



Thermal and mechanical properties of polypropylene-iron-diamond composites



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ABSTRACT

Polypropylene-iron and polypropylene-iron-diamond composites with various amounts of iron and natural or synthetic diamond particles were prepared in a co-rotating laboratory kneader. Test samples were made by injection molding. Heat flow measurements with a differential scanning calorimeter and dynamic mechanical properties in the forced vibration mode were measured from 225 K to 475 K, and thermal diffusivity was measured from 225 K up to 425 K by the laser flash method. Specific heat capacity of the polypropylene-iron-diamond composite can be significantly reduced by the addition of iron particles, while the addition of diamond particles show only a minor influence. Conventional, reversal and non-reversal heat flow measurements are showing two different monoclinic crystal structures (α_1 , α_2) and crystallinities around 50% in the polypropylene. In a heating cycle a conversion between both crystalline structures can appear. Due to different crystallinities and crystal structures the activation energies for the main relaxations at glass transition temperature vary between 275–400 kJ/(mol·K) in the amorphous and between 65–190 kJ/(mol·K) in the crystalline part of the polypropylene. The storage modulus of polypropylene is increased by the iron and diamond particles from 4000 MPa up to 12500 MPa. The thermal diffusivity can be increased from 0.13 mm²/s up to 1.05 mm²/s with iron and up to 1.45 mm²/s with the secondary addition of diamond powder. With increasing temperature the thermal diffusivity decreases. The particle network shows a dominant contribution of the polymer matrix for mechanical properties whereas the particles show a dominant contribution to thermal properties. The mean free path of phonons in polypropylene decreases with increasing temperature, while it decreases up to 300 K and increases then up to 15 times the value of neat polypropylene again for temperatures above 375 K in the composites.

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1. Introduction

Soft magnetic polymer-particle composites become increasingly important in many application, e.g. as magneto-active shape memory materials [1], as magnetic core or as electromagnetic shielding material in planar inductive devices [2]. By means of a novel concept, composites containing soft magnetic iron (Fe) particles and diamond particles were injection molded to spherical grinding tools. The soft magnetic properties of the iron particles enable a position-control of the grinding tool while grinding can be performed by diamond particles embedded in the surface of grinding tool. Fluidic drive allows rotation and cooling of grinding

tool. Magnetic and thermal properties as well as grinding performance are mainly affected by the amount of iron- and diamond particles in the composite. Hence, customized composite materials having defined properties for grinding application can be engineered. On the scientific point of view, the effects of iron- and diamond particle proportions on the thermal and mechanical properties as well as on polymer structure have to be analyzed to calculate mean free path length of phonons. Based on these results, the phenomenological heat transport in the composite will be characterized.

2. Theory

In many theoretical approaches for the calculation of thermal conductivity of composites the resistance of the boundary between

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matrix and particle is neglected. The presence of this resistance is due to an acoustic mismatch evoked by differing Young's moduli of particles and matrix material. In a simple model acoustic phonons can be treated like particles of an ideal gas. Then the thermal conductivity λ can be defined using Fourier's law

$$\vec{q} = -\lambda \nabla T \quad (1)$$

with the heat flux \vec{q} and the temperature gradient ∇T . Thermal conductivity is connected with the thermal diffusivity α , specific heat capacity c_p and specific density ρ by

$$\lambda = \alpha c_p \rho \quad (2)$$

In this equation the product $C = c_p \rho$ is the specific heat per volume. Furthermore, the thermal diffusivity can be described with the Einstein approximation [3] by the product of the phonon's velocity v and its mean free path β

$$\alpha = \frac{1}{3} v \beta \quad (3)$$

In general, the speed of an acoustic wave in a medium is approximately given by

$$v \approx \sqrt{\frac{K}{\rho}} \quad (4)$$

and the bulk modulus K can be estimated from Young's modulus E and Poisson's ratio μ from Ref. [4].

$$K = \frac{E}{3(1-2\mu)} \quad (5)$$

In a previous paper the authors used a simple serial model to calculate the Poisson's ratio μ_c of a composite consisting of polypropylene (PP) and iron-silicon (FeSi) particles [5]. This model can be extended to three components, which leads to a Poisson's ratio of the composite which is close to the Poisson's ratio of the polymer. Another possibility is the use of the Hashin–Shtrikman lower bound model which agrees quite well with the Poisson's ratio of two phase composites [6] and which can be used also for three-phase composites using Ref. [7].

$$\mu_c = \mu_1 + \frac{A}{1 - \frac{1}{3\mu_1} A} \quad (6)$$

$$A = \sum_{t=2}^3 \frac{x_t}{\frac{1}{\mu_t - \mu_1} + \frac{1}{3\mu_1}} \quad (7)$$

with Poisson's ratios and volume fractions of diamond (μ_1, x_1), iron (μ_2, x_2) and polypropylene (μ_3, x_3). Using Eqs. (4)–(6) in Eq. (3) the phonon's mean free path can be estimated by

$$\beta = \frac{3\alpha}{\sqrt{3\rho_c \frac{E}{1-2\mu_c}}} \quad (8)$$

whereas ρ_c denotes the density of the composite. Furthermore, Eq. (8) shows a proportionality of $\alpha \sim \sqrt{E}$.

