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Poly(butylene terephthalate) Nanocomposites Containing Carbon Nanotube

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1. Introduction

Carbon nanotube (CNT) has attracted of great interest as advanced nanoreinforcements in new kinds of polymer nanocomposites because of the combination of its unique extraordinary properties with high aspect ratio and small size (Ebbesen, 1997; Dresselhaus et al., 2001; Schadler et al., 1998; Ajayan, 1999; Bokobza, 2007; Paul & Robesson, 2008). In particular, excellent mechanical strength, thermal conductivity, and electrical properties of CNT have created a high level of activity in materials research and development for potential applications such as fuel cell, hydrogen storage, field emission display, chemical or biological sensor, and advanced polymer nanocomposites (Iijima, 1991; De Heer et al., 1995; Wong et al, 1997; Fan et al., 1999; Kim & Lieber, 1999; Liu et al., 1999; Kong et al., 2000; Ishihara et al., 2001; Alan et al., 2003; Wu & Shaw, 2005). This feature has motivated a number of attempts to fabricate CNT/polymer nanocomposites in the development of high-performance composite materials (Kim et al., 2006; Kim et al., 2007; Kim et al., 2008; Kim, 2009; Kim et al., 2009; Kim et al., 2010). In this regard, much research and development have been performed to date for achieving the practical realization of excellent properties of CNT for advanced polymer nanocomposites in a broad range of industrial applications. However, because of high cost and limited availability, only a few practical applications in industrial fields such as electronic and electric appliances have been realized to date.

The CNT consisting of concentric cylinder of graphite layers is a new form of carbon and can be classified into three types (Dresselhaus et al., 2001; Iijima, 1991; Shonaike & Advani, 2003): single-walled CNT (SWCNT), double-walled CNT (DWCNT), and multi-walled CNT (MWCNT). SWCNT consists of a single layer of carbon atoms through the thickness of the cylindrical wall with the diameters of 1.0-1.4 nm, two such concentric cylinders forms DWCNT, and MWCNT consists of several layers of coaxial carbon tubes, the diameters of which range from 10 to 50 nm with the length of more than 10 μm (Dresselhaus et al., 2001; Iijima, 1991; Shonaike & Advani, 2003). The graphite nature of the nanotube lattice results in a fiber with high strength, stiffness, and conductivity, and higher aspect ratio represented by very small diameter and long length makes it possible for CNTs to be ideal nanoreinforcing fillers in advanced polymer nanocomposites (Thostenson et al., 2001). Both theoretical and experimental approaches suggest the exceptional mechanical properties of CNTs ~100 times higher than the strongest steel at a fraction of the weight (Goze et al., 1999; Yao et al., 2001;

Yu et al., 2000a; Yu et al., 2000b): The Young's modulus, strength, and toughness of SWCNT shows 0.32~1.47 TPa of Young's modulus, 10-52 GPa of strength, and ~770 J/g of toughness, respectively (Yu et al., 2000a). For MWCNT, the values of strength, Young's modulus, and toughness were found to be 11-63 GPa, 0.27-0.95 TPa, and ~1240 J/g, respectively (Yu et al., 2000b). In addition, CNTs exhibit excellent electrical properties and electric current carrying capacity ~1000 times higher than copper wires (Frank et al., 1998). In general, MWCNTs show inferior mechanical performance as compared to SWCNTs. However, MWCNTs have a cost advantage, in that they can be produced in much larger quantities at lower cost compared with the SWNT. In addition, MWCNTs are usually individual, longer, and more rigid than SWCNTs. Because of their remarkable physical properties such as high aspect ratio and excellent mechanical strength, MWCNTs are regarded as prospective reinforcing fillers in high performance polymer nanocomposites. For these reasons, extensive research and development have been directed towards the potential applications of CNTs for novel composite materials in a wide range of industrial fields. The fundamental research progressed to date on applications of CNTs also suggests that CNTs can be utilized as promising reinforcements in new kinds of polymer nanocomposites with remarkable physical/chemical characteristics (Shonaike & Advani, 2003).

Poly(butylene terephthalate) (PBT) is a semi-crystalline polymer with good mechanical properties and excellent processability, and it has been widely used as structural materials in the automotive, electrical, and electronic industries (Fakirov, 1993; Rubin, 1990; Tjong & Meng, 1999). Recently, there are continuing practical demands for realizing higher performance of PBT with various processing conditions, thus making it possible to be utilized in various advanced industries. For this reason, much research has been performed to date to extend and develop commercial applications of PBT for high performance polymer nanocomposite using reinforcing fillers as well as to displace PBT (Kim et al., 2005; Kim et al., 2006c; Wu et al., 2007; Kim, 2009b). Although promising, however, insufficient mechanical properties and thermal stability of PBT composites have often hindered its potential application in a broad range of industrial fields. From both an economic and industrial perspective, the major challenges for high performance polymer nanocomposites are to fabricate the polymer nanocomposites with low costs and to facilitate large scale-up for commercial applications.

During the rapid advancement in the materials science and technology, much research has extensively undertaken on high-performance polymer composites for targeted applications in numerous industrial fields. Furthermore, a great number of efforts have been made to develop high-performance polymer nanocomposites with the benefit of nanotechnology (Ajayan, 1999; Bokobza, 2007; Paul & Robesson, 2008). These attempts include studies of the polymer composites with the introduction of nanoreinforcing fillers into the polymer matrix (Ajayan, 1999; Bokobza, 2007; Paul & Robesson, 2008; Kim et al., 2006; Kim et al., 2007; Kim et al., 2008; Kim, 2009; Kim et al., 2009; Kim et al., 2010). Polymer nanocomposites, which is a new class of polymeric materials based on the reinforcement of polymers using nanofillers, have attracted a great deal of interest in fields ranging from basic science to the industrial applications because it is possible to remarkably improve the physical properties of composite materials at lower filler loading (Paul & Robesson, 2008). Therefore, the fabrication of the polymer nanocomposites reinforced with various nanofillers is believed to a key technology on advanced composites for next generation.

Currently, four processing techniques are in common use to incorporate CNT into the polymer matrix for fabricating CNT/polymer nanocomposites (Kim et al., 2006; Kim et al., 2007; Kim et al., 2008; Kim, 2009; Kim et al., 2009; Kim et al., 2010; Haggenmuller et al., 2000; Pötschke et al.,

2002; Jung et al., 2008; Mu et al., 2008; Pegel et al., 2008; Moniruzzaman & Winey, 2006): direct mixing, solution method, in situ polymerization, and melt blending. Of these processing techniques, a melt blending has been accepted as the simplest and the most effective method, particularly from a commercial perspective, because this process makes it possible to fabricate high-performance polymer nanocomposites at low process cost and facilitates commercial scale-up (Kim et al., 2006; Kim et al., 2007; Kim et al., 2008; Kim, 2009; Kim et al., 2009; Kim et al., 2010). Furthermore, the combination of a very small quantity of expensive CNT with conventional cheap thermoplastic polymers provides attractive possibility for enhancing the physical properties of polymer nanocomposites using a simple and cost-effective method.

This chapter focuses on the fabrication and characterization of CNT-reinforced PBT nanocomposites. The PBT nanocomposites were characterized by means of advanced rheometric expansion system (ARES) rheometer, dynamic mechanical thermal analysis (DMTA), transmission electron microscopy (TEM), scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA) to clarify the effects of CNT on the physical properties of PBT nanocomposites. It is expected that this chapter will help in the preliminary understanding of the fabrication and enhanced properties of PBT nanocomposites reinforced with a very small quantity of CNT. This chapter also suggests a simple and cost-effective method that will facilitate the industrial realization of CNT-reinforced PBT nanocomposites with enhanced physical properties.

