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A Study on Thermal and Electrical Conductivities of Ethylene-Butene Copolymer Composites with Carbon Fibers

Ethylene-butene copolymer (EBC)/carbon-fiber (CF) composites can be utilized as an electromechanical material due to their ability to change electric resistance with mechanical strain. The electro-mechanical properties and thermal conductivity of ethylene butene copolymer (EBC) composites with carbon fibers were studied. Carbon fibers were introduced to EBC with various concentrations (5 to 25 wt%). The results showed that carbon fibers' addition to EBC improves the electric conductivity up to 10 times. Increasing the load up to 2.9 MPa will raise the electric resistance change by 4500% for a 25% fiber sample. It is also noted that the EBC/CF composites' electric resistance underwent a dramatic increase in raising the strain. For example, the resistance change was around 13 times higher at 15% strain compared to 5% strain. The thermal conductivity tests showed that the addition of carbon fibers increases the thermal conductivity by 40%, from 0.19 to 0.27 Wm⁻¹K⁻¹.

1 Introduction

Polymer composites containing carbon fibers are widely used in industry due to their excellent thermal (Imiela et al., 2016; Liang et al., 2015), electrical (Bautista-Quijano et al., 2016; Wang et al., 2006), and mechanical properties (Hamid et al., 2020; Shen et al., 2017). They can be used as a flexible electric sensor because of the high sensitivity to strain and low industrial costs (Akhtar et al., 2020; Ferreira et al., 2016). The piezoresistive sensors, which convert the sensor's pressure to resistance signal, have been widely used due to their attractive advantages, such as ease of fabrication, low cost, and accessible signal collection (Patole et al., 2019). Many researchers indicated that the polymer/carbon-fiber composite's electrical conductivity could be influenced by its deformation (Ibrahim et al., 2013; Slobodian et al., 2016a; Slobodian et al., 2013; Slobodian et al., 2012). This change favorably responds to the size and direction of the carbon fiber. Most sensors based on pure cracking mechanisms exhibit a limited detection range

due to the easy breakage of conductive pathways, inhibiting their application in large deformation detections (Liang et al., 2012). Carbon black has an advantage over other carbon based fillers like graphene and carbon nanotubes. It is cheaper and more accessible; however, carbon black-composites have a gauge factor (G.F.) of lower than 100 in strain sensing (Xiao et al., 2010). Some researchers studied the influence of deformation on electric resistance for carbon nanotube (CNT)/polyurethane (PUR) and found increased resistance and Gauge factor. Gauge factor defines the sensitivity of strain as a relative resistance change divided by the applied strain to explain the effect of applied preload on the increase of relative resistance change of CNT/PUR composites when introducing the strain (Slobodian et al., 2016a). Previous works show that the electrically conductive composite can be used in real-time like electric skins, entertainment systems, human health monitoring for Parkinson's disease patients due to the high response of electrical resistance to strain (Liao et al., 2017). Certain researchers reported a change of resistance of multiwalled carbon nanotube film that could be used as an electric sensor as it favors response to strain (Li et al., 2008). Studies have indicated that the addition of fiber to a polymer matrix could influence the composites' mechanical properties and thermal conductivity (Hamid et al., 2013; Ren et al., 2018; Svoboda et al., 2012; Theravalappil et al., 2014; Zhou et al., 2016). It was reported (Svoboda et al., 2012) that ethylene-octene copolymer/graphite composite's thermal conductivity increased by 245%, where the filler content was increased from 0 to 50 wt% due to the uniform distribution of the graphite fibers. Some researchers reported that carbon black's (C.B.s) addition to polyvinyl alcohol could increase the gauge factor up to 100 due to the high potential capability of these novel nanocomposite films with carbon black (Huang et al., 2020). It is considered that the porous structure of the C.B.s provides a high sensitivity of flexible sensing elements that can be implemented into microfabrication processes easily.