In metallic materials the thermal conductivity λ_m is composed additively of an electronic component λ_e related to thermal transport by electrons and a lattice component due to phonons λ_p

$$\lambda_m = \lambda_e + \lambda_p \quad (9)$$

wherein the electronic component can be described by the Wiedemann-Franz law [3]

$$\lambda_e = L\sigma T \quad (10)$$

$L = \pi^2/3(k_B/e)^2 = 2.44 \cdot 10^{-8} \text{ W}\Omega/\text{K}^2$ (with Boltzmann constant k_B , electrical conductivity σ , and elementary charge e) is the theoretical Lorenz number while experimental values are lying between $2.1 \cdot 10^{-8} \text{ W}\Omega/\text{K}^2 \leq L \leq 2.9 \cdot 10^{-8} \text{ W}\Omega/\text{K}^2$ [8]. With Eqs. (2) and (10) the thermal diffusivity of the electronic component holds

$$\alpha_e = LT \frac{\sigma}{\rho c_p} \quad (11)$$

3. Experimental

As matrix material for the composite the semi-crystalline isotactic polypropylene homopolymer (DOW H 734 52 RNA, Dow Customer Information Group, Edegem, Belgium), with specific density of $\rho_{PP} = 0.9 \text{ g/cm}^3$ was chosen. The temperature dependent Poisson's ratio can be taken from Ref. [9]. As filler material an irregular shaped iron (Fe) powder ASC300 with a mean particle diameter of $d_{50} = 53 \mu\text{m}$ was used (Höganäs AB, Höganäs, Sweden). The iron powder particles are having a specific density of $\rho_{Fe} = 7.87 \text{ g/cm}^3$, Young's modulus of $E_{Fe} = 210 \text{ GPa}$ [10] and a Poisson's ratio of $\mu_{Fe} = 0.27$ [10,11]. Furthermore, two types of diamond particles were used - synthetic (SD) and natural (ND) diamond with grain size of $d = 7 \mu\text{m}$, specific density of $\rho_D = 3.52 \text{ g/cm}^3$. With reference to [12] Young's modulus of natural diamond is around $E_{ND} = 1140 \text{ GPa}$ and for synthetic diamond $750 \text{ GPa} \leq E_{SD} \leq 1165 \text{ GPa}$. Poisson's ratio is given for natural diamond as $\mu_{ND} = 0.07$ and for synthetic diamond $0.07 \leq \mu_{SD} \leq 1.09$ dependent on preparation method and crystal orientation [12] while Gercek [11] reports $0.1 \leq \mu_{ND} \leq 0.29$ and $\mu_{SD} = 0.2$. Thermal conductivity of natural diamond is at room temperature between $\lambda_{ND} = 600 \text{ W/(m}\cdot\text{K)}$ and $\lambda_{ND} = 2000 \text{ W/(m}\cdot\text{K)}$ [12].

Polypropylene and pre-heated (100°C) iron powder as well as diamond microparticles were compounded for 10 min at 475 K and 50 rpm in a co-rotating lab kneader (PolyLab Rheomix 600p, ThermoHaake, Thermo Fisher Scientific, Karlsruhe, Germany). All materials were dosed gravimetrically using a laboratory balance (Aculab Arilon, Sartorius GmbH, Göttingen, Germany). The composites were granulated and further injection molded to rectangular test samples ($30 \times 10 \times 4 \text{ mm}^3$) and circular shaped disks (diameter $d = 25 \text{ mm}$, thickness $t = 1 \text{ mm}$) using a hydraulic injection molding machine (Arburg Allrounder 220 S, Arburg, Lossburg, Germany). Temperature of the injected sample material at the nozzle of the injection unit was 200°C and temperature of the mold was 80°C . Time from injection of the mass to ejection of samples were 12 s. Samples of neat polypropylene were produced as reference.

Heat flow measurements of samples were carried out in the temperature range 225 K – 475 K at heating/cooling rate of 5 K/min under nitrogen atmosphere using a differential scanning calorimeter (DSC Q2000, TA Instruments, Alzenau, Germany). For temperature calibration the melt temperature of indium, lead, tin and zinc standards have been measured to achieve a minimum temperature deviation of 0.2 K. The fraction of crystallization was estimated using a standard melting enthalpy of 207 J/g for 100% crystalline PP [13].

The thermal diffusivity was investigated in a laser flash apparatus (LFA 427, Netzsch-Gerätebau GmbH, Germany) from 225 K to 425 K under helium atmosphere. Samples were covered by a thin graphite layer to ensure a good absorption of the laser light and to

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