



Preparation and photothermal characterization of nanocomposites based on high density polyethylene filled with expanded and unexpanded graphite: Particle size and shape effects

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ABSTRACT

This work aimed at thermal transport characterization of high density polyethylene (HDPE) filled with 5 and 50 μm expanded graphite (EG) particles and with 0.4 μm unexpanded graphite (UG) particles. Sample platelets were produced by melt mixing followed by compression molding. Thermal conductivity k was determined by combining measurements of density, specific heat capacity and thermal diffusivity, the latter by modulated photothermal radiometry (PTR). Starting from an effective medium approximation model, we derived a linearized expression for the effective k of composites with low particle charge. It explains the unusually high experimental k values (up to four-fold increase) as the effect of strongly non-spherical EG particles (aspect ratio $1/p = 110\text{--}290$). Larger particle sizes produce higher k enhancement, while the interfacial thermal resistance ($R_{bd} = 2.1 \cdot 10^{-7} \text{ m}^2 \cdot \text{K/W}$) has an opposite effect. The same model is consistent with experimental k for low particle charge HDPE/UG composites. At higher particle charge the model fails due to particle interaction leading to validity break of the effective medium approximation.

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1. Introduction

Because of their potential applications in advanced technologies, such as in light emitting devices, batteries, electromagnetic shielding, anti-static, corrosion resistant coatings, and other functional applications, conducting nanocomposites with polymeric matrices have attracted considerable attention. The introduction of electrically and thermally conductive fillers such as graphite, carbon black, metal and metal oxide powders into the polymeric matrix in molten state is a promising approach to fabricate electrically and thermally conductive polymeric materials [1,2]. Recently, polymer based nanocomposites reinforced with expanded graphite (EG) [3,4] have shown notable improvements in mechanical, electrical conductivity, thermal conductivity and other thermophysical properties over those containing traditional unexpanded graphite (UG). The reason for this improvement is the sheet-like structure of natural graphite where the atoms are strongly bonded on a hexagonal plane but weakly bonded normal to that plane. If these sheets layers could be separated down to

a nanometer thickness, they would form high aspect ratio (200–1500) graphene nanosheets having an enormous surface area (up to 2630 m^2/g considering that both sides of the sheets are accessible). EG flakes are obtained from UG particles by chemical process and consist of stacks of hundreds of graphene nanosheets. Due to their special properties, the dispersion homogeneity of such nanoparticles in a matrix plays a key role in the improvement of thermophysical properties of the resultant nanocomposite. In this study we report on the preparation and thermophysical characterization of HDPE/EG and HDPE/UG composites. Thermal conductivity data fitting with an effective medium model elucidates the role played by the conductivity contrast, the particle size and aspect ratio, and by the interfacial thermal resistance in determining high effective conductivity enhancement of the composites.

2. Materials and preparation of composites

Two high density polyethylene (HDPE) brands were used for the preparation of the nanocomposites. BP 5740 3 VA supplied by British Petroleum UK, was used as matrix material with two types of expanded graphite fillers having nominal particle sizes of 50 μm (EG50) and 5 μm (EG5) in diameter (Fig. 1a, b) [5]. The respective

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Nomenclature

C	ρC_p [J/(m ³ ·K)], volumetric heat capacity
e	$(kC)^{1/2}$ [W·s ^{1/2} /(m ² ·K)], thermal effusivity
α	k/C [m ² /s], thermal diffusivity
ϕ_{vol}, ϕ_{wt} [-]	particle volumetric fraction, particle mass fraction
δ [m]	sample thickness
μ	$[\alpha/(\pi f)]^{1/2}$ [m], thermal diffusion length
τ	$(1 + i)/(\kappa\mu)$ [-], thermo-optical thickness
z [m ² ·K/W]	specific thermal impedance
R_{bd} [m ² ·K/W]	interfacial thermal boundary resistance
a_k	$R_{bd}k_m$ [m], Kapitza radius
a_i [m]	ellipsoid radii
p	a_3/a_1 [-], particle aspect ratio

trade name is Ecophit G (GFG50 and GFG5), supplied by SGL Technologies GmbH. For the preparation of nanocomposites with UG, HDPE- I-668 supplied by Petkim A.Ş.–Izmir, Turkey, was used as matrix material. The unexpanded graphite particles with nominal size of 0.40 μm (UG04) were supplied as type D50 by Nanoamor Inc. The SEM image of Fig. 1c shows that the UG04 particles are also not spherical. The nominal size of 0.40 μm corresponds rather to the thickness of the platelet-like particles, while their diameter is on the order of 2.5 μm .