There are only a few papers that consider the electrical and thermal conductivity of polymer nanocomposites together. Most polymer materials possess an undesired thermal conductivity lower than 0.2 W m⁻¹K⁻¹ (Hu et al., 2018). Typically, adding fillers to polymer materials leads to achievement of highly thermally conductive polymer composites (Kim et al., 2015; Mehra et al., 2018). However, the intrinsic phonon spectrum mismatch between polymer material and fillers heavily

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deteriorates phonon transferring efficiency (Mehra et al., 2018). Krause et al. reported that carbon fiber's addition to polypropylene and carbon nanotube would slightly increase the thermal resistance. It was also reported that graphite nanotubes would increase thermal conductivity two times compared to pure polypropylene (Krause et al., 2019). This research investigates the effect of carbon fiber (CF) loading on electromechanical properties of the ethylene-butene copolymer (EBC) under the elongation/relaxation cycle with a variety of CF contents. Moreover, the influence of carbon-fiber material on thermal conductivity of EBC was investigated.

2 Experimental

2.1 Materials

Ethylene-butene copolymer with a density of 0.862 g/cm^3 , ultimate tensile strength 2.0 MPa, melt index 1.2 dg/min, tensile elongation 600% was purchased from DOW Chemical Company, Midland, Michigan, USA. The Carbon fiber was obtained from Torayca T700SC 12000-50C provided by Toray Carbon Fibers America Inc., Decatur, Alabama, USA. The tensile strength and modulus are 4900 MPa and 230 GPa, respectively. The carbon fiber has a $7 \mu\text{m}$ thickness with a 1.8 g/cm^3 density and a 2.1% strain (trade name : Tor, thermal conductivity is $28.13 \text{ Wm}^{-1}\text{K}^{-1}$, electric resistivity is $1.6 \times 10^{-3} \Omega \cdot \text{cm}$ as indicated by Toray Carbon Fibers America Inc).

2.2 Characterization

EBC/CF composites were prepared using a two-roll mill for 5 min at 150°C . Then, compression molding was used to prepare the sheets with a thickness of 1 mm at 10 MPa with 5 min preheating and 6 min pressing at 150°C . Finally, the dumbbell-shaped specimens were prepared with a compression cutter for the test. During strain-relaxation cycles, the electrical resistance change was analyzed with a UT71C multimeter (UNI-Trend Technology China Co Ltd., Dongguan, PRC) using a two-point technique.

For electrical characterization, several methods are used. The two-point probe method using a digital multimeter and four-point probe method using Hall effect measurements is more common. However, due to four-probe measurement limitations during the stretching, the EBC/CB's electrical resistance change in strain-relaxation cycles was analyzed using the two-point probe technique. Two single-terminal electrodes are attached to the surface of the conductive structure, this being called the two-point probe technique. A DC source current is then connected through the two electrodes, and the subsequent voltage over the same electrodes is estimated. The electrical resistance between these two electrodes is then determined, according to Ohm's law. To increase the results' reliability, copper plates used as electrodes that were attached to the sample, dissolved the backing adhesive, flooded into butanone solution, and then washed using tap water. After drying and cleaning the copper plates, the sheet was sandwiched between upper and lower Ag's electrodes using silver paint around the surface of dumbbell specimens. The tests were done using various stresses (0.442, 0.884, 1.325, 1.768, and 2.219 MPa) with the strain and electric resistance change with

time. When the samples were ready, an electrical circuit powered by a DC power source was applied to the DC source for the tests. However, it is worth to mention that in reality, 1 kHz is commonly used to prevent polarization inaccuracy (Hamid and Svoboda, 2020).

Tensile deformation and gauge factors, the ratio of relative change in electrical resistance R to the mechanical strain ε of EBC/carbon-fiber composites, were determined by a two-point technique electric resistance as a function of the strain. In this research, the change in electrical resistance was defined as (Svoboda et al., 2012)

$$\Delta R = \frac{R - R_0}{R_0}, \quad (1)$$

where R_0 is the initial electrical resistance of the sample before the first elongation and R is the resistance during elongation. The elongation is defined as:

$$\varepsilon = \frac{L - L_0}{L_0}, \quad (2)$$

L_0 represents the EBC/CF specimen's initial length, while L is the stretching experiment's length.

The Fitch (1935) method was used to determine the thermal conductivity of the EBC/CF composites (Hamid and Svoboda, 2020; Rahman 1991), where the sample is sandwiched between a heat source insulated on all faces but one and a constant temperature brass cylinder as heat sink. A schematic representation of the instrument used for thermal conductivity measurements is shown in Fig 1.

