conductivity of the composite under the described conditions could be additional thermal resistance at the polymer–filler interface [13]. Below, we will try to assess the quantitative effect of these two factors on the thermal conductivity of the filled polymer composite.

Discussion of the results. What could be the reason for the discovered effect of the size of micron particles on the coefficient of thermal conductivity? We believe that special attention in the considered experimental example should be paid to the powder production method. For silicon carbide, the first stage of production is the Acheson process [14]. In this process, an array of macroscopic silicon carbide crystals of the order of ~ 5-15 mm is grown in a high-temperature industrial furnace at temperatures in the core of the charge up to 2500 °C (Figure 2). After cooling the product by mechanical crushing of crystals using grinding bodies or in gas jet impingement, the corresponding powders are obtained [15]. Any crushing introduces defects into the crystal. It is important that defects have almost no effect on the heat capacity and temperature coefficient of linear expansion, but they strongly affect the thermal conductivity and, in particular, the thermal conductivity of silicon carbide crystals [16]. The thermal conductivity coefficient of a crystal directly depends on the number and density of defects [17]. The finer the powder, the more collisions it has undergone, the more potential defects it has accumulated, and the lower its coefficient of thermal conductivity should be. This is a qualitative explanation of the discovered effect.



Figure 2. Polycrystals of silicon carbide with a characteristic size of 5-15 mm obtained in the Acheson industrial process.

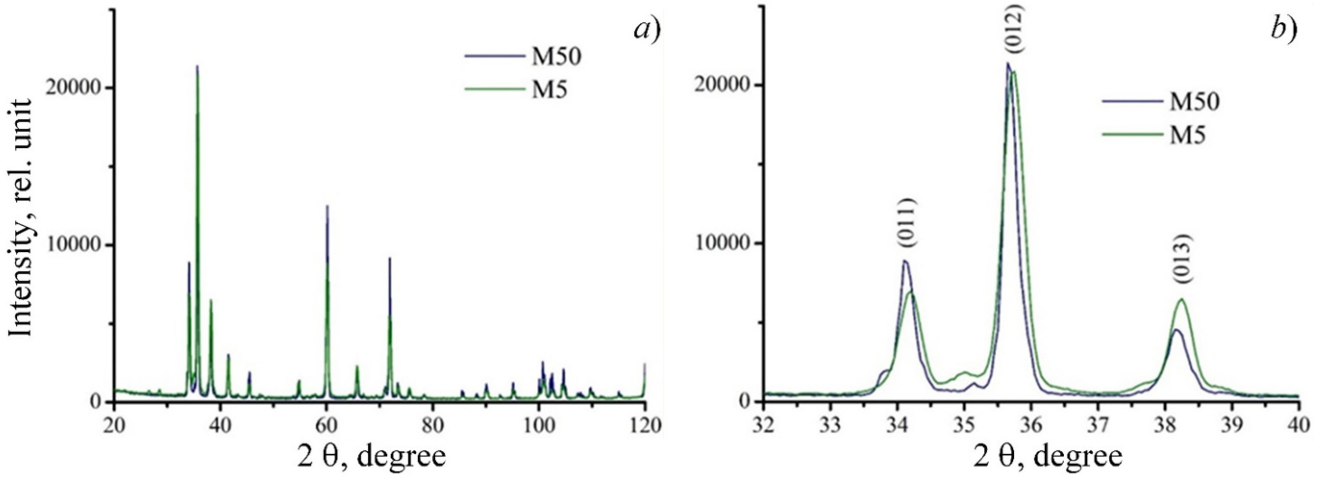


Figure 3. XRD-pattern of silicon carbide powders of the fractions M50 and M5 used in the work in the range of 20-120° 2θ (a) and an enlarged fragment of this radiograph in the range from 32 to 40° 2θ (b)

Table 2. Peak widths at their half-height for the diffractogram of silicon carbide powders of fractions M50 (F220) and M5 (F2000).

The crystallographic plane 6H SiC corresponding to the signal	Width at half height, 2θ	
	M50 (F220)	M5 (F2000)
(011)	0,283	0,403
(012)	0,247	0,358
(013)	0,383	0,400
(110)	0,308	0,425
(116)	0,316	0,475

An indirect confirmation of our reasoning can be the results of [18], which describes the effect of an anomalous decrease in the thermal conductivity of micron diamond particles from 2000 W/(m K) to 400 W/(m K) with a decrease in particle size from 300 to 20 microns. The authors attribute this effect to *the internal defects* of diamond microparticles during their growth. We also note that the above examples of composites, in which the thermal conductivity coefficient did not change even by orders of magnitude when the particle size changed, can be characterized by one common quality – the filler particles in them were obtained by synthesis methods that introduce fewer defects into the structure of the material.

