

A Universal Particle-Size-Dependent Calculation Formula for the Thermal Conductivity of Epoxy/Barium Hexaferrite ($\text{BaO} \cdot 6\text{Fe}_2\text{O}_3$) Two-Phase Composites

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Abstract

The thermophysical behaviour of dispersion-filled polymer composites can be tailored over a wide range by varying the type, the loading and, importantly, the dispersity of the filler introduced into the matrix. The classical relations most often used to estimate the effective thermal conductivity of such two-phase systems are formulated only in terms of the conductivities of the constituent phases and their fractions, and contain no explicit dependence on the size of the filler particles; consequently, no single classical relation can reproduce the experimental conductivity curves obtained for the same composition but for fillers of different particle diameter. In the present work, a single particle-size-dependent calculation formula is proposed for the effective thermal conductivity λ of a two-phase composite and is validated experimentally. Composites were prepared from ED-20 epoxy-diane resin, cured with a polyethylene-polyamine hardener, and filled with M-type barium hexaferrite ($\text{BaO} \cdot 6\text{Fe}_2\text{O}_3$) powder of two dispersities, with effective particle diameters of 2 μm and 20 μm , at filling degrees of 0% - 60% by mass; the thermal conductivity was measured with an IT- λ -400 instrument. The proposed formula, which supplements the linear rule of mixtures with a correction term proportional to the square root of the effective particle diameter, reproduces the measured curves for both the 2 μm and the 20 μm fillers, whereas the classical size-independent relations cannot describe both data sets simultaneously.

Keywords

Polymer Composite, Barium Hexaferrite, Thermal Conductivity, Mixing Rule, Particle Size, Effective Medium Theory, Epoxy Resin

1. Introduction

Composite materials in which a continuous polymer matrix is structured with a dispersed solid filler occupy an important place in modern materials science and engineering, and interest in such systems continues to grow steadily. The controlled, dosed introduction of ferroelectric, mineral, metallic or carbon particles of different shape and size into a polymer base makes it possible to combine, within a single material, a unique set of chemical, physico-mechanical and service properties that none of the individual components possesses on its own. Among these properties, the thermophysical characteristics of the composite, and in particular its effective thermal conductivity, are of considerable practical significance, because the ability to remove or redistribute heat is decisive for many applications in electrical engineering, electronics and instrument-making, where polymer-based components increasingly operate under appreciable thermal loads.

It is well established that the thermophysical and electrophysical properties of filled polymers depend in a complex manner on a large number of factors: the grade and chemical nature of the matrix polymer, the composition of the system, the shape and size of the filler particles, and the way those particles are distributed throughout the volume of the matrix. The distribution of the dispersed phase can, in the most general case, be regular, statistical or structured, as illustrated schematically in **Figure 1**, and each morphology leads to a somewhat different macroscopic response. The targeted design of polymer composite materials with prescribed thermal properties therefore relies on careful control of the structural organization of the filler within the matrix.

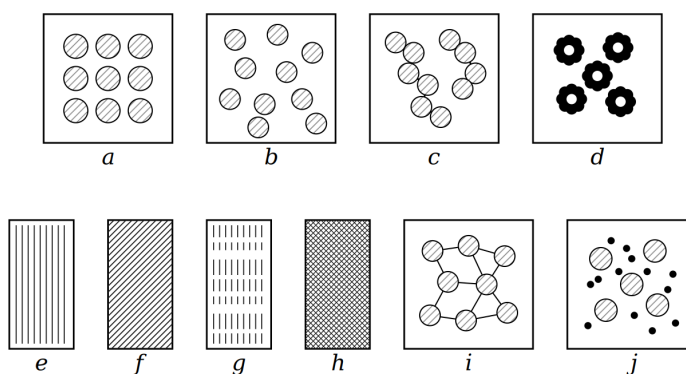


Figure 1. Typical structures of dispersion-filled composite materials and distribution of the filler within the matrix: (a) regular (matrix) packing; (b) statistical (random) distribution; (c) (d) structured (chain-like and network) arrangements; (e)–(j) fibre-reinforced and mixed morphologies.