The process of measurement and the instrument are explained below. At first, the 5 cm diameter central brass cylinder (CBC) was heated to $t_2 = 45^\circ\text{C}$ with the assistance of another chamber which was connected with an indoor water regulator by elastic hoses with a water thermostat with 0.1°C accuracy; it took about 3 min to reach a suitable temperature. Then the hot chamber was quickly removed, and the sample with a 5 cm diameter was placed on top of the CBC with the second brass cylinder on top of it with a weight of 100 g, which was connected to a second water thermostat with the temperature set to 25°C . The heat was transferred from the hot CBC to the cold cylinder by passing through the sample. The temperature of the CBC is decreasing rapidly. The CBC data were collected every 5 s for 30 min by a thermocouple (type copper-constantan) that was connected with a National Instruments data acquisition equipment (NI USB-9211A, Portable USB-Based DAQ (Data Acquisition), National Instruments,

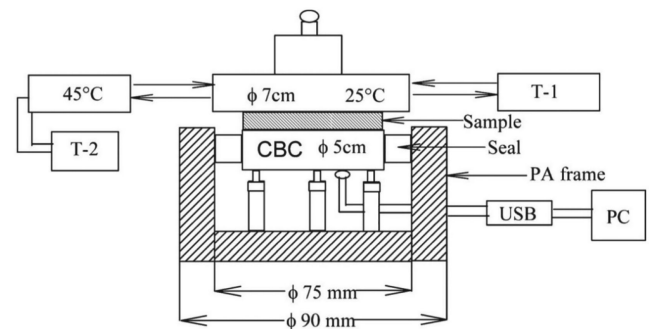


Fig. 1. Schematics of the device utilized for the measurement of thermal conductivity

Austin, USA) for thermocouple software and using the computer through the USB port (Slobodian et al., 2016b). The data was analyzed using LabVIEW Signal Express 2.5 (National Instruments, Austin, USA).

The (EBC)/(CF) films were melt pressed (at 200 °C) with a thickness of 0.5 mm on the surface of the microscope hot stage LINKAM. A polarized optical microscope (model RX41, Olympus, Tokyo, Japan) was used to observe the composite structure.

The dependence of temperature on time is described with the following equation (Svoboda et al., 2012):

$$-K \frac{dt}{d\tau} = \frac{S\lambda(t - t_1)}{\delta} + B(t - t_1), \quad (3)$$

where temperature t (at time $\tau = 0$) = $t_2 = 45^\circ\text{C}$, K = the central brass cylinder heat capacity which is 317.5 J K^{-1} , S = sample area (m^2), λ = thermal conductivity ($\text{W m}^{-2} \text{ K}^{-1}$), t = acquired temperature of the central brass cylinder ($^\circ\text{C}$), t_1 = hollow brass cylinder temperature $25 \text{ } (^\circ\text{C})$, t_2 = initial temperature of CBC (45°C), δ = specimen thickness (m), B = coefficient accounting for a heat loss ($\text{J s}^{-1} \text{ K}^{-1}$), τ = time (s). B is calculated according to $B = \alpha S_z$,

where α = heat transfer coefficient ($\text{W m}^{-2} \text{ K}^{-1}$), S_z = heat loss area (m^2). By solving Eq. 3:

$$t = t_1 - (t_1 - t_2) * e^{-(A_1 + A_2)\tau}, \quad (5)$$

where:

$$A_1 = \frac{S\lambda}{\delta K}, \quad (6)$$

$$A_2 = \frac{B}{K}. \quad (7)$$

However, Eq. 5 can be simplified with exponential growth with three parameters as:

$$y = y_0 + ae^{(-bx)}. \quad (8)$$

The coefficient b is obtained from the nonlinear regression of the temperature versus time plot:

$$A_1 = b - A_2, \quad (9)$$

where A_2 is the heat loss of the instrument obtained by control experiment. Then the thermal conductivity λ is calculated by Eq. 10:

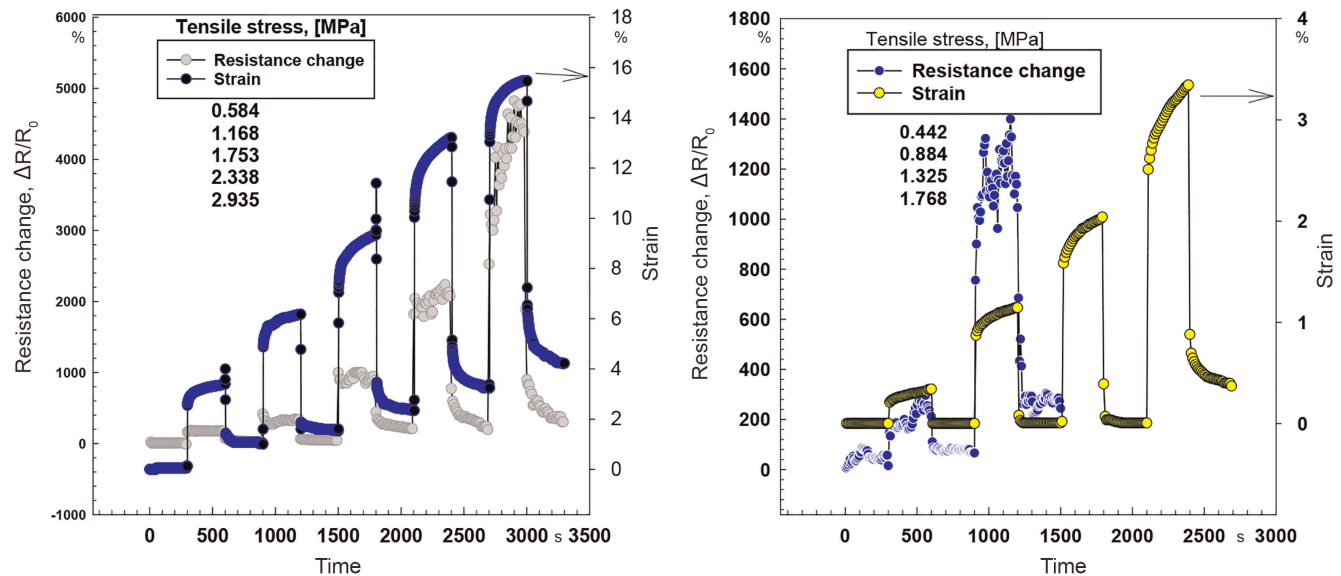
$$\lambda = \frac{A_1 \delta K}{S}. \quad (10)$$

3 Results and Discussion

3.1 Electrical Properties

Tensile deformation and gauge factors were determined by a two-point technique to measure the electric resistance as a function of the strain. The tensile stress deformed the composite samples with the various carbon fiber contents (5, 10, 15, 20, and 25 wt%) with increasing stress values (0.442, 0.884, 1.325, and 1.768, and 2.219 MPa), respectively. The strain caused by tensile stress and the change of electrical resistance were measured as a function of time. The change of electric resistance of EBC with 5% and 10% of carbon fibers could not be measured because the values of resistivity were below the multimeter's sensitivity level. It was also observed that the EBC composite with 15% carbon fiber had a low change of electrical resistance with a change of strain. The results suggest that higher concentrations of carbon fibre (more than 15%) are beneficial for the strain induced resistivity change.

Furthermore, the EBC/CF resistance change with 25 wt% CF increased with increasing deformation due to increasing number of connections between carbon fibers (Fig. 2) (Slobodian et al., 2016a; 2013; 2016b). The number of fibers in EBC/CF composite containing 15 wt% was much lower, i.e., the 25 wt% sample and the resistance could only be measured up to 0.884 MPa. Figure 2 shows that five cycles of increasing



A) B) Fig. 2. Relative resistance change, $\Delta R/R_0$, and the strain, ϵ , of A) EBC/CF 75/25 wt%, B) EBC/CF 85/15 wt%

tensile strain were possible up to 2.935 MPa for the 25 wt% CF and only four cycles composites up to 1.768 MPa for the 15 wt% of CF. The grey circles in Fig. 2 represent the relevant resistance changes. In addition, the dark grey circles illustrate the change in strain values. Figure 2A shows that EBC/CF 25 wt% is conductive also at the peak strain of about 16% once the resistance change is about 4500%.

In Fig. 3 the micrographs of optical microscopy from EBC/carbon fiber for 15 wt% and 25 wt% are shown. In case of EBC with 5 wt% and 10 wt% of CF the concentration of CF was not sufficient to form a conducting path.