2. Fabrication of PBT nanocomposites containing CNT

2.1 General features

PBT nanocomposites containing CNT were prepared by simple melt blending in a twin-screw extruder to create high performance polymer nanocomposites at low manufacturing cost for practically possible application in a broad range of industry. There is significant dependence of thermal, rheological, and mechanical properties of PBT nanocomposites on the content and dispersion state of CNT. Storage modulus and loss modulus of PBT nanocomposites increased with increasing frequency, and this enhancing effect was more pronounced at low-frequency region. Non-terminal behavior of PBT nanocomposites was attributed to nanotube–nanotube or polymer–nanotube interactions, and the dominant nanotube–nanotube interactions at high CNT content resulted in the formation of the interconnected or network-like structures of CNT in PBT nanocomposites. A very small quantity of CNT substantially improves the mechanical properties and heat distortion temperature of PBT nanocomposites. Unique character of CNT dispersed in PBT matrix resulted in the physical barrier effect against the thermal decomposition, leading to the enhanced thermal stability of PBT nanocomposites. The key to improve the overall properties of PBT nanocomposites depend on the optimization of the unique geometry and dispersion state of CNT in PBT nanocomposites during melt processing. This chapter also suggests a simple and cost-effective method that will facilitate the industrial realization of CNT-reinforced PBT nanocomposites with enhanced physical properties.

2.2 CNT-reinforced PBT nanocomposites

PBT was used as the thermoplastic polymer with an intrinsic viscosity of 1.1 dl/g and a melt-flow index of 20 g/min, supplied by Samyang Corp., Korea. The nanotubes used were multiwalled CNT (degree of purity > 95%) synthesized by a thermal chemical vapor deposition process, purchased from Iljin Nanotech, Korea. According to the supplier, their

length and diameter were 10–30 nm and 10–50 μm, respectively, indicating that their aspect ratio reaches 1000. All materials were dried at 120ºC *in vacuo* for at least 24 h before use to minimize the effects of moisture. PBT nanocomposites were prepared by a melt blending process in a Haake Rheometer (Haake Technik GmbH, Germany) equipped with the intermeshing co-rotating type of a twin-screw. The temperatures of the heating zone, from the hopper to the die, were set to 250, 260, 265, and 255ºC, and the screw speed was fixed at 45 rpm. Prior to melt blending, PBT and CNT were physically premixed before being fed in the hopper of a twin-screw extruder to achieve better dispersion of CNT with PBT. For the fabrication of PBT nanocomposites, PBT was melt-blended with the addition of CNT content, specified as 0.5, 1.0, and 2.0 wt % in the polymer matrix. Upon completion of melt blending, the extruded strands were cooled in the water-bath and then cut into pellets using a rate-controlled pelletizer.

3. Effect of CNT on properties of PBT nanocomposites

3.1 Rheological properties

The complex viscosity ($|\eta^*|$) of PBT nanocomposites as a function of frequency is shown in Figure 1. The $|\eta^*|$ values of PBT nanocomposites decreased with increasing frequency, indicating a non-Newtonian behavior over the whole frequency range measured. The shear thinning behavior of PBT nanocomposites was attributed to random orientation and entangled molecular chains in the polymer nanocomposites during the applied shear force. The effect of CNT on the $|\eta^*|$ value of PBT nanocomposites was more significant at low-frequency region than at high-frequency region, and this effect decreased with increasing frequency because of strong shear thinning behavior of PBT nanocomposites induced by CNT. As shown in Figure 1B, PBT nanocomposites exhibited higher $|\eta^*|$, particularly at low frequency as compared to pure PBT, indicating the formation of interconnected or network-like structures as a result of particle–particle and particle–polymer interactions (Kim, 2009b; Kim et al., 2008; Kim et al., 2006). The PBT nanocomposites exhibited shear thinning behavior, resulting from the breakdown of these structures with increasing frequency. For understanding the effect of CNT on the rheological behavior of PBT nanocomposites, it is also very instructive to characterize the variations of the shear thing exponent for PBT nanocomposites (Abdel-Goad & Pötscke, 2005). In the case of an ideal Newtonian fluid, the shear thinning exponent approaches or equals 0 and the viscosity is independent of the frequency, thus exhibiting the terminal flow behavior, whereas for the polymer nanocomposites, as the shearing thinning behavior develops, the shearing thinning exponent increases with increasing filler concentration (Abdel-Goad & Pötscke, 2005; Costa et al., 2006; Krishnamoorti & Giannelis, 1996; Krishnamoorti & Giannelis, 1997). The shear thinning exponent can be obtained from the slope of the plot of $|\eta^*|$ versus ω for PBT nanocomposites. As shown in Table 1, the shear thinning exponent of PBT nanocomposites slightly decreased with the introduction of CNT, and this effect was more pronounced at high CNT content, indicating significant dependence of the shear thinning behavior of PBT nanocomposite on CNT content.

The storage modulus (G') and loss modulus (G'') of PBT nanocomposites as a function of frequency are shown in Figure 2. The values of G' and G'' for PBT nanocomposites increased with increasing frequency, and this enhancing effect was more pronounced at low-frequency region. This rheological response is similar to the relaxation behavior of the typical filled-polymer composite systems (40-43). If polymer chains are fully relaxed and
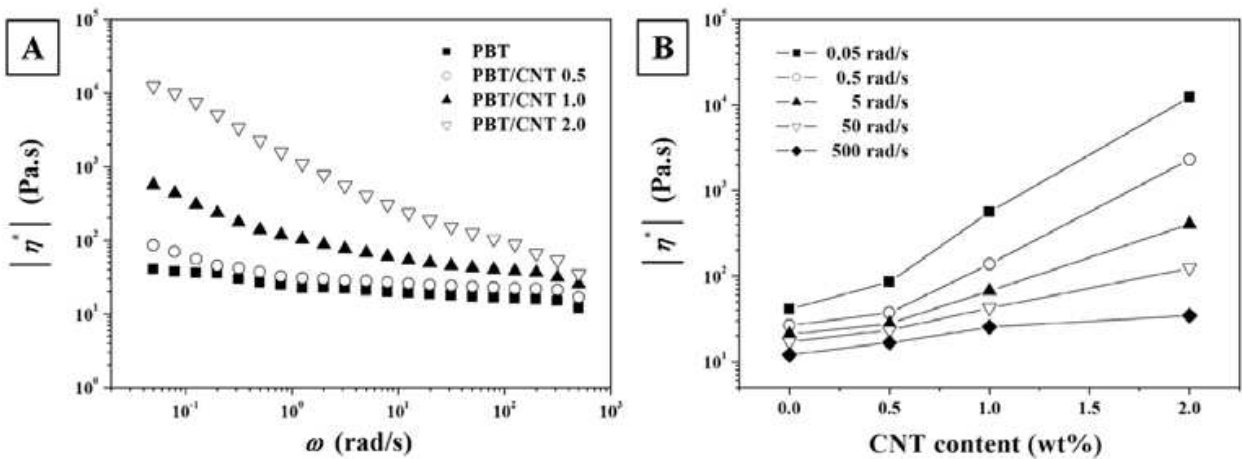
Fig. 1. (A) Complex viscosity ($|\eta^*|$) of PBT nanocomposites as a function of frequency. (B) Variations of $|\eta^*|$ of PBT nanocomposites with the CNT content at different frequencies. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