Among the inorganic fillers used for the modification of polymers, M-type barium hexaferrite ($\text{BaO} \cdot 6\text{Fe}_2\text{O}_3$, equivalently $\text{BaFe}_{12}\text{O}_{19}$) deserves particular attention. It combines high electrical resistivity and low loss with a relatively high permittivity, a pronounced magnetocrystalline anisotropy and good chemical and thermal stability, which together make hexaferrite-loaded polymers attrac-

tive for a number of microwave, magnetic and heat-management applications [1] [2]. Epoxy resin, in turn, is a convenient and widely used matrix for such composites, because it is easy to process at room temperature, is thermochemically stable in the cured state, possesses a high dielectric breakdown strength and is comparatively inexpensive. The combination of a hexaferrite filler with an epoxy matrix therefore constitutes a representative and technologically relevant model system for the study of the thermophysical behaviour of two-phase composites.

A considerable number of mixing and effective-medium relations have been proposed for the prediction of the effective thermal conductivity of two-phase heterogeneous systems [3] [4]. A common and practically important limitation of these classical relations is that each is expressed as a function only of the thermal conductivities of the two phases and of their fractions; none contains, in any form, the characteristic size of the filler particles. Because of this omission, a single classical relation necessarily predicts one and the same conductivity curve for fillers of different dispersity at a given composition, and is therefore unable to reproduce the experimental curves measured for powders of different particle diameter.

The aim of the present work is, accordingly, to propose a single particle-size-dependent calculation formula that predicts the effective thermal conductivity of a two-phase composite as an explicit function of the effective diameter of the filler particles, and to verify this formula against experimental data. For this purpose, ED-20 epoxy composites filled with barium hexaferrite powder of two distinct dispersities, with effective particle diameters of 2 μm and 20 μm , were prepared and characterized over a range of filling degrees, and the measured conductivities were compared both with the classical relations and with the proposed formula.

2. Materials and Methods

2.1. Materials and Sample Preparation

The polymer base of the composites was ED-20 epoxy-diane resin, cured with a polyethylene-polyamine (PEPA) hardener. Under the chosen conditions the resin passes from the liquid to the solid state over approximately 20 min at room temperature, which makes it convenient for the reproducible preparation of filled specimens. The hardener was added to the resin at the manufacturer-recommended ratio and the mixture was stirred mechanically for several minutes until homogeneous; the barium hexaferrite powder was then introduced gradually into the liquid resin and dispersed by continued mechanical stirring before the blend was cast into moulds and left to cure at room temperature, followed by ambient post-curing. The filler was magnetically active M-type barium hexaferrite powder with the chemical formula $\text{BaO} \cdot 6\text{Fe}_2\text{O}_3$. Two separate sets of samples were prepared, differing in the effective diameter of the filler particles: 2 μm in the first set and 20 μm in the second. For each set, the degree of filling of the ED-20 with

hexaferrite powder was varied over the values 0%, 20%, 40% and 60% by mass, expressed as the mass fraction of filler in the composite, $\varphi_2 = m_{\text{filler}}/(m_{\text{filler}} + m_{\text{resin}})$, so that $\varphi_2 = 0, 0.2, 0.4$ and 0.6 with $\varphi_1 + \varphi_2 = 1$, so that the influence of both the loading and the particle size on the thermal conductivity could be examined systematically. Because the classical relations of Section 2.3 are formulated in terms of volume fractions, the mass fractions were converted to volume fractions using the densities of the two phases, $\rho_1 = 1.16 \text{ g}\cdot\text{cm}^{-3}$ for the cured ED-20 matrix and $\rho_2 = 5.2 \text{ g}\cdot\text{cm}^{-3}$ for the barium hexaferrite filler; the resulting volume fractions are given in Section 4.

2.2. Measurement of the Thermal Conductivity

The thermal conductivity λ of the composites was measured with an IT- λ -400 instrument, a standard apparatus for the determination of the thermal conductivity of solid materials by the monotonic-heating method. The specimens were discs of 15 mm diameter prepared to the thickness required by the instrument; two to three specimens were measured for each composition and the reported value is the mean over the specimens. The measurement uncertainty is taken as the rated accuracy of the IT- λ -400 instrument, which is of the order of $\pm 5\%$, combined with the observed scatter between replicate specimens. The measurements were carried out for the full set of compositions and, importantly, for both filler dispersities, so that two complete conductivity curves could be obtained and compared, one for the $2 \mu\text{m}$ powder and one for the $20 \mu\text{m}$ powder. The use of a single, well-defined measurement method for all specimens ensures that the differences observed between the two sets of samples can be attributed to the particle size of the filler rather than to any difference in the measurement procedure. The thermal conductivity of the neat resin was $\lambda_1 = 0.2 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ (the $\varphi_2 = 0$ point), and that of the pure (100%) hexaferrite specimen was $\lambda_2 = 7.6 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$; both were measured in this study with the same instrument.