The optical microscopy examination indicated that the fibers were homogeneously distributed in the EBC composites. It was also observed that at low fiber content, 15 wt%, fewer fibers were lying on the surface than for 25 wt% carbon fiber. The composite with 25 wt% carbon fiber had higher fiber agglomerates because of low dispersion caused by excess filler self-association (Sanchez-Garcia et al., 2008). These results are in agreement with other researchers (Slobodian et al., 2011). The figure also shows that the fibers preserved relatively high length which is essential for the conductive path formation. This was caused by gentle mixing in two-roll mill. We have tried to do the mixing also in twin-screw extruder and in Brabender but the fibers were in these cases broken to much smaller pieces and the strain induced resistance change effect could not be achieved.

Figure 4 illustrates the gauge factor as a function of strain for EBC/CF 25 wt%. The gauge factor was approximately 50 for the small strain values and then increased to 300 at the strain of 16%. This is a remarkable growth that keeps an EBC/CF mixture with 25 wt% within the variety of mixtures of substances demonstrating excellent sensitivity to tensile deformation.

3.2 Thermal Conductivity

The calculation of the thermal conductivities of EBC/CF is shown in Table 1 and Fig. 5. It is indicated that by the addition of carbon fiber to ethylene butene copolymer, the heat transfer rate is increased in comparison with pure EBC because the intrinsic phonon spectrum mismatch between polymer material and fillers heavily deteriorates the efficiency of phonon transfer. The Thermal conductivity of the EBC/CF composites increased with increasing CF (Fig. 6). The highest measured thermal conductivity was $0.263 \text{ W m}^{-1} \text{ K}^{-1}$ for an EBC/CF 20 wt%, an increase of 35% compared with pure EBC. The uniform distribution of CF had a large effect on

the growth in thermal and electric conductivities. Our results correspond well with other researchers (Sonawane et al., 2020). CF has high conductivity for both heat and electricity. Increased CF loading in EBC increases the mixture's thermal and electric conductivities (George et al., 2009; Imiela et al., 2016; Liang et al., 2015).

4 Conclusions

EBC/CF composites were prepared by mixing the EBC with various carbon fiber levels using a two-roll mill. The observations from optical microscopy indicated a relatively good dispersion of carbon fibers in the matrix and the length of the fibers was preserved. The electromechanical testing showed that the composite's straining led to a change in its macroscopic electrical resistance. The EBC/CF composites had high degree of sensitivity of electric resistivity to strain, and the changes were reversible. Therefore, the results indicate the composite's good potential to be used as an electrical strain sensor. We found out that the mixtures exhibit an increase in thermal conductivity with loading carbon fiber due to the high