To confirm our assumptions, we performed X-ray phase analysis of silicon carbide powders of the M50 fraction and the M5 fraction close to M1 in size (average size of 5 microns) (Figure 3). X-ray analysis allows us to draw the following conclusion. In the range from 32 to 40 ° 2θ for silicon carbide, signals from planes (011), (012) and (013) for polytype 6H are allocated. It can be noted that the signals for the M5 are broadened relative to the signals from the M50. The most likely reason for this broadening may be related to an increase in structural defects with a decrease in particle size. Determining the width of peaks at their half-height allows us to obtain semi-quantitative estimates (Table 2). It can be noted that the width at half height for the M50 is on average one and a half times less than this parameter for the M5. This clearly indicates a lower number of defects in a larger fraction and provides additional grounds for assuming that the coefficient of thermal conductivity depends on the particle size.

The effect of the size dependence of the thermal conductivity coefficient for micron particles is also observed for other materials. For example, in [19], the results for the thermal conductivity of an epoxy resin - aluminum oxide composite with a filler concentration of 50 wt.% are presented (see Figure 10 in [19]). For two filler sizes of 10 and 100 μm, the values of the thermal conductivity coefficients of 0.40 and 0.658 W/(m K), respectively, were obtained. Unfortunately, the paper does not specify the source of origin of the powders and their other characteristics, except for the size. In another paper [20], the effect of adding aluminum oxide particles of various sizes to silicone rubber was also experimentally shown. With a volume fraction of aluminum oxide powder of 17 and 28 %, the difference between the thermal conductivity of the composite is 25 and 19%, respectively, for a filler size of 0.5 and 25 μm. Below, we will once again refer to [19, 20] in the quantitative analysis of the problem.

Aluminum oxide is also an abrasive material (corundum). Crushing of larger polycrystals is also one of the stages of obtaining the powder of this material [21]. There are a large number of synthesis methods for aluminum oxide nanopowders, such as synthesis in arc plasma, hydrothermal treatment, sol-gel technology, and deposition [22]. Micrographs of the resulting nanoparticles indicate that they are characterized by an irregular structure and a significant number of internal defects, since they are obtained in rapid processes [23]. Low-defect structures can be obtained only with slow crystal growth. Qualitatively, the results of [19] fit into the theory discussed in the paper on the dependence of the thermal conductivity of particles on the size and method of their preparation.

Boron nitride BN is also a traditional material for thermally conductive polymer composites. The considering effect is also found for this filler. In [24], for the epoxy resin-boron nitride composite, the difference in thermal conductivity coefficients for particles of 0.3 microns and 20 microns reaches 35% at 30 vol.% of the filler concentration. In [25], where, apparently, a more thermally conductive BN powder was used, for an epoxy resin-based composite, the difference in the coefficient of thermal conductivity when using particles of 6 and 180 μm reaches 60% at a filler concentration of 13-35 vol. %. We will also analyze the quantitative data of these studies in more detail below.

Another experimental result obtained by our group earlier could not be explained. Within the framework of the presented approach, this explanation has become possible. In [26], the dependence of the thermophysical properties of a composite made of high-pressure polyethylene filled with various fractions of silicon carbide with a mass content of 60% was investigated. In this work, a stable increase in the coefficient of thermal conductivity was observed with an increase in the average size of the filler: 0,91 W/(m K) for a size of 1 μm; 1,13 W/(m K) for 10-14 μm; 1,43 W/(m K) for 40-50 μm. And this fits within the framework of the theory discussed in the paper. An unexpected result of the work [26] was the thermal conductivity coefficient obtained when using silicon carbide powder synthesized in an electrothermal fluidized bed reactor during carbothermal reduction of quartz sand by coal [27,28]. The granulometric composition of the synthesized powder, studied using a photometric sedimentometer FSH-4, showed that about 80% of its mass is contained in particles up to 14 microns in size with a relatively uniform weight distribution. So, the average mass size of the synthesized powder can be estimated as 10 μm. This filler made it possible to obtain the same coefficient of thermal conductivity for the polymer composite as when using a commercial powder fraction of 40-50 μm in size. When compared with the commercial powder M14 (10-14 μm), which is similar in size, the thermal conductivity coefficient, all other things being equal, is 27% higher for a composite with powder obtained in an electrothermal layer. The key point explaining this difference is that this powder was obtained not by crushing, but by synthesis. The reactor's operating time in fusion mode was about 5 hours [29]. Such a relatively slow synthesis should not generate a large number of defects. Therefore, the thermal conductivity coefficient of the powder synthesized in the electrothermal layer should be higher. And this explains the experimental result obtained. It is important that the synthesized particles have a more developed surface and should, by definition, have a higher interfacial thermal resistance in the composite. However, we observed the opposite effect, which refutes the essential role of interfacial resistance in this case.

