

Mechanical and thermal properties of PEI/Polycarbosilane coated MWCNT Nanocomposite

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Abstract

Natural inorganic filler are added to thermoplastic polymers in a number of situations. The main aim of producing these polymer composites are both the reduction of industrial cost and also modification of certain characteristics such as mechanical properties, HDT and morphological properties. For the enhancement of mechanical and thermal properties, nanofiller particles are added in polymer matrix. Polyether imide (PEI) is an important class of thermoplastics polymer, which are widely used as matrix for the composite materials for high tech applications. The present study is focused to prepare PEI based nano composite incorporated with various loading of PCS coated MWCNT. Mechanical properties results demonstrate a significant improvement in strength, modulus, impact and hardness. The HDT also increases with the incorporation in PEI matrix. Enhancement in mechanical properties and HDT may be attributed to excellent dispersion of PCS coated MWCNT on the entire PEI matrix. Scanning electron microscopy (SEM) results reveal a good interfacial adhesion between PCS coated MWCNT and PEI matrix.

Key words: PEI, PCS, MWCNT, Mechanical properties, SEM.

1. Introduction

Composites materials are defined as a multicomponent material having multiple different phase domains in which at least one type of phase domain is continuous. If, however, one of the phases has dimension $> 100\text{nm}$, then the material is thought to be nanocomposites. Recently carbon nanotubes composites have drawn greater attentions of the investigators all over the world owing to their unique and excellent mechanical, thermal and electrical properties and many structural and smart applications of them have been proposed [1]. Among them one of the most intriguing applications of CNTs in the polymer/CNTs nanocomposites. However, challenges for the development of high performance CNTs/polymer nanocomposites are: (a) homogeneous dispersion of CNTs in the matrix and (b) strong interfacial interactions so as to affect load transfer from the polymer matrix to CNTs. There has been an increasing interest in the studies of polymer/CNTs nanocomposites due to the unique combinations of promising properties and fabrications of multifunctional structures of each component [2]. These nanocomposites have superlative physical properties such as enhanced tensile strength [3, 4] and improved electrical properties [5, 6]. Several polymers such as epoxy [7], polycarbonate [8, 9], polyamide [3, 4], polyimide [10, 11] polystyrene [12] and polypropylene [13] have been utilized to prepare polymer/CNTs

nanocomposites. The benefit of formation of CNTs nanostructures in polyimide based composites throughout the matrix has been successfully demonstrated [14].

Polyetherimide (PEI) has been chosen in the study because it belongs to the polyimide family of polymers and has been proven to have the excellent combination of mechanical prosperities, thermo-oxidative stability and processability desirable in aerospace applications [15]. Moreover, it shows a much wider processing window than other polyetherimide that are intractable with high melting temperature and viscosity with less solubility. It can be easily processed using conventional extrusion, injection or blow moulding machines. However, compared with other conventional polyimides, it is relatively poor solvent resistance, dimensional stability and thermal stability limit to wider applications in many fields. It is expected the significant property enhancement for polyetherimide can be achieved by uniform dispersion of carbon nanotubes in PEI matrix, thus extending their utilities [16]. It is normally difficult to achieve homogeneous dispersion of CNTs in PEI matrix. To overcome this problem and to improve the dispersion of CNTs in the polymer matrix, fictionalization of CNTs have been done by many research groups [17, 18].

In the present investigations multiwall carbon nanotubes (MWCNTs) are modified with polycarbosilane (PCS) derived β -silicone carbide particles. The composites have been prepared by melt mixing process with the help of co-rotating twin screw extruder and their mechanical properties have been studied with special attains to the dispersion of modified MWCNTs in the PEI matrix.

2. Materials and methods

2.1. Materials

PEI (grade ULTEM 1000) purchased from Sabic innovation plastics (USA) is used as matrix materials. The multiwall carbon nano tubes (MWCNT) and polycarbosilane (PCS) were synthesised by DMSRDE, Kanpur. MWCNT having a diameter 2-4 nm and length 20-30 micrometer and the aspect ratio 10,000.

2.2. Coating of inorganic polymer on MWCNT

Took 2 liter tetra hydro furan (THF) and 100gm KOH pass it over 500 gm Alumina to remove the moisture, press sodium in this solution and leave the solution for two days a bluish colour is produced. Distillation of this solution was done and then took 100 ML of this solution in a bottle and add 100 mg PCS in it. Now took 40 ML of the above solution and add 0.4 gm MWNT in it then reflux it for 6 to 7 hours. Now distillation of the above solution is done at 90⁰C then they obtained residues is heated up to 25⁰C and then leave it to cool as a result we get PCS coated MWCNT.