Materials	Slope of $|\eta^*|$ versus ω	Slope of G' versus ω	Slope of G'' versus ω
PBT	-0.1389	0.7639	0.7342
PBT/CNT 0.5	-0.1548	0.4286	0.5081
PBT/CNT 1.0	-0.3643	0.4030	0.5043
PBT/CNT 2.0	-0.7592	0.3128	0.4255

Table 1. Low-frequency slopes of $|\eta^*|$, G', and G'' versus ω for PBT nanocomposites

exhibit a characteristic homopolymer-like terminal behavior, the flow curves of polymers can be expressed by a power law of $G' \propto \omega^2$ and $G'' \propto \omega$ (Ferry, 1980). Krishnamoorti and Giannelis reported that the slopes of G' and G'' for the polymer/layered silicate nanocomposites were much smaller than 2 and 1, respectively, suggesting that large deviations in the presence of a small quantity of layered silicate were caused by the formation of network-like structures in the molten state (Krishnamoorti & Giannelis, 1997). The variations of the slopes of the terminal zone of G' and G'' for PBT nanocomposites indicate the non-terminal behavior with the power-law dependence for G' and G'' of PBT nanocomposites (Table 1). Similar observations have been reported in the ordered block copolymers and the smectic liquid-crystalline small molecules (Rosedalev & Bates, 1990; Larson et al., 1993). The decrease in the slopes of G' and G'' for PBT nanocomposites with increasing CNT content was explained by the fact that the nanotube–nanotube or the polymer–nanotube interactions (Kim, 2009b; Kim et al., 2008; Kim et al., 2006) can lead to the formation of the interconnected or network-like structures, resulting in the pseudo solid-like behavior of PBT nanocomposites. As shown in Figure 2, the extent of the increase in G' of PBT nanocomposites was higher than that of G'' over the whole frequency range measured, and the values of G' and G'' of PBT nanocomposites were higher than those of pure PBT, particularly at low frequency. This result demonstrates that the interconnected or network-like structures can be formed in PBT nanocomposites via the nanotube–nanotube or polymer–nanotube interactions in the presence of CNT, resulting in more elasticity of PBT nanocomposites. As the applied frequency increased, the interconnected or network-like structures were broken down due to high levels of shearing force, and PBT nanocomposites exhibited almost similar or slight higher G' and G'' values than those of pure PBT at high frequency.
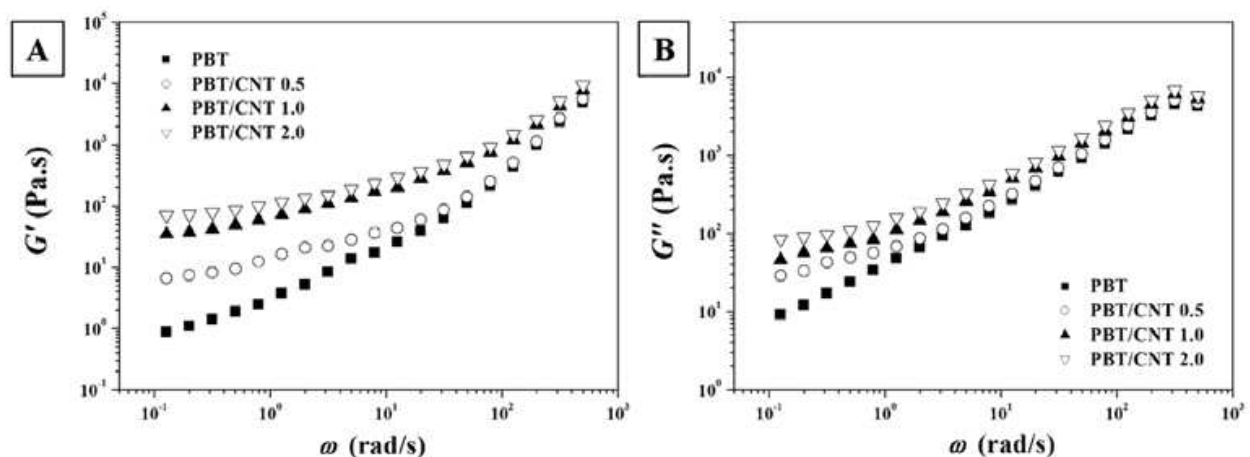
Fig. 2. (A) Storage modulus (G') and (B) loss modulus (G'') of PBT nanocomposites as a function of frequency. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

The variations of tan δ as a function of frequency for the PBT nanocomposites are shown in Figure 3A. Shear deformation can lead to partial orientation of the molecules in polymer chains, and tan δ decreased with increasing frequency. The maximum of tan δ for PBT nanocomposites shifted toward high-frequency region with increasing CNT content, indicating the formation of densely interconnected or network-like structures in PBT nanocomposites (Kim, 2009b; Kim et al., 2008; Kim et al., 2006). Figure 3B shows the plots of the phase angle (δ) versus the absolute values of the complex modulus ($|G^*|$) for the PBT nanocomposites, which is known as the Van Gulp-Palmen plot (Van Gulip & Palmen, 1998). It can be seen that a significant change in the δ values occurred with the introduction of CNT. For PBT nanocomposites, the decrease in the δ values with decreasing the $|G^*|$ values indicated that the incorporation of CNT into PBT matrix enhanced the elastic behavior of PBT nanocomposites. The plots of log G' versus log G'' for PBT nanocomposites are shown in Figure 4. In general, this plot provides a master curve with a slope of 2 for isotropic and homogeneous polymer melts, irrespective of temperature (Han et al., 1989). However, PBT nanocomposites did not provide a perfect single master curve and exhibited the shifting and change of the slope with the introduction of CNT. The slopes in the terminal regime of PBT nanocomposites were less than 2, indicating that PBT nanocomposite systems were heterogeneous and they underwent some chain conformational changes because of the interconnected or network-like structures via the nanotube–nanotube or nanotube–polymer interactions in the presence of CNT (Kim, 2009c; Kim et al., 2008; Kim et al., 2006). However, over the higher G'' values, the slopes of PBT nanocomposites increased and approached similar slope of pure PBT, indicating that the interconnected or network-like structures formed in PBT nanocomposites were broken down by high levels of shear force.