Polymer-filler interfacial layer and its effect for nanofillers. One of the key ideas of our work is that powder crushing introduces defects, and defects reduce the thermal conductivity coefficient of microparticles. To what extent can this effect work in the direction of decreasing size? Even if advanced particle crushing methods are used, such as gas jet impingement, the size of the smallest fraction "stops" at approximately 200 nm (Figure 4). Nanoscale powders are obtained primarily by synthesis. For this reason, we believe that at particle sizes of about ~200 nm or less, the described effect should no longer manifest itself.

We believe that for a composite with a nanoscale filler, another effect dominates for the thermal conductivity coefficient. An interfacial polymer layer is formed on the surface of the filler particles, the characteristic thickness of which can range from units to tens of nanometers. Since the specific surface area of such a filler is very large, this modified layer occupies a significant volume fraction in the composite and can affect the thermophysical properties of the composite.

We believe that we have observed the manifestation of this effect at low concentrations in our experimental data. For a low filler concentration (pairs of samples 1,2 and 7,8 in Table 1), the thermal conductivity coefficients are almost identical, taking into account the measurement error. However, one can also see an interesting trend of a small, at the level of several percent, difference in the thermal conductivity of a composite with a finer filler. This fact also does not fit into the framework of the idea

of an increase in interfacial resistance with an increase in the specific surface area of particles. We believe that this can be explained qualitatively by the formation of the polymer-filler interfacial layer and the contribution of the increased thermal conductivity of this layer. Judging by the measurement results, this effect exceeds the effect of high thermal conductivity of a large fraction so much that it inverts the dependence of thermal conductivity on particle size. At high concentrations of filler, this effect is lost against a more significant background, due to the dependence of thermal conductivity on the size of microparticles. As a hypothesis, it can be assumed that the parameters of this interfacial layer may also depend on the concentration of the filler. Perhaps, at high concentrations, the percolation effect from direct particle contacts also works [30]. It is likely that finding the filler concentration at which both effects are equalized and we do not see a difference in the thermal conductivity coefficients will be able to provide us with new information about the microstructure and thermal behavior of the composite.

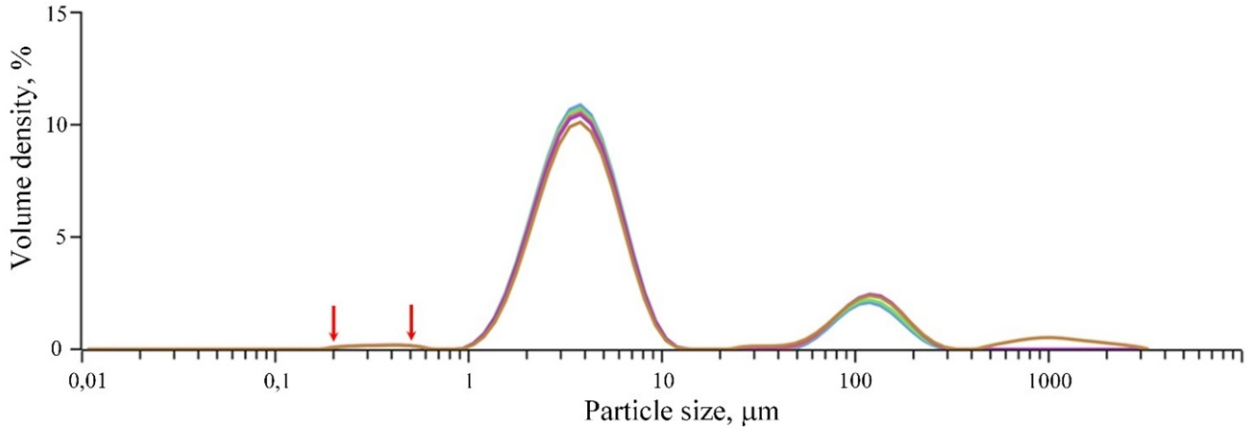


Figure 4. Size distribution for commercial silicon carbide powder of the M5 fraction, obtained using an optical particle size analyzer Mastesizer 3000. There are a small number of nanoparticles with sizes of 200-500 nm.

There are other examples that indirectly confirm our theory. Note that if the idea [13] of increasing the interfacial thermal resistance with a decrease in the particle size of the filler worked, then for nanoscale fillers we would have to observe a multiple decrease in the thermal conductivity coefficient. The facts show the opposite – nanoscale fillers lead to a noticeable increase in the effective coefficient of thermal conductivity of the composite. For example, for polymers filled with carbon nanotubes, at a filler concentration of about 1-2%, the thermal conductivity coefficient of the composite can increase by 20-30 times compared with the thermal conductivity of a pure matrix [31]. The explanation of this fact fits within the framework of the idea of the formation of an interfacial filler-polymer layer with increased thermal conductivity and a significant contribution of this layer under certain conditions.