2.3. Classical Thermal-Conductivity Mixing Models

For the purpose of comparison, six classical relations for the effective thermal conductivity of a two-phase system were compiled from the literature on heterogeneous media [5] [6] and are collected in Table 1. In all expressions the subscripts 1 and 2 refer, respectively, to the matrix (the epoxy resin) and to the filler (the barium hexaferrite); λ_1 and λ_2 denote the thermal conductivities of these two phases; and φ_1 and φ_2 are the corresponding volume fractions (obtained from the mass fractions as described in Section 2.1), which satisfy the normalization condition $\varphi_1 + \varphi_2 = 1$. These relations span the most widely used limiting cases, from the dilute-suspension approximation through the logarithmic and square-root mixing laws to the variational bound, and provide a representative basis against which the proposed particle-size-dependent formula can be assessed.

Table 1. Classical relations for the effective thermal conductivity λ of a two-phase composite.

No.	Model	Effective thermal conductivity λ
1	Maxwell [7]	$\lambda = \lambda_1 \frac{(2\lambda_1 + \lambda_2) + 2\varphi_2(\lambda_2 - \lambda_1)}{(2\lambda_1 + \lambda_2) - \varphi_2(\lambda_2 - \lambda_1)}$
2	Lichtenecker [8]	$\lambda = \sqrt{\frac{\lambda_2 \lambda_1 (\varphi_1 \lambda_1 + \varphi_2 \lambda_2)}{\varphi_2 \lambda_1 + \varphi_1 \lambda_2}}$
3	Wagner	$\lambda = \lambda_2 \left[1 + \frac{3\varphi_1(\lambda_1 - \lambda_2)}{\lambda_1 + 2\lambda_2} \right]$
4	Birchak (square-root)	$\lambda = \left(\varphi_1 \sqrt{\lambda_1} + \varphi_2 \sqrt{\lambda_2} \right)^2$
5	Hashin-Shtrikman [9]	$\lambda = \lambda_1 \left[1 + \frac{\varphi_2}{\frac{\lambda_1}{(\lambda_2 - \lambda_1)} + \frac{\varphi_1}{2}} \right]$
6	Zarichnyak-Novikov [3]	$\lambda = \lambda_1 \varphi_1^2 + \lambda_2 \varphi_2^2 + 4\varphi_1 \varphi_2 \frac{\lambda_1 \lambda_2}{\lambda_2 + \lambda_1}$

3. A Particle-Size-Dependent Universal Relation

At the two endpoints of the composition range, the relations of **Table 1** reduce, as they must, to the thermal conductivity of one or the other of the pure phases; in between, however, each depends only on the conductivities λ_1 and λ_2 and on the fractions φ_1 and φ_2 , and not on the size of the filler particles. To incorporate the influence of the dispersity of the filler, which the experiments show to be substantial, the linear rule of mixtures is here supplemented with an additional correction term. This term is proportional to the contrast between the conductivities of the two phases, to the product $\varphi_1 \varphi_2$ of their fractions (which vanishes identically for either pure phase, so that the endpoints are preserved), and to the square root of the effective particle diameter d . The resulting relation takes the form

$$\lambda = (\lambda_2 \varphi_2 + \lambda_1 \varphi_1) - k(\lambda_2 - \lambda_1) \varphi_1 \varphi_2 \sqrt{d} \quad (1)$$

where λ is the effective thermal conductivity of the composite; λ_1 and λ_2 are the thermal conductivities of the polymer matrix and of the powder filler, respectively; φ_1 and φ_2 are the fractions of polymer and powder, with $\varphi_1 + \varphi_2 = 1$; d is the effective diameter of the filler particles; and k is an empirical constant coefficient. Because the correction term contains the factor $\varphi_1 \varphi_2$, it vanishes both at $\varphi_2 = 0$ and at $\varphi_2 = 1$, so that the relation reproduces the thermal conductivities of the pure matrix and of the pure filler exactly, while its magnitude at intermediate compositions is governed by the effective particle size through the factor $\sqrt{d}$.