In both cases HDPE/graphite mixtures were prepared by using different weight percentages of graphite loadings. The composites were prepared in the 30 ml mixing chamber of a Brabender Plastocorder PLE 331 apparatus and were mixed at 180 °C for a total mixing time of 15 min. The rotors turned at 35 rpm in a counter-rotating fashion with a speed ratio of 1.1. After 15 min, the mixing chamber of the Brabender apparatus was opened and the resulting mixture was taken out. Finally, after passing through the rollers the mixture was solidified. In the next step, samples in the shape of sheets with a thickness between 0.25 and 0.45 mm and with a surface of few cm²

were produced by compression molding at 180 °C under 40 kPa pressure for 4 min. Upon mixing, the expanded graphite intercalates with the molten matrix and exfoliates into tens of nanometers-thick flakes due to its sheet-like structure and to weak bonds normal to the sheet plane. The observation of low concentration HDPE/EG50 samples under optical microscope suggest that part of the large particle flakes (Fig. 1d) were broken during mixing and formed smaller fragments, comparable to EG5 flakes. In the HDPE/EG5 samples (Fig. 1e) and HDPE/UG04 samples (Fig. 1f) the particle size is homogeneous and corresponds to the nominal value.

3. Volumetric heat capacity determination

3.1. Density measurement

Density ρ was determined by weighing the samples in air and in water with a Mettler H15 balance having a resolution of 10⁻⁴ g. The difference represents the Archimedes's force and allowed determining the volume of the samples. The mixture law for ρ of the composite writes:

$$\rho = \phi_{vol}\rho_f + (1 - \phi_{vol})\rho_m = (\rho_f - \rho_m)\phi_{vol} + \rho_m \quad (1)$$

where ρ_f and ρ_m refer to filler and matrix, respectively. The relation between ϕ_{vol} (unknown) and ϕ_{wt} (known from the sample preparation procedure) involves in turn the knowledge of the densities of the pure components:

$$\phi_{vol} = \left[1 + (\phi_{wt}^{-1} - 1) \left(\rho_f / \rho_m \right) \right]^{-1} \quad (2)$$

In order to fit the measured densities with the linear Eq. (1), one needs to transform ϕ_{wt} into ϕ_{vol} using Eq. (2), but this requires the knowledge of the densities of pure components. ρ_f can be determined in turn by the extrapolation of Eq. (1) to $\phi_{vol} = 1$. The whole procedure implies an iterative process which is convergent. Starting from the literature value of graphite for ρ_f , we obtained after nine iterations the following fit equations (Fig. 2a):

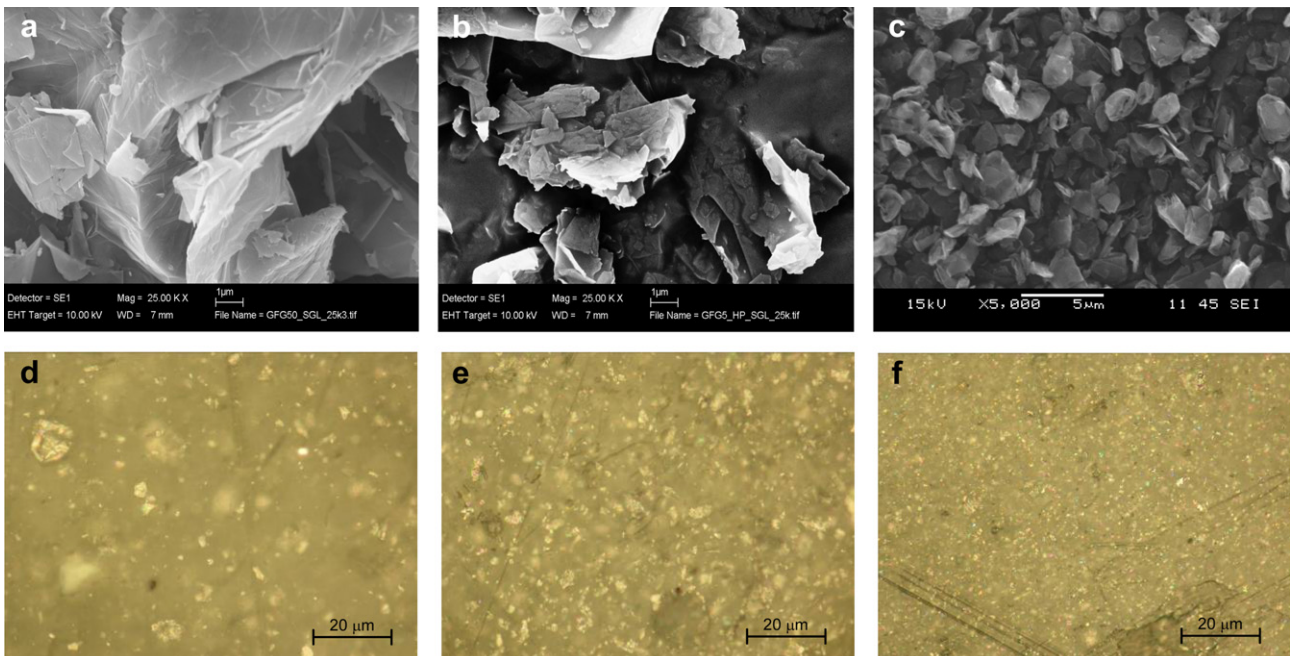


Fig. 1. SEM images of (a) EG50, (b) EG5 and (c) UG04 expanded and unexpanded graphite particles before mixing with the HDPE; optical microscopy images obtained in reflected light, for HDPE nanocomposites containing 2% (wt.) of (d) EG50, (e) EG5 and (f) UG04 particles.

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