3.2 Dynamic mechanical properties
The dynamic mechanical properties of PBT nanocomposites are shown in Figure 5. There is a significant dependence of the storage modulus (E') and the tan δ for PBT nanocomposites on the temperature and the presence of CNT. As the molecular motions within the polymer nanocomposites change, the storage modulus of the polymer nanocomposites varied with the temperature. The E' values of PBT nanocomposites decreased rapidly, whereas the tan δ underwent a maximum when the polymer nanocomposites were heated through the glass
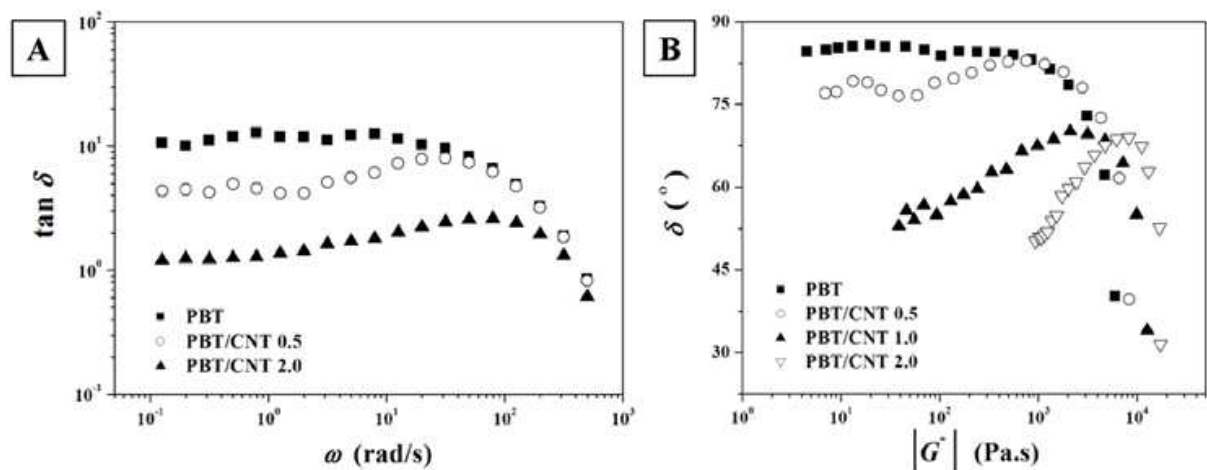
Fig. 3. (A) Variations of tan δ of PBT nanocomposites as a function of frequency. (B) Plots of the phase angle (δ) versus the complex modulus ($|G^*|$) of PBT nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.
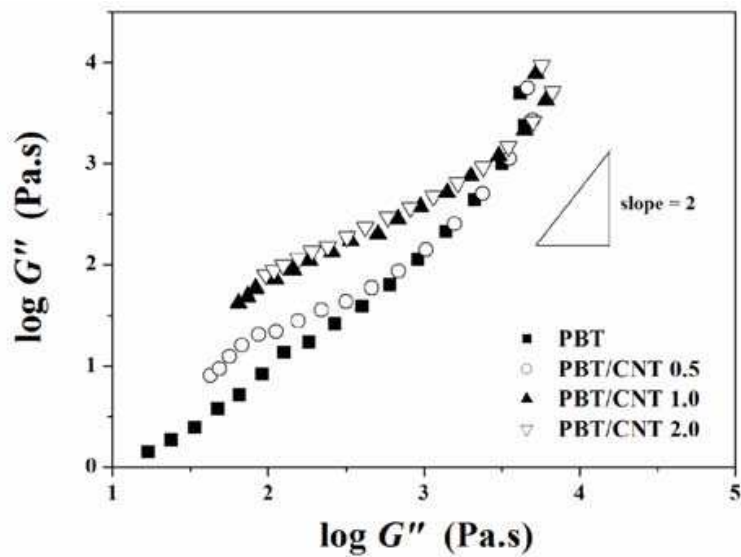

Fig. 4. Plots of log G' versus log G'' for PBT nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

transition region. The incorporation of CNT into PBT matrix significantly increased the E' values of PBT nanocomposites, which was attributed to the physical interactions between PBT and CNT with high aspect ratio and large surface areas as well as the stiffening effect of CNT as nanoreinforcing fillers, making it possible for them to allow efficient load transfer in PBT nanocomposites. As shown in Figure 5, the tan δ peaks of PBT nanocomposites as a function of temperature were not significantly affected in the presence of CNT, whereas the peak height was decreased with the introduction of CNT.

3.3 Morphology
SEM and TEM microphotographs of CNT and PBT nanocomposites are shown in Figure 6. The CNT exhibited highly curved and randomly coiled features and typically tends to bundle together or some agglomerated organization because of the intrinsic van der Waals attractions between the individual nanotubes in combination with high aspect ratio and large surface area
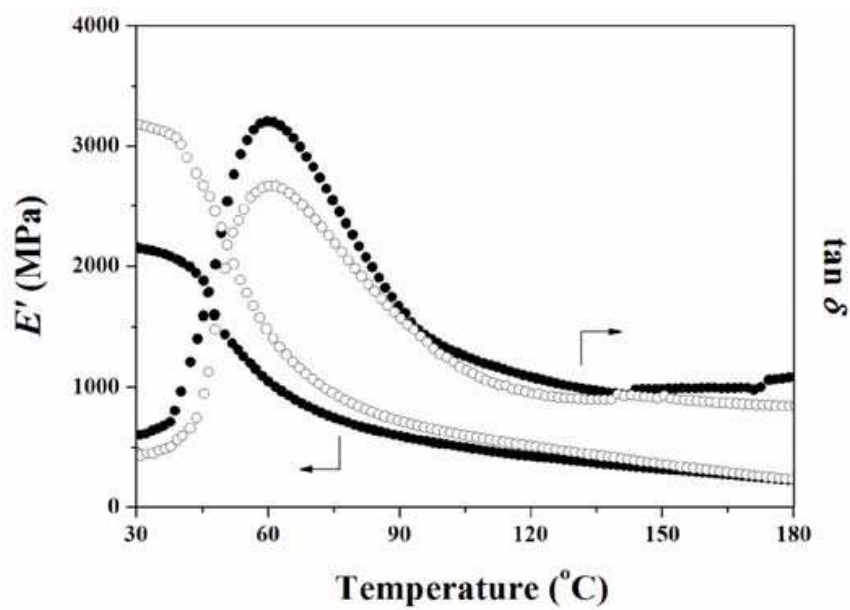
Fig. 5. Dynamic mechanical properties of PBT nanocomposites as a function of temperature. Filled and open circles represent the values of E' and tan δ for pure PBT and PBT/CNT 2.0 nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.
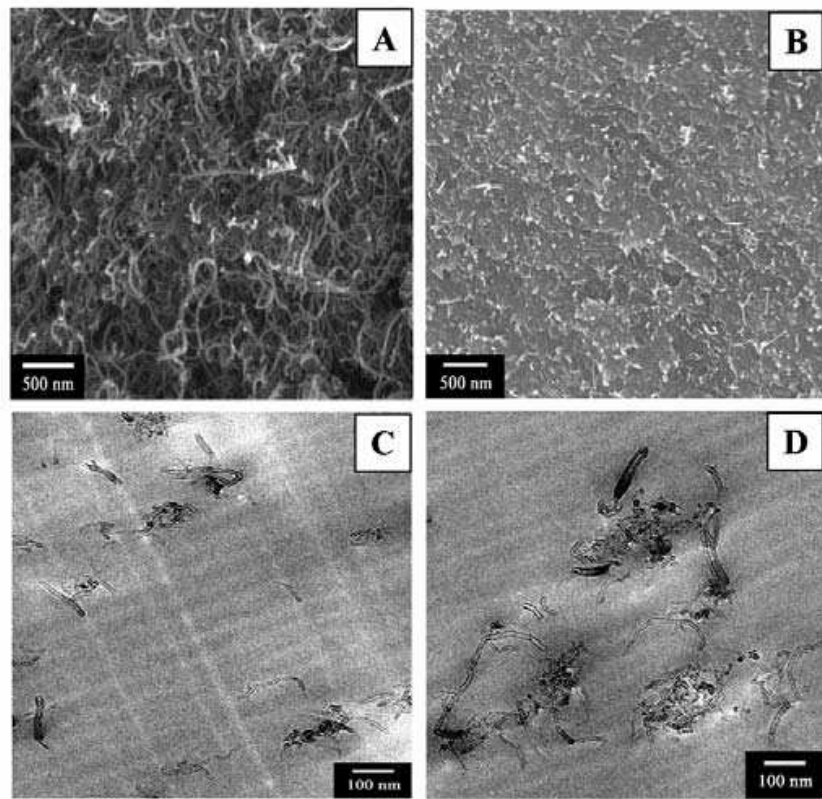