The physical basis of the correction is as follows. At a fixed volume fraction, the number of filler–filler contacts and the connectivity of the conductive paths formed by the particles scale inversely with the particle size: a finer powder pre-

sents a larger number of interparticle contacts per unit volume and a more continuous conductive skeleton, and therefore conducts heat more efficiently than a coarse powder of the same loading. The deviation of the measured conductivity from the linear rule consequently increases with the particle size, which the term in $\sqrt{d}$, taken with a negative sign, reproduces. The square-root form is preferred to the simplest alternatives because it provides the mildest monotonic size scaling able to describe both particle-size series with a single constant: a term linear in d over-weights the coarse powder and can drive λ below zero, whereas a term in $1/d$ diverges as $d \rightarrow 0$. The interpretation in terms of interfacial contacts and conductive networks is consistent with established treatments of size effects and interfacial thermal resistance in particulate composites [10]. The constant k is determined empirically by fitting the relation to the combined 2 μm and 20 μm data; the best-fit value is $k \approx 0.092 \mu\text{m}^{-1/2}$ (with d expressed in μm).

4. Results and Discussion

The thermal conductivity of the composites measured with the IT- λ -400 instrument is summarized in **Table 2** for both particle diameters. For both the 2 μm and the 20 μm fillers, the conductivity increases monotonically with the filling degree, as expected when a poorly conducting polymer matrix is progressively replaced by a more conductive ceramic filler; however, at any given loading the finer (2 μm) powder yields a noticeably higher conductivity than the coarser (20 μm) powder—precisely the kind of particle-size effect that the classical relations are unable to reproduce.

Table 2. Measured thermal conductivity λ ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) of the ED-20/BaO-6Fe₂O₃ composite versus filler mass fraction φ_2 for the two particle diameters.

Filler mass fraction φ_2	λ ($d = 2 \mu\text{m}$)	λ ($d = 20 \mu\text{m}$)
0	0.2	0.2
0.2	2.1	1.4
0.4	3.4	2.5
0.6	4.2	3.5

Table 3. Input parameters and conversion of the filler mass fractions to volume fractions ($\rho_1 = 1.16$, $\rho_2 = 5.2 \text{ g}\cdot\text{cm}^{-3}$).

Parameter	Value
Matrix conductivity, λ_1 (measured)	$0.2 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$
Filler conductivity, λ_2 (measured, 100% specimen)	$7.6 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$
Mass fraction $\varphi_2 = 0.2 \rightarrow$ volume fraction	0.053
Mass fraction $\varphi_2 = 0.4 \rightarrow$ volume fraction	0.129
Mass fraction $\varphi_2 = 0.6 \rightarrow$ volume fraction	0.251
Effective particle diameter, d	2 and 20 μm
Fitted size coefficient, k	$0.092 \mu\text{m}^{-1/2}$

As emphasized above, the classical relations of **Table 1** contain no particle-size term, so a single relation predicts one and the same conductivity curve irrespective of the filler diameter. When these relations are evaluated at the true volume fractions (**Table 3**), their predictions fall well below both measured curves: the values calculated from any individual classical relation fail to follow either data set, and the corresponding goodness-of-fit is poor (**Table 4**). The fact that the measured conductivities lie above the simple mean-field estimates is itself consistent with the formation of connected, partially percolating networks of contacting filler particles, whose contribution is not captured by the isotropic mean-field relations and which becomes more effective as the particle size decreases. This is exactly the shortcoming that the proposed particle-size-dependent formula is designed to overcome.

Table 4. Goodness of fit (root-mean-square error, $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) of the classical relations and of the proposed formula against the measured data for the two particle sizes.