Why is it possible to assume that the interfacial polymer layer has precisely increased thermal conductivity compared to the volume of the polymer matrix? We believe that the surface of the filler particle causes the polymer chains adjacent to it to orient and partially align perpendicular to this surface. In this case, a single polymer chain provides an effective phonon transport path with low scattering. For example, for a single polymer chain of polyethylene, the group velocity of phonons reaches more than 16 000 m/s. Along with this, the thermal conductivity in crystalline polymer structures can exceed the thermal conductivity of amorphous polymers by an order of magnitude or more [8]. Nanoscale fillers can also serve as crystallization centers, which helps to increase the thermal conductivity of the composite through this mechanism.

To obtain additional arguments in support of our hypothesis about the prevailing influence of thermal conductivity of the interfacial layer for nanoscale fillers, we will make small estimates. Let's consider how the presence of an interfacial layer can affect the concentration at which the effects of

increasing the thermal conductivity of the composite are observed. If the mechanism of increasing thermal conductivity is the direct contact of thermally conductive polymer fillers, then we should observe a noticeable increase in thermal conductivity at a volume concentration of about 30% of the filler, which corresponds to the percolation threshold for three-dimensional continuum percolation model. For many micron-sized fillers in the concentration range of 30-40 vol.% we are seeing this effect. But the picture is different for nanoscale fillers. If the interfacial layer has a coefficient of thermal conductivity comparable to or higher than that of the filler, then the volume of the interfacial layer will determine the effect of increasing thermal conductivity. The characteristic thicknesses of the interfacial layer range from units to tens of nanometers. If we work with a filler with a characteristic size of 10 μm, then the change in the threshold concentration when adding the interfacial layer will be about 1 vol. % or less. The effect associated with the interfacial layer will not manifest itself. If the size of the filler is comparable (equal to) the thickness of the interfacial layer, then it is easy to estimate that the critical concentration of the filler decreases to a value of about 1 vol. %. The remaining conditional 29% of the well-conducting phase is formed due to the interfacial layer. For example, it was shown in [32] that the thickness of the interfacial layer for soot-filled linear polyurethanes is 17 nm. Then, for an average particle diameter of 10 nm, we obtain a value of the critical volume concentration of the filler equal to 0,4%. There are a number of experimental results that report just such an effect. So, in [33], the addition of only 0.9 vol. % of nanoscale copper wires (diameter about 120 nm, length-to-diameter ratio about 100 to 1) into polyacrylate increased the thermal conductivity of the composite by 14.5 times compared with a pure polymer matrix. In another work, when adding 0.2 vol. % for single-walled carbon nanotubes, the thermal conductivity coefficient of the composite increased by 7 times [34]. The thickness of the samples in this case was 1 mm. In [35], when adding 1.46 wt. % of graphene in silicone rubber received an 8-fold increase in thermal conductivity. In [36], when adding another nanoscale carbon allotrope, graphidine (graphdiyne), to a fluoroplast (polyvinylidene fluoride), a 23-fold increase in thermal conductivity was achieved. And the list goes on.

It can be assumed that in these studies, the increase in thermal conductivity can be interpreted by a strong change in the geometry of the filler. Thus, it is known that with a ratio of the length and diameter of filler particles of 100 to 1, the percolation threshold decreases from approx. 29 % to 1.2 vol. % [37]. Let's analyze this situation. When the filler is introduced into the polymer matrix at concentrations corresponding to the percolation threshold, two new substructures appear in the material. The first is a macroscopic cluster of filler particles (fibers) that are connected through mutual touching. Additional thermal resistances occur at the points of contact. These resistances can significantly inhibit heat transfer through the material [38]. The second substructure is the interfacial layer under discussion, which is also a macroscopically large structure extending over the entire sample. We believe that there will be no contact resistance in this structure. If, when these structures were added, the thermal conductivity coefficient in the material increased by an order of magnitude and it reaches absolute values of the order of 1.5 - 5 W/(m K), then it is logical to assume that heat transfer through these new structures determines the properties of the composite. Which of the two substructures, a percolation cluster of contiguous touching particles or a cluster formed by interfacial polymer layers, makes a decisive contribution to heat transfer? It is definitely difficult to answer this question. But certain estimates are possible here. Let's assume that we have removed the entire bulk polymer and all interfacial layers from the material, leaving only a framework of conductive particles. If heat transfer through particles determines an increase in the thermal conductivity of the material by an order of magnitude, then the effective thermal conductivity of the frame of these particles should be comparable to the thermal conductivity of the composite (1-10 W/(m K)). The volume concentration of solid particles of the frame is at the level of 1%, and the porosity of such a structure is about 99%. But such materials are well known – these are aerogels and actively developed materials based on them, called superinsulation. The density of the framework under consideration can be estimated at 20 kg/m^3 for carbon material. Carbon aerogels with a density of 80 kg/m^3 (4 times higher) at room temperatures are characterized by an effective thermal conductivity of the frame of about 0.018 W/(m K) (see Fig. 3b in [39], [40]), which is 2 orders of