Fig. 6. SEM micrograph of (A) CNT and (B) PBT/CNT 2.0 nanocomposites. TEM images of PBT nanocomposites containing (B) 0.5 wt% and (C) 2.0 wt% of CNT. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

(Ebbesen, 1997; Dresselhaus et al., 2001). As shown in Figure 6A, CNTs were randomly oriented and formed the interconnected or network-like structures in PBT nanocomposites. The CNT with a small size, high aspect ratio, and large surface area are often subjected to self-agglomeration or bundle formation at higher concentration of CNT and thus easily form interconnected or network-like structures in the molten polymer matrix (Schadler et al, 1998). Some CNT bundles were pulled out from PBT matrix and some of them were individually dispersed in the polymer matrix. As shown in Figure 6C, TEM image of PBT nanocomposites containing 0.5 wt% of CNT showed that the dispersion of CNT was quite good. However, at high CNT content, less uniformly dispersed and highly entangled CNT structures were observed in PBT nanocomposites. The CNT was, on a large scale, uniformly dispersed in the polymer matrix despite some agglomerated CNT structures, and PBT nanocomposites exhibited relatively more uniform dispersion of CNT in PBT matrix particularly at lower CNT content in comparison with them at higher CNT content.

3.4 Mechanical properties

The mechanical properties of PBT nanocomposites with the CNT content are shown in Figure 7. There is a significant dependence of the mechanical properties of PBT nanocomposites on the CNT content. The incorporation of a very small quantity of CNT into PBT can substantially improve the mechanical properties of PBT nanocomposites due to the nanoreinforcing effect of CNT with high aspect ratio and uniform dispersion in the polymer matrix. The PBT nanocomposites exhibited higher tensile strength and tensile modulus than those of pure PBT. For instance, on the incorporation of CNT, the tensile strength and tensile modulus of PBT nanocomposites were significantly increased by 35.1 and 21.7%, respectively, and this enhancing effect was more significant at low CNT content as compared to high CNT content. The fact that the improvement in the mechanical properties of PBT nanocomposites was not increased at higher CNT content as expected, in comparison with that at low CNT content, can be explained by the characteristics of CNT that tended to bundle together because of their intrinsic van der Waals attractions between the individual nanotubes in combination with high aspect ratio and large surface area and could lead to some agglomeration, causing the stress concentration phenomenon and preventing efficient load transfer to the polymer matrix (Ajayan, 1999; Bokobza, 2007; Paul & Robesson, 2008). Similar observation has been reported that for CNT/polypropylene (PP) nanocomposite systems, adding a low level of CNT into PP improved the mechanical properties of CNT/PP nanocomposites, while at high CNT content, their mechanical properties decreased via stress concentration by the nanotube aggregation (Gorga & Cohen, 2004). The poor adhesion between CNT and PP as well as imperfection and defects in the nanotube structures resulted in the reduced mechanical properties of CNT/PP nanocomposites. For achieving further enhanced mechanical properties of PBT nanocomposites, the improvement in the dispersion state of CNT and the interfacial adhesion between two phases should be required (Kim et al., 2008). The elongation at break of PBT nanocomposites decreased with the introduction of CNT, indicating that PBT nanocomposites became somewhat brittle as compared to pure PBT due to the increased stiffness of PBT nanocomposites and the micro-void formed around the nanotubes during tensile testing. As shown in Figure 7B, the flexural strength and flexural modulus of PBT nanocomposites also increased with the introduction of CNT. This enhancement of the flexural strength/modulus was attributed to the reinforcement of PBT by incorporating the dispersed CNT and the moderate transfer of the applied stress between PBT and CNT. In addition, the enhancement of the flexural modulus of PBT nanocomposites was closely related to the improvement in the value of heat distortion temperature, which will be elaborated in the following section.
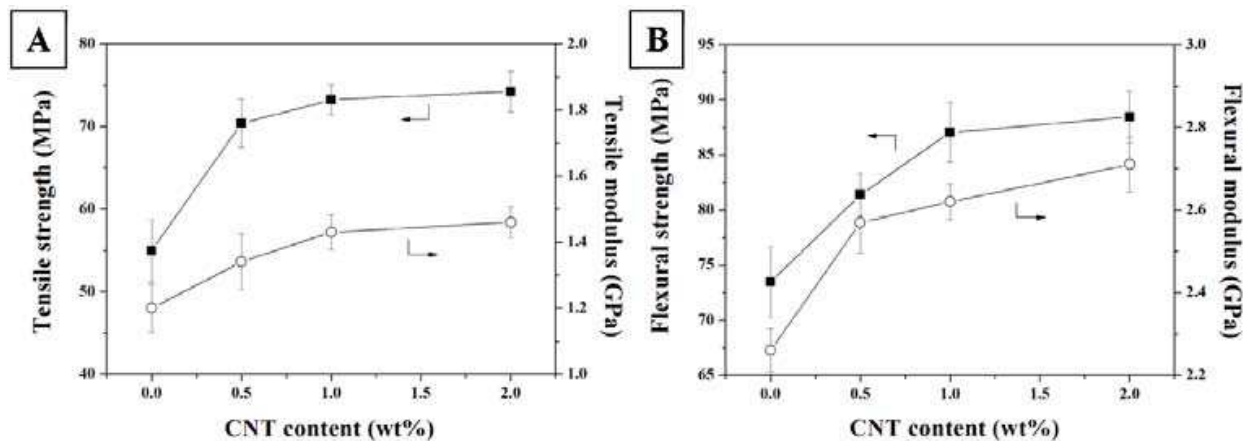
Fig. 7. Variations of (A) tensile strength and tensile modulus and (B) flexural strength and flexural modulus of PBT nanocomposites with the CNT content. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

For characterizing the effect of CNT on the mechanical properties of PBT nanocomposites, it is also very instructive to compare the reinforcing efficiency of CNT for a given content in PBT nanocomposites. The variations of the reinforcing efficiency of CNT in PBT nanocomposites are shown in Figure 8. The reinforcing efficiency of CNT can be defined as the normalized mechanical properties of PBT nanocomposites with respect to those of pure PBT as follows:

$$\text{Reinforcing efficiency (\%)} = \frac{M_c - M_m}{M_m} \times 100 \tag{1}$$

where M_c and M_m represent the mechanical properties, including tensile strength, tensile modulus, flexural strength, and flexural modulus, of PBT nanocomposites and pure PBT, respectively. The enhancing effect of the mechanical properties by incorporating CNT was more significant at low CNT content than at high content, indicating that a low CNT loading was more effective in improving the overall mechanical properties of PBT nanocomposites. At higher CNT content, CNT tends to bundle together and to form some agglomeration because of intrinsic van der Waals attractions between the individual nanotubes and lead to the stress concentration phenomenon, thus preventing the efficient load transfer to the polymer matrix. This result demonstrates that the incorporation of relatively small quantity of CNT into PBT matrix is more effective in the enhancement of the overall mechanical properties of PBT nanocomposites induced by high nanoreinforcing efficiency of CNT as well as good dispersion of CNT in the polymer matrix at lower concentration.