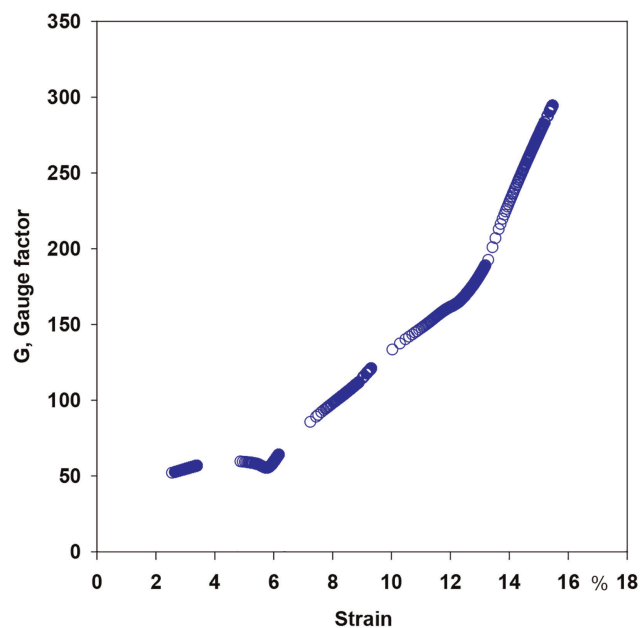


Fig. 4. Strain dependence of gauge factor GF EBC/CF 25 wt%

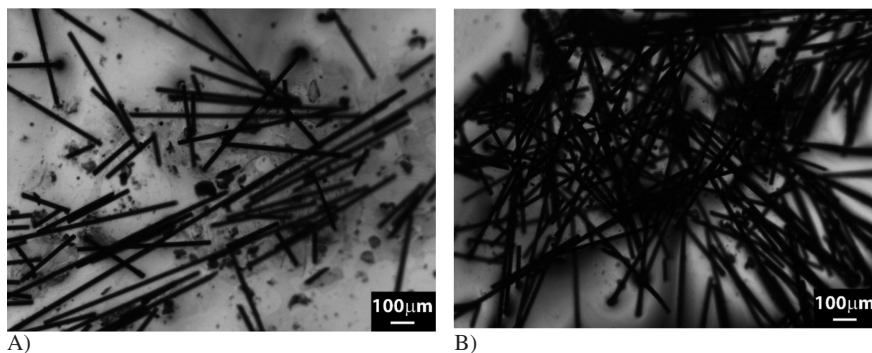


Fig. 3. Micrographs from optical microscopy, A) ethylene-butene copolymer (EBC) and carbon fiber (CF) with 15 wt%, B) ethylene-butene copolymer (EBC) and carbon fiber (CF) 25 wt%

CF %	b	A_1	λ $W\ m^{-2}\ K^{-1}$	R^2
0	0.0015611	0.0012981	0.1952	0.999976
5	0.0019353	0.0016723	0.2266	0.999961
10	0.0020541	0.00179114	0.2413	0.999968
15	0.0021213	0.00185826	0.2477	0.999968
20	0.0023160	0.002053	0.2635	0.999979

Table 1. The thermal conductivity parameters of EBC/CF

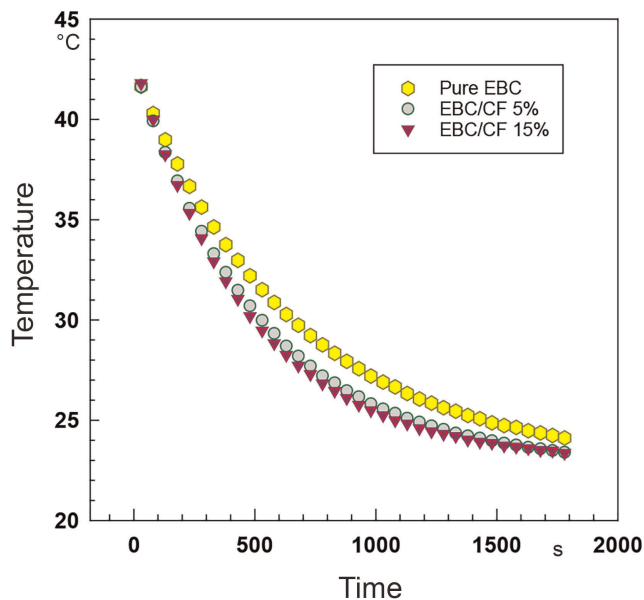


Fig. 5. Thermal conductivity measurement: temperature vs. time for EBC/CF for different concentrations of carbon fiber with the initial temperature of 45 °C

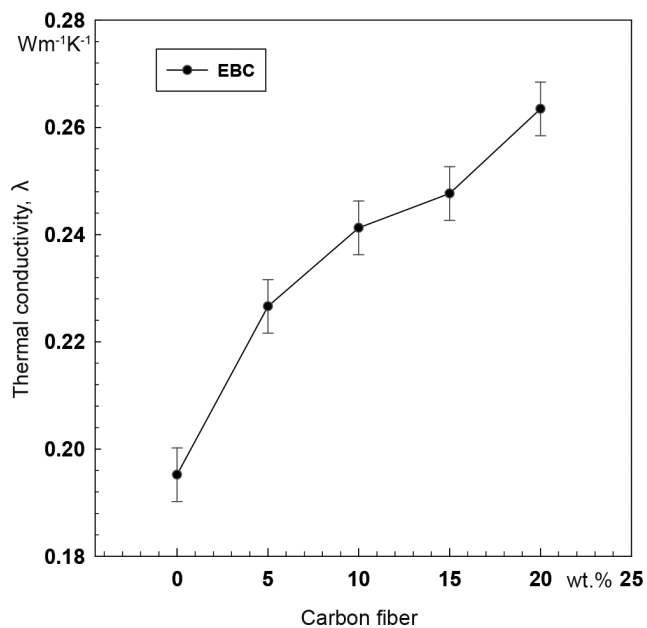


Fig. 6. Thermal conductivity as a function of the carbon fiber content

intrinsic phonon spectrum mismatch between polymer material and fillers, leading to increased heat transfer.

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