magnitude less than the thermal conductivity of the composite under consideration. A review of the work on the thermal conductivity of aerogels made of various materials shows that such a loose fractal framework of solid particles cannot provide any noticeable heat transfer through the solid phase. For example, even for an aerogel composite and ceramic microfibers with a density of 50 kg/m^3, the effective thermal conductivity coefficient is 0.02 W/(m K) [41]. In our opinion, this is a strong argument in favor of the fact that the interfacial layer makes a decisive contribution to the increase in the thermal conductivity coefficient of the composite.

The combination of these, albeit indirect, facts and judgments allow us to conclude that the interfacial layer plays a key role in increasing the thermal conductivity of polymers filled with nanoparticles. We believe that the mechanism of increasing the thermal conductivity of the composite at low filler concentrations is as follows. In an efficiently working material, the concentration of nanoscale filler is at the level of 1-2 vol. % makes it possible to form a solid percolation frame, around which a highly heat-conducting interfacial layer of polymer "freezes" in a volume exceeding the volume of the filler by an order of magnitude. The frame particles do not necessarily have to touch each other. The macroscopic connectivity condition is touching and/or overlapping of the interfacial polymer layers.

Theoretical estimates for the thermal conductivity of microparticles. When constructing a theoretical model for the thermal conductivity of a polymer composite with a filler, we make certain assumptions. As a rule, the main of such assumptions is the uniformity of the distribution of fine filler over the volume of the polymer matrix and the correspondence of its coefficient of thermal conductivity to the coefficient for bulk material. The uniformity of distribution is influenced not only by the actions of the experimenter during sample preparation, but also by other factors: polymer viscosity, polymer polarity, electrification and cohesion of filler powders, special physical and chemical surface treatment of the filler, sedimentation of the filler during polymerization, etc. The sample preparation technique allows us to assume that the condition for uniform distribution of the filler in the case of our samples in progress. Then we will analyze the simplest limiting cases for the thermal conductivity of the composite in order to obtain a method for quantifying the thermal conductivity of the filler.

Let's consider the estimates from below and from above for the thermal conductivity coefficient of a polymer composite with filler. To do this, we use approximations of interpenetrating continua based on the model of plates oriented parallel and perpendicular to the heat flow, respectively. Thus, the thermal conductivity of a medium (composite material) λ_{com} formed by plates normal to the flow (estimated from below) is defined as

$$\lambda_{com} = \lambda_{min}(p) = \frac{1}{\frac{p}{\lambda_1}+\frac{1-p}{\lambda_2}} = \frac{\lambda_1\lambda_2}{\lambda_2 p+\lambda_1(1-p)}. \quad (1)$$

Here λ_1 and λ_2 are the coefficients of thermal conductivity of the filler and the polymer matrix, respectively, p are the volume fraction of the filler (p= v_s). For the second limiting case of a medium consisting of cylinders parallel to the flow (estimated from above), the conductivity is determined by the equation

$$\lambda_{com} = \lambda_{max}(p) = p\lambda_1 + (1-p)\lambda_2. \quad (2)$$

Expressions (1) and (2) are exact solutions of the Laplace equation corresponding to a homogeneous flow and a homogeneous field. In turn, the equation of heat transfer in the stationary case degenerates into the Laplace equation.

If no special treatments have been carried out with the filler or polymer material that significantly change the properties of the polymer or filler material itself, and if an interfacial layer with a thickness comparable to the particle size of the filler does not form at the boundary between the filler and the polymer (for this, the particle size must be greater than 100 nm), then by definition the thermal conductivity coefficient of the composite does not it can be more than (2) and less than (1). If additional

analysis methods are used, this simple approach may be useful for analyzing the properties and contribution of both the interfacial layer to the thermal conductivity of the composite, as well as for analyzing the thermophysical properties of the filler particles themselves.