3.5 Heat distortion temperature

The elevated temperature property, typically estimated by heat distortion temperature (HDT), plays a critical role in determining the performance of engineering plastics. The HDT value is in common use to represent the upper limit of the dimensional stability of polymers in service without significant physical deformations under a normal load and thermal effect, providing important information for product design (Petaro, 2000; Wong, 2003). The HDT values can be influenced by various factors such as the melt and mold temperatures, the nucleating agent, and various processing conditions, which can be related to the mechanical behavior of polymer composites. As shown in Figure 9, the HDT values of PBT nanocomposites increased
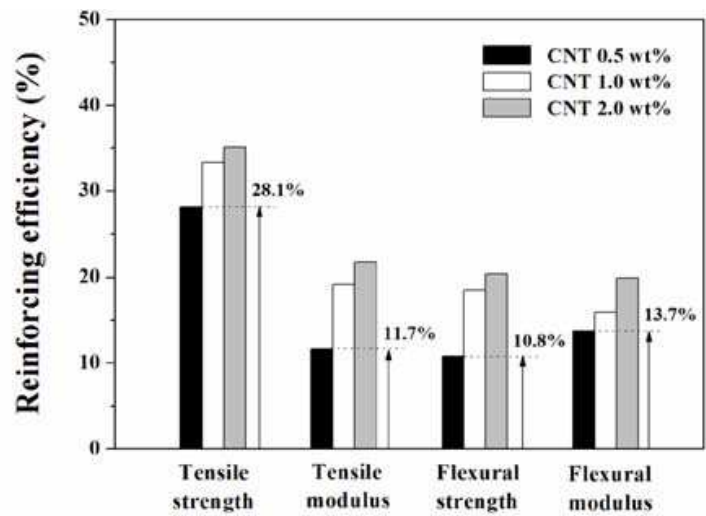
Fig. 8. Reinforcing efficiency of CNT on the mechanical properties of PBT nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

with increasing CNT content, which can be explained by the improvement in the modulus of the PBT nanocomposites. According to Nielsen's prediction (Nielsen, 1974), the variation of the HDT value was closely related to the behavior of flexural modulus with the filler content. The increase in the HDT values of PBT composites with increasing CNT content was in good agreement with the results of the flexural modulus of PBT nanocomposites as shown in Figure 7B, conforming to the Nielsen's prediction. The increased HDT values of PBT composites were attributed to the improvement in the flexural modulus with increasing CNT content. In the HDT measurements, the ability of polymeric materials to retain stiffness with increasing temperature is important for high HDT values (Thomasson & Groenewoud, 1996). As shown in Figure 5, the introduction of CNT could make it possible for PBT nanocomposites to maintain moderate modulus and high temperature stiffness with increasing temperature, which was also contributed to the enhancement of the HDT values of PBT nanocomposites. Thus, the improvement in the HDT of PBT composites resulted from the enhanced flexural modulus of PBT nanocomposites as well as the increased ability of PBT nanocomposites to retain high stiffness induced by CNT.
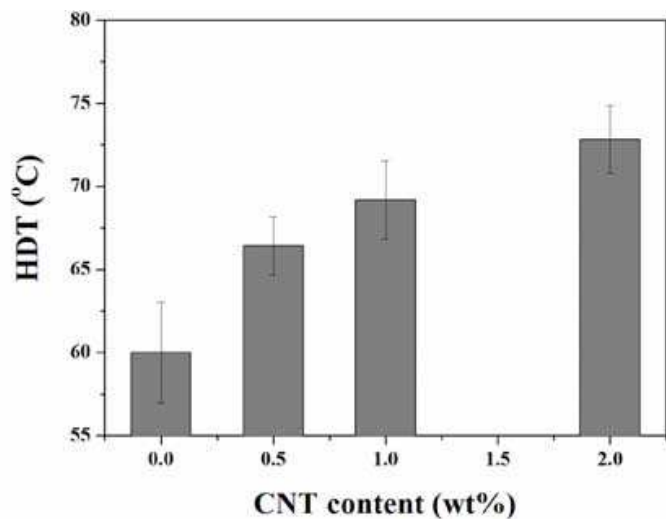

Fig. 9. Heat distortion temperature (HDT) of PBT nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

3.6 Thermal behavior

The incorporation of CNT into PBT matrix has less effect on the glass transition temperature (T_g) and melting temperature (T_m) of PBT nanocomposites (Table 2). The crystallization temperature (T_c) significantly increased with the introduction of CNT, and this enhancing effect was more pronounced at low CNT content. This result indicates the efficiency of CNT as strong nucleating agents for the crystallization of PBT, suggesting the enhancement of the crystallization of PBT nanocomposites in the presence of CNT. As shown in Table 2, the increase in the T_c of PBT nanocomposites with increasing CNT content, together with the fact that the degree of supercooling (ΔT) for crystallization decreased with increasing CNT content, also demonstrated that CNT could effectively act as strong nucleating agents in PBT matrix, resulting in the enhancement of the PBT crystallization. Thus, the incorporation of a very small quantity of CNT into PBT can effectively enhance the crystallization of PBT nanocomposites through heterogeneous nucleation. Similar observations have been reported for the CNT/polymer nanocomposites, i.e., the accelerated crystallization by the introduction of CNT through heterogeneous nucleation (Kim et al., 2006a; Kim et al., 2007; Kim et al., 2008; Kim, 2009b; Kim et al., 2009; Kim et al., 2010).

Materials	T_g (ºC)	T_m (ºC)	ΔH_m (J//g)	T_c (ºC)	ΔT [a] (ºC)
PBT	56.9	224.4	41.7	176.8	47.6
PBT/CNT 0.5	56.2	224.6	42.4	190.3	34.3
PBT/CNT 1.0	56.7	223.9	43.1	191.4	32.5
PBT/CNT 2.0	57.1	224.3	44.3	192.2	32.1

Table 2. Thermal behaviour of PBT nanocomposites with the CNT content [[a] The degree of supercooling, $\Delta T = T_m - T_c$]

Wide-angle X-ray diffraction (WAXD) analysis was conducted on the PBT nanocomposites to investigate the effect of the CNT on the structure of the PBT nanocomposites. WAXD patterns of PBT nanocomposites are shown in Figure 10. For pure PBT, strong diffraction peaks observed at near 15.8º, 17.1º, 20.4º, 23.1º, and 25.0º, respectively, were assigned to the (0-11), (010), (011), (100), and (1-11) reflections, indicating the α-form of PBT crystals with a triclinic configuration (Fakirov, 1993). The characteristics peaks of pure PBT were also observed in PBT nanocomposite and the position of their peaks remained almost unchanged with the introduction of CNT, despite some changes in the peak intensity. This result demonstrates that the incorporation of CNT into PBT matrix does not change the crystal structure of PBT nanocomposites. The crystallinity of PBT nanocomposites was slightly increased with the introduction of CNT, which can be explained by the supercooling temperature. For PBT nanocomposites, CNT acts as a strong nucleating agent in PBT matrix, and the crystallization temperature shifts to higher temperature, implying that the supercooling of PBT nanocomposites decreased with the introduction of CNT (Table 2). When polymers crystallized with less supercooling, it crystallized more perfectly than with more supercooling (Cheng & Wunderlich, 1988) and thus, the crystallinity of PBT nanocomposites slightly increased with the introduction of CNT.