Model	RMSE ($d = 2 \mu\text{m}$)	RMSE ($d = 20 \mu\text{m}$)
Maxwell	3.04	2.31
Lichtenecker	2.79	2.05
Wagner	5.01	4.24
Birchak (square-root)	2.65	1.91
Hashin-Shtrikman	3.08	2.35
Zarichnyak-Novikov	2.87	2.12
Proposed formula, Equation (1)	0.45	0.27

Figure 2 compares the thermal conductivity predicted by the proposed relation with the experimental values for both particle diameters. The same relation, used with the same value of the constant k , follows the experimental points for the $2 \mu\text{m}$ and for the $20 \mu\text{m}$ filler, and in particular captures the stronger enhancement produced by the finer powder at a given loading. Quantitatively, the proposed formula reduces the root-mean-square error from $2 - 3 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for the best classical relation to about $0.3 - 0.4 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for both particle sizes (**Table 4**), which confirms that the introduction of the $\sqrt{d}$ term provides an adequate, if simple, description of the observed size dependence.

The residual deviations that remain between the calculated and the measured curves, most clearly visible at the higher filling degrees, may be attributed to the dispersity of the real filler powder. In the calculation a single, fixed effective diameter is used for each set of samples—strictly $2 \mu\text{m}$ or strictly $20 \mu\text{m}$ —whereas the actual powder inevitably contains a distribution of particle sizes about the nominal value, together with a corresponding distribution of interparticle contacts and conducting paths. That a single expression with a single constant is nevertheless able to follow the experimental curves for two quite different particle sizes supports the form of the proposed correction and suggests that a refinement taking the full particle-size distribution into account could further improve the agreement.

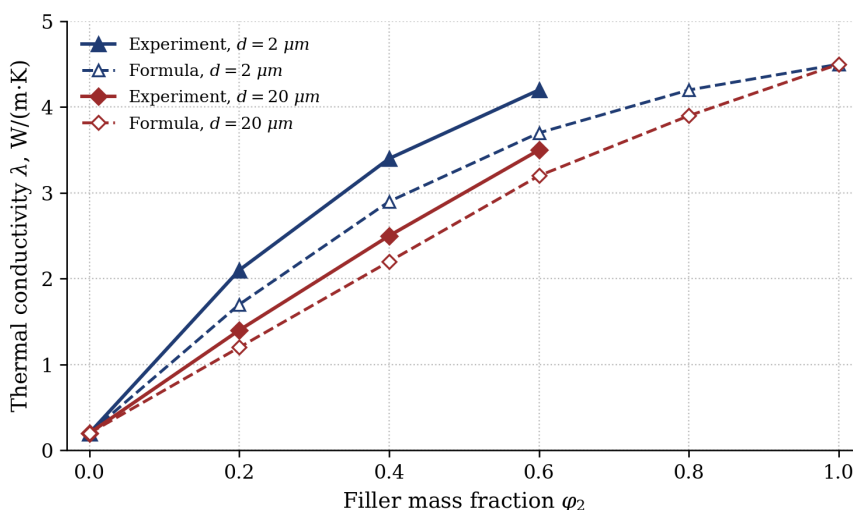


Figure 2. Thermal conductivity predicted by the proposed relation (dashed lines, open symbols) compared with experiment (solid lines, filled symbols) for filler particle diameters of 2 μm and 20 μm .

5. Conclusion

A single particle-size-dependent calculation formula has been proposed for the effective thermal conductivity of two-phase polymer composites. The formula supplements the conventional linear rule of mixtures with a correction term proportional to the contrast between the conductivities of the two phases, to the product of their fractions, and to the square root of the effective diameter of the filler particles, thereby introducing an explicit dependence on the filler dispersity that the classical relations lack. The formula was validated on composites prepared from ED-20 epoxy resin filled with M-type barium hexaferrite ($\text{BaO} \cdot 6\text{Fe}_2\text{O}_3$) powder of two dispersities, with effective particle diameters of 2 μm and 20 μm , at filling degrees of up to 60% by mass, the thermal conductivity of which was measured with an IT- λ -400 instrument. Whereas the classical, size-independent relations cannot reproduce the conductivity curves for both particle sizes at the same time, the proposed formula, using one and the same constant k , follows the measured curves for both the 2 μm and the 20 μm fillers, reducing the root-mean-square error by roughly an order of magnitude. The relation thus offers a simple and convenient means of predicting the thermophysical behaviour of two-phase systems consisting of a carrier matrix and micron-sized filler particles of different diameters, and is of practical interest for the design of tailored, thermally conductive polymer composites for electrical-engineering, electronic and instrument-making applications.

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Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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