Suppose that

$$\lambda_{com} = g\,\lambda_{max}(p) + (1-g)\lambda_{min}(p), \quad 0 \le g \le 1. \tag{3}$$

Here, g is the weighting factor, which is an unknown parameter of the problem. Let's perform transformations of equation (3) taking into account (1) and (2). This leads to a quadratic equation for the thermal conductivity of the filler particle material, assuming that we know all the other thermal conductivity coefficients and the g parameter:

$$gp(1-p)\lambda_1^2 + \{[1-2gp(1-p)]\lambda_2 - (1-p)\lambda_{com}\}\lambda_1 + \{g(1-p)\lambda_2 - \lambda_{com}\}p\lambda_2 = 0 \tag{4}$$

The solution of this equation gives an expression for finding the coefficient of thermal conductivity of the filler particle material, assuming that we know the value of the coefficient g:

$$\lambda_{1,\pm} = \frac{1}{2gp}\lambda_{com} \pm \frac{\sqrt{[1-2gp(1-p)]\lambda_2^2 - [1-2gp]2(1-p)\lambda_{com}\lambda_2 + (1-p)^2\lambda_{com}^2}}{2gp(1-p)} \tag{5}$$

Note that equation (5) has two solutions, $\lambda_{1,-}$ and $\lambda_{1,+}$, corresponding respectively to the subtraction or addition of the second term. A study of solutions (5) showed that $\lambda_{1,-}$ gives strongly underestimated values of the thermal conductivity coefficient, and realistic values for the thermal conductivity of filler particles are given only by the second solution $\lambda_{1,+}$. This is what we will consider below. The unknown coefficient g does not allow us to determine the thermal conductivity coefficient of a particle unambiguously from equation (5). But equation (5) allows us to draw a number of interesting and important conclusions.

To fix the value of the free parameter g, we use the following data processing technique. For any general reasons, we will determine at least approximately the coefficient of thermal conductivity for a large fraction of the filler. Then, based on equation (5), we will find the value of the coefficient g, which, with other specified parameters, will give the value of the thermal conductivity coefficient of the composite corresponding to the experimental one. Next, for the second sample from this pair of samples with the same polymer matrix and the same filler concentration, but with a *small fraction*, we take *the same value of the coefficient* g and calculate the thermal conductivity of the filler material for it, which will give an experimental value. Thus, for a large fraction, we consider the unknown value of the g factor, and for a small fraction, the value of the thermal conductivity coefficient λ_1.

For the thermal conductivity coefficient of silicon carbide, we will first use the results of the review [42]. It can be noted that $\lambda_{SiC} \approx 50 \pm 10$ W/(m K) is one of the most common ranges of thermal conductivity values for both technical silicon carbide ceramics (see, for example, Fig. 5 in [43]; Fig. 7 in [44]) and polycrystals with small faces (7 in [42]) at room temperature. Moreover, a comparison of our experimental data with the simulation results for a new model that does not contain empirical parameters [30], for composites with a matrix of polyethylene and polyamide, and with fillers of silicon carbide, showed that the value of $\lambda_{SiC} \approx 50 \pm 10$ W/(m K) most accurately describes the thermal conductivity of the composite. All of these arguments allow us to select $\lambda_1 = 50$ W/(m K) for one of the comparison options. The comparison results for this variant are presented in Table 3.

Analyzing the results of experimental data processing, we note the following. The presented quantitative analysis consistently confirms our assumptions about a decrease in the coefficient of thermal

conductivity of particles with a decrease in their size. If we assume the thermal conductivity coefficient of the large fraction $\lambda_1 = 50$ W/(m K), then for the small fraction for high concentrations of filler, the proposed processing method gives a value of 30-40 W/(m K) for HDPE and about 40 W/(m K) for the PA matrix (Table 3). It looks quite physical and fits into all the assumptions made. However, to unambiguously determine the thermal conductivity coefficient of all components, a more accurate mathematical model is needed that does not contain free parameters.

Table 3. The results of comparing the measured values of the thermal conductivity of the polymer composite and those calculated according to equation (5). The thermal conductivity of the large SiC fraction is estimated as 50 W/(m K) [42-44].

#	Polymer	Filler	p	λ_2	λ_{com}	g	$\lambda_{1,+}$
1	HDPE	SiC, F220, ~54 μm	0,00142	0,27	0,269	2,040	50,0
2	HDPE	SiC, F2000, ~1 μm	0,00142	0,27	0,275	2,040	51,2
3	HDPE	SiC, F220, ~54 μm	0,064	0,27	0,405	0,082	50,0
4	HDPE	SiC, F2000, ~1 μm	0,064	0,27	0,292	0,082	**30,6**
5	HDPE	SiC, F220, ~54 μm	0,271	0,27	1,046	0,064	50,0
6	HDPE	SiC, F2000, ~1 μm	0,269	0,27	0,851	0,064	**39,2**
7	PA	SiC, F220, ~54 μm	0,00173	0,27	0,287	1,823	50,0
8	PA	SiC, F2000, ~1 μm	0,00178	0,27	0,27	1,823	**45,1**
9	PA	SiC, F220, ~54 μm	0,079	0,27	0,416	0,070	50,0
10	PA	SiC, F2000, ~1 μm	0,081	0,27	0,367	0,070	**39,9**
11	PA	SiC, F220, ~54 μm	0,368	0,27	1,6	0,076	50,0
12	PA	SiC, F2000, ~1 μm	0,345	0,27	1,245	0,076	**40,1**