3.7 Thermal stability and thermal decomposition kinetics

Thermal stability of polymer nanocomposites plays a critical role in determining the limit of working temperature and the environmental conditions for use of polymer nanocomposites, which is closely related to their thermal decomposition temperatures and decomposition
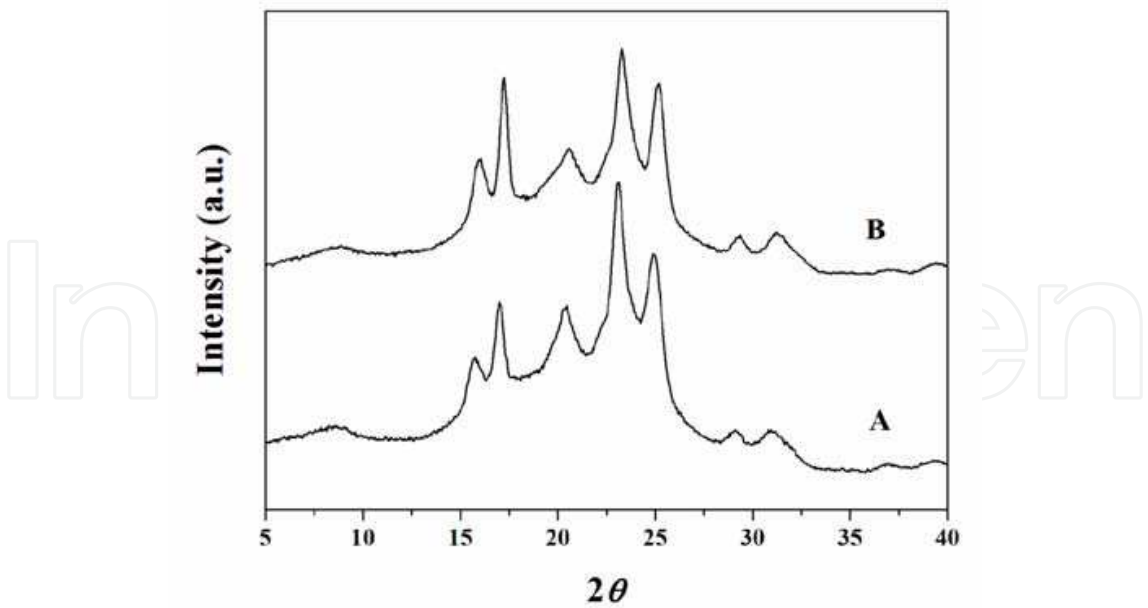
Fig. 10. Wide-angle X-ray diffraction (WAXD) patterns of (A) pure PBT and (B) PBT/CNT 2.0 nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

rates (Nair, et al., 1995). The TGA thermograms of the PBT nanocomposites with CNT content are shown in Figure 11A, and the results are summarized in Table 3. TGA curve of the thermal decomposition for pure PBT exhibited only one dominant decline of the residual weight, indicating the random scission of PBT main-chains as the prevailing decomposition reaction (Vijayakumar & Fink, 1982). The patterns of TGA curves for PBT nanocomposites are similar to that of pure PBT, indicating that the thermal decomposition of PBT nanocomposites primarily stems from PBT. The incorporation of CNT into PBT matrix can increase the thermal decomposition temperatures and the residual yields of PBT nanocomposites, indicating that the presence of CNT can lead to the stabilization of PBT matrix, resulting in the enhanced thermal stability of PBT nanocomposites. The CNT can effectively act as physical barriers to hinder the transport of volatile decomposed products out of PBT nanocomposites during thermal decomposition. The CNT layers exhibited a good barrier effect on the thermal degradation process, leading to the retardation of the weight-loss rate of thermal degradation products as well as the thermal insulation of polymers in the nanocomposites (Kashiwagi et al., 2002).

Materials	T_5 (°C)	T_{10} (°C)	T_{dm} (°C)	$IPDT$ [a] (°C)	W_R [b] (°C)
PBT	371.3	378.2	400.9	481.0	5.82
PBT/CNT 0.5	372.9	380.2	402.8	499.8	6.81
PBT/CNT 1.0	373.9	381.1	404.4	507.5	7.32
PBT/CNT 2.0	374.8	382.7	407.5	529.9	8.51

Table 3. Thermal stability of PBT nanocomposites with the CNT content [[a] The intergral procedure decomposition temperature, $IPDT = A \cdot K(T_f - T_i) + T_i$, where A is the area ratio of total experimental curve divided by total TGA curves, K is the coefficient A, T_i is the initial experimental temperature, and T_f is the final experimental temperature; [b] The residual yield in TGA thermograms at 800°C under N_2]
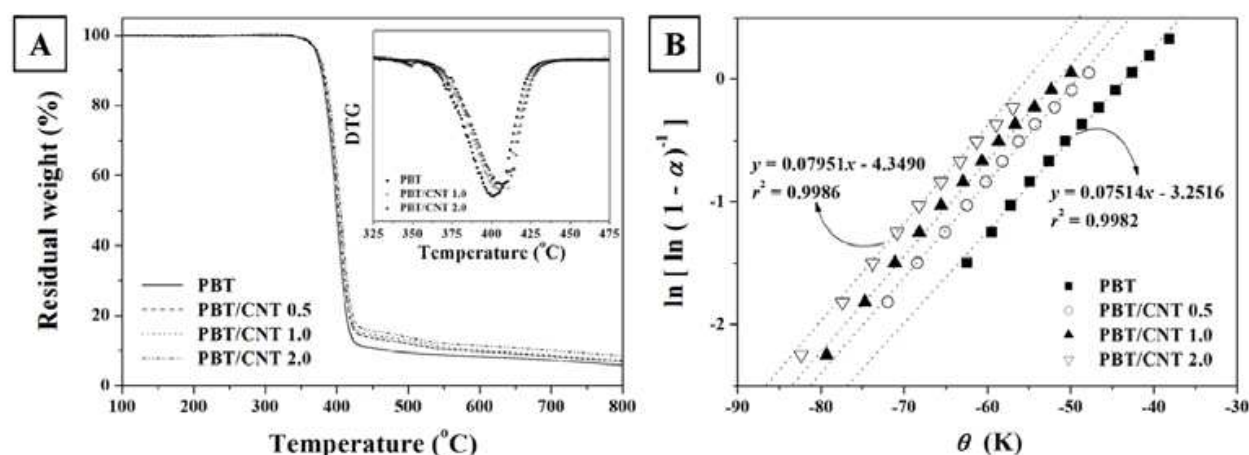
Fig. 11. (A) TGA thermograms of PBT nanocomposites. (B) Plots of $\ln[\ln(1 - \alpha)^{-1}]$ versus θ as shown for PBT nanocomposites. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

The TGA kinetic analysis was conducted on PBT nanocomposites to clarify the effects of CNT on the thermal stability of PBT nanocomposites. The thermal decomposition temperatures and decomposition kinetic parameters, including the thermal decomposition temperatures at 5 and 10% of the weight loss (T_5 and T_{10}), the temperature at maximum rate of the weight loss (T_{dm}), the integral procedure decomposition temperature (*IPDT*), and the activation energy for decomposition (E_a) are in common use to characterize the thermal stability of polymer nanocomposites (Park & Cho, 2000). As shown in Table 3, the thermal stability factors, including T_{d5}, T_{d10}, T_{dm}, and *IPDT*, of PBT nanocomposites were higher than that of pure PBT and they tended to increase with the CNT content. This result indicated that the incorporation of a very small quantity of CNT into PBT could substantially improve the thermal stability of PBT nanocomposites, and the thermal volatilization of PBT could be retarded in the presence of CNT during thermal decomposition. Because PBT molecular chains were more difficult to thermally decompose with the introduction of CNT, the residual yields of PBT nanocomposites also slightly increased with the CNT content. For PBT nanocomposites, the introduced CNT to induce protective barriers against thermal decomposition for organic species retarded the thermal decomposition of PBT nanocomposites, resulting from the physical barrier effects induced by CNT acting as the mass and heat transfer barriers (Kashiwagi et al., 2005). As a consequence, the thermal stability of PBT nanocomposites can be enhanced with the introduction of a very small quantity of CNT. Kashiwagi et al. reported that CNT layers exhibited a good barrier effect on the thermal degradation process and could lead to the retardation of the weight-loss rate of thermal degradation products as well as the slow down of thermal decomposition with the introduction of CNT, insulation of polymers in the polymer nanocomposites, resulting in the enhanced thermal stability of CNT/polymer nanocomposites (Kashiwagi et al., 2002). TGA results demonstrate that a very small quantity of CNT is beneficial to act as the thermal decomposition-resistant nanoreinforcing fillers in PBT nanocomposites.