The value of the proportionality coefficient g, obtained as a result of the implementation of the data processing algorithm, is noteworthy. For high concentrations of filler, it assumes physically correct values of ~ 0,06-0,08 for $\lambda_1 = 50$ W/(m K)(Table 3). This means that a more precise model for the composites under consideration is the approximation of conductive inclusions oriented perpendicular to the direction of the heat flow. The value of the g coefficient is also interesting for small filler concentrations of about 0,5% by weight. It is $g \approx 1{,}8 - 2{,}0$, which is allowed mathematically, but within the accepted physical assumptions means only one thing – there is some other mechanism that affects the increase in thermal conductivity of the composite. This mechanism is especially pronounced at low concentrations and small sizes of the filler, when the thermal conductivity of the particles themselves has no significant effect. From our point of view, the increased thermal conductivity of the polymer-filler interfacial layer discussed above can be such a mechanism.

We will provide examples and similar quantitative estimates for other types of fillers to further support the hypothesis and its implications. As noted above, the effect of the dependence of the coefficient of thermal conductivity on the size of filler microparticles was observed when using corundum. Micro-powders of this material are also obtained by crushing crystals. In [20], the thermal conductivity of a composite based on silicone rubber filled with corundum was investigated. The powder sizes were 0.5 and 25 μm, i.e. they differed by 50 times. We assumed that the thermal conductivity coefficient of a powder measuring 20 μm is equal to 14 W/(m K) based on the estimates made in [45]. Otherwise, the assessment methodology was similar. We found the coefficient g corresponding to the thermal conductivity of the composite for the large fraction, and with the same value of the coefficient g, the coefficient of thermal conductivity for the small fraction was determined. Along with these data, we reviewed the data from [20], where the polymer matrix was epoxy resin, and the sizes of corundum were 10 and 100 μm. In this case, for the self-consistency of the estimates, we assumed that for a fraction

of 10 μm, the coefficient of thermal conductivity is also 14 W/(m K), and based on this, the estimate was performed for the thermal conductivity of an even larger fraction. The calculation results are shown in Table 4. In this case, realistic estimates were also obtained for the thermal conductivity of particles from 0.5 to 100 μm, which fit into the physical picture of the phenomenon proposed in the work. It is important that for three different filler concentrations, the proposed approximation gives virtually the same estimate of the thermal conductivity of the smallest fraction of 10.3-10.6 W/(m K). Note that for the largest fraction of 100 μm, the estimate of thermal conductivity of 25 W/(m K) approaches the value of thermal conductivity for macroscopic sintered material of 33 W/(m K) [46].

Table 4. Results of comparison of experimental values of thermal conductivity of polymer composites and calculated according to equation (5) for filler Al_2O_3. The thermal conductivity coefficient of Al_2O_3 particles with a size of ~20 μm is taken from [45].

#	Polymer	Filler	p	λ_2	λ_{com}	g	$\lambda_{1,+}$	Reference
1	Silicone rubber	Al_2O_3, 0,5 μm	0,165	0,12	0,130	0,0448	10,4	[21]
2	Silicone rubber	Al_2O_3, 25 μm	0,165	0,12	0,175	0,0448	**14,0**	[21]
3	Silicone rubber	Al_2O_3, 0,5 μm	0,284	0,12	0,186	0,0375	10,3	[21]
4	Silicone rubber	Al_2O_3, 25 μm	0,284	0,12	0,230	0,0375	**14,0**	[21]
5	Silicone rubber	Al_2O_3, 0,5 μm	0,444	0,12	0,249	0,0322	10,6	[21]
6	Silicone rubber	Al_2O_3, 25 μm	0,444	0,12	0,303	0,0322	**14,0**	[21]
7	Epoxy resin	Al_2O_3, 10 μm	0,245	0,19	0,400	0,0944	**14,0**	[20]
8	Epoxy resin	Al_2O_3, 100 μm	0,245	0,19	0,658	0,0944	25,1	[20]

Similar estimates can be performed for another traditional filler, boron nitride. The peculiarity of this material is the strong anisotropy of thermal conductivity in various directions. In many cases, the filler particles have the shape of plates. Probably, these features are reflected in the behavior of the thermal conductivity coefficient. We took two papers for the assessments. In both works, epoxy resin was used as a polymer matrix. In [24], the particle size of the filler varied from 0.3 to 20 μm. In the same work, the thermal conductivity coefficient for boron nitride was given as 68 W/(m K), which we used for estimates according to our methodology (Table 5). The estimates obtained fully correspond to the physical picture of the phenomenon proposed in the work. Again, we note a fairly small range of estimates for the thermal conductivity of both the fine fraction of 39-44 W/(m K) and the fraction of 9 μm – 54-58 W/(m K).