The activation energy for the thermal decomposition (*Ea*) of PBT nanocomposites can be estimated from the TGA thermograms by the Horowitz-Metzger integral kinetic method (Horowitz & Metzger, 1963) as follows:

$$\ln\left[\ln\left(1-\alpha\right)^{-1}\right] = \frac{E_a\theta}{RT_{dm}^2} \qquad (2)$$

where α is the weight loss; θ is the variable auxiliary temperature defined as $\theta = T - T_{dm}$, and R is the universal gas constant. The E_a values of PBT nanocomposites can be estimated from the slope of the plot of $\ln[\ln(1 - \alpha)^{-1}]$ versus as shown in Figure 11B. The E_a values of PBT nanocomposites were 295.0, 297.5, and 306.2 kJ/mol, respectively. As compared to pure PBT (E_a = 283.8 kJ/mol), higher E_a values of PBT nanocomposites indicated that PBT nanocomposites were more thermally stable than pure PBT. The presence of CNT in PBT nanocomposites increased the activation energy for thermal decomposition of PBT matrix. The introduction of CNT, effectively acting as physical barriers or protective layers against the thermal decomposition resulted in the enhanced thermal stability of CNT-filled polymer nanocomposites. For PBT nanocomposites, the Horowitz-Metzger analysis demonstrated that the incorporation of CNT into PBT matrix increased the E_a values of PBT nanocomposites, which was related to the enhancement of the thermal stability of PBT nanocomposites. In addition, it can be deduced that the E_a values of PBT nanocomposites exhibits a good reliance on describing the thermal decomposition kinetics of PBT nanocomposites, which can be confirmed by the fact that the values of the correlation coefficient (r^2) were greater than 0.99.

The morphology of the residues of PBT nanocomposites containing 2.0 wt% of CNT after thermal decomposition is shown in Figure 12. The PBT nanocomposites exhibited the dispersed structure of CNT in PBT matrix kept after thermal decomposition, despite some collapse or loss of their form. This feature may also contribute to the enhancement of the thermal stability of PBT nanocomposites. This enhancing effect of CNT resulted from high thermal resistance of CNT to increase the energy required for thermal decomposition as well as physical barrier effect against thermal decomposition (Kashiwagi et al., 2005).
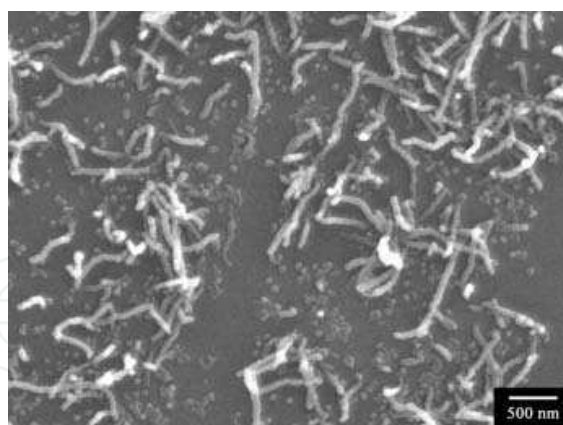

Fig. 12. SEM micrograph of the residues of PBT/CNT 2.0 nanocomposites after the thermal decomposition process. Reproduced with permission from Ref. (Kim, 2009b). © 2009 Wiley Periodicals, Inc.

4. Summary and outlook

This chapter describes the fabrication and characterization of poly(butylene terephthalate) (PBT) nanocomposites containing carbon nanotube (CNT). PBT nanocomposites reinforced with a very small quantity of CNT were prepared by a simple melt blending in a twin-screw

extruder to create high performance polymer nanocomposites for practical applications in a broad range of industries. There is significant dependence of the rheological, thermal, and mechanical properties of PBT nanocomposites on the CNT content. The non-terminal behavior of PBT nanocomposites was caused by the nanotube–nanotube or polymer–nanotube interactions, and the dominant nanotube–nanotube interactions at high CNT content resulted in the formation of the interconnected or network-like structures of CNT in PBT nanocomposites. The presence of a very small quantity of CNT can effectively act as a strong nucleating agent in PBT nanocomposites, resulting in the enhancement of PBT crystallization through heterogeneous nucleation. The mechanical properties of PBT nanocomposites substantially improved with the introduction of CNT, and this enhancing effect was more pronounced at lower CNT content, resulting from the nanoreinforcing effect of CNT with high aspect ratios and large surface areas to allow the efficient load transfer from the polymer matrix to the nanotube. The heat distortion temperature of PBT composites increased with the CNT content, which was related to the enhanced flexural modulus of PBT nanocomposites as well as the increased capability of PBT nanocomposites to retain high stiffness induced by CNT. The incorporation of CNT into PBT matrix increased the activation energy for thermal decomposition, and led to the enhanced thermal stability of PBT nanocomposites, resulting from physical barrier effects of CNT against thermal decomposition. Future development of PBT nanocomposite containing CNT for targeted applications in a broad range of industry will be performed by balancing high performance against their multiple functionality and manufacturing cost.

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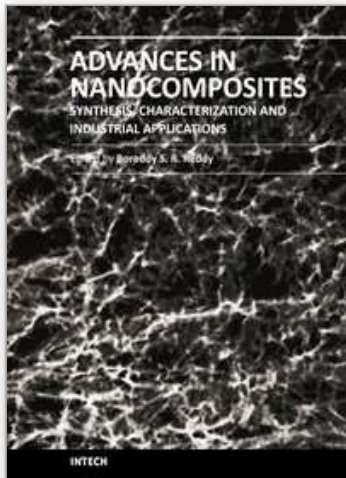
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Advances in Nanocomposites - Synthesis, Characterization and Industrial Applications was conceived as a comprehensive reference volume on various aspects of functional nanocomposites for engineering technologies. The term functional nanocomposites signifies a wide area of polymer/material science and engineering, involving the design, synthesis and study of nanocomposites of increasing structural sophistication and complexity useful for a wide range of chemical, physicochemical and biological/biomedical processes. "Emerging technologies" are also broadly understood to include new technological developments, beginning at the forefront of conventional industrial practices and extending into anticipated and speculative industries of the future. The scope of the present book on nanocomposites and applications extends far beyond emerging technologies. This book presents 40 chapters organized in four parts systematically providing a wealth of new ideas in design, synthesis and study of sophisticated nanocomposite structures.

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