Table 5. The results of comparing the experimental values of the thermal conductivity of polymer composites and those calculated by equation (5) for the BN filler according to [24]

#	Polymer	Filler	p	λ_2	λ_{com}	g	$\lambda_{1,+}$
1	Epoxy resin	BN, 0,3 μm	0,10	0,192	0,228	0,0312	40,6
2	Epoxy resin	BN, 9 μm	0,10	0,192	0,273	0,0206	54,0
3	Epoxy resin	BN, 20 μm	0,10	0,192	0,317	0,0328	68,0
4	Epoxy resin	BN, 0,3 μm	0,20	0,192	0,299	0,0312	44,4
5	Epoxy resin	BN, 9 μm	0,20	0,192	0,357	0,0206	58,3
6	Epoxy resin	BN, 20 μm	0,20	0,192	0,397	0,0328	68,0
7	Epoxy resin	BN, 0,3 μm	0,30	0,192	0,517	0,0312	39,1
8	Epoxy resin	BN, 9 μm	0,30	0,192	0,678	0,0206	55,4
9	Epoxy resin	BN, 20 μm	0,30	0,192	0,798	0,0328	68,0

In other work with boron nitride, high quality and high purity of the material are noted [47]. The authors note the high anisotropy of the thermal conductivity of the material – 600 W/(m K) in one plane, and 30 W/(m K) in the other plane. Therefore, for estimates in this case, we took the average between these values – 300 W/(m K). The evaluation results are shown in Table 6. Among other things, attention is drawn to the fairly close values of the coefficient $g \approx 0.02$-0.04 obtained for different composites (Tables 4-6).

Table 6. Results of comparison of experimental values of thermal conductivity of polymer composites and calculated by equation (5) for BN filler according to [47].

#	Polymer	Filler	p	λ_2	λ_{com}	g	$\lambda_{1,+}$
1	Epoxy resin	BN, 2 μm	0,128	0,197	0,402	0,0228	100
2	Epoxy resin	BN, 30 μm	0,128	0,197	0,721	0,0240	209
3	Epoxy resin	BN, 180 μm	0,128	0,197	0,994	0,0282	**300**
4	Epoxy resin	BN, 2 μm	0,257	0,197	0,873	0,0228	121
5	Epoxy resin	BN, 30 μm	0,257	0,197	1,624	0,0240	242
6	Epoxy resin	BN, 180 μm	0,257	0,197	1,988	0,0282	**300**
7	Epoxy resin	BN, 2 μm	0,340	0,197	1,305	0,0228	121
8	Epoxy resin	BN, 30 μm	0,340	0,197	2,345	0,0240	229
9	Epoxy resin	BN, 180 μm	0,340	0,197	3,028	0,0282	**300**

In general, it can be noted that all the experimental data presented do not contradict our assumptions about the dependence of the thermal conductivity coefficient of microparticles on their size for the case when these particles were obtained by breaking and crushing larger ones. *We also note that it is possible that the contribution of the interfacial thermal resistance discussed is present in our estimates of the effective thermal conductivity of microparticles.* Special experimental and theoretical work is required to evaluate this contribution.

Conclusions.

The paper proposes and substantiates a hypothesis about the dependence of the thermal conductivity coefficient of microparticle on the size. The dependence is due to the method of obtaining micron powders and is associated with the creation of defects during their crushing. A method for quantifying the thermal conductivity of micron powders is proposed. The assumption about the key influence of the thermal conductivity of the particle-polymer interfacial layer on the thermal conductivity of polymer composites filled with nanoparticles is considered and substantiated. We believe that the proposed quantitative analysis will make it possible to further contrast the effect of the two mechanisms on the coefficient of thermal conductivity – the particle size-dependent properties and the properties of the interfacial layer. This will probably allow us to create a methodology for assessing the as yet unknown thermal conductivity of the interfacial layer. Moreover, the proposed approach and analysis can become the theoretical basis for constructing a technique for indirectly measuring the thermal conductivity of micron-sized particles.

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Declaration of interests.

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

P.S. Grinchuk: Writing – review & editing, Writing – original draft, Visualization, Methodology, Conceptualization. S.M. Danilova-Tretiak: Writing – review & editing, Validation, Formal analysis, Data curation, Conceptualization. K.V. Nikolaeva: Validation, Data curation, Experimental investigation (thermal properties). L.E. Evseeva: Validation, Data curation, Experimental investigation (thermal properties). V.B. Shcherbakova - Validation, Data curation, Experimental investigation (XRD analysis). Writing – part of original draft,

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