2.3 Dispersion of inorganic polymer coated MWCNT's

Before incorporation of PCS coated MWCNT's from 0.1 to 0.3 phr in PEI . The nanotubes have been sonicated in 10 ml of toluene for a period of 30 minutes to separate the agglomerated polycarbosilane coated MWCNT's at in order to induce an efficient dispersion of nanotubes.

2.4 Preparation of organic/inorganic polymer Nanocomposites

Before mixing, the organic polymer (PEI) was dried under vacuum at 80°C for at least 12 hrs. Then after organic polymer and inorganic polymer with nanoclay (Polycarbosilane coated MWCNT) were blended in twin screw extruder (at RPM 145) at temperature processing condition given in Table 2.1 for PEI blend with inorganic polymer as per formulation Table 2.2. Testing specimens were prepared using injection moulding machine at temperature 340°C for PEI & PEI/PEI blend with inorganic polymer / nanoclay.

Table – 2.1 Processing temperature of Twin Screw Extruder during the compounding of PEI nanocomposites

Heating Zone	PEI
Zone 1	320 °C
Zone 2	350 °C
Zone 3	360 °C
Zone 4	375 °C
Zone 5	380 °C
Die	385 °C

Table – 2.2 Formulation of the nanocomposite based on PEI/PCS coated MWCNT blend

Sr. No.	PEI (gm)	PCS coated MWCNT (%)
1.	500	0.1
2.	500	0.3
3.	500	0.5

2.5 Testing and characterisation

Test specimens were prepared by injection moulding for the analysis of Mechanical and thermal properties.

2.5.1 Mechanical & Thermal properties

Density were measured as per ASTM D 792, Tensile properties were determined using dumbbell shaped specimen as per ASTM D 638, Flexural properties were measured as per

ASTMD 790 using universal testing machine (model INSTRON 3382, USA). The thermal properties heat deflection temperature (HDT) was measured at 1.82 MPa as per ASTMD 648. During testing the test atmosphere was maintained at 23+/- 2 deg C and 50+/- 5 RH.

2.5.2 Morphological Studies

Scanning electron microscopy (SEM) techniques was used to analyse the morphological properties of PEI/ Polycarbosilane coated MWCNT. Prior to SEM analysis the tensile strength broken samples were gold coated with the help of gold sputtering unit for avoiding charge effect. SEM studies were done using Carl Zeiss EVO-50*VP low volume scanning electron microscopy.

3.0 Results and Discussion

3.1: Mechanical and Thermal Properties

It has been observed that there is significant enhancement in mechanical properties i.e. (Tensile strength, tensile modulus, elongation at break, flexural strength, flexural modulus and hardness) at the developed nanocomposites with the incorporation of PCS coated MWCNTs by varying loadings in the PEI matrix. Mechanical properties results reveals that the incorporation of 0.3 wt% PCS coated MWCNTs provide an appreciable enhancement in mechanical properties. This increase in mechanical properties may be because of higher aspect ratio and surface area of MWCNTs. Higher surface area of MWCNTs may provides an excellent interaction and orientation of nanofillers with PEI matrix. The increased in the heat deflection temperature (Table 3.4) may be due to the incorporation PCS coated MWCNTs which reduces the chain mobility of the PEI matrix by imposing vast number of resicted sides that reduces the thermal vibration of carbon-carbon bond. The mechanical and thermal properties are shown in tables 3.1, 3.2, 3.3 and 3.4.

Table 3.1: Density, Rockwell hardness of PEI with Inorganic polymer coated MWCNT composite

Composition		Density, g/cc (PEI density – 1.26 g/cc)	Rockwell Hardness, (M Scale) (PEI Hardness – 108)
PEI, gm	Filler %	PCS coated MWCNT	
500	0.1	1.28	111
500	0.3	1.29	114
500	0.5	1.29	112

Table 3.2: Tensile Strength, Elongation and Modulus of PEI with Inorganic polymer coated MWCNT composite

Composition		Tensile strength, MPa (PEI Tensile Strength – 104.32 MPa)	Tensile Elongation, % (PEI Tensile Elongation – 7.11%)	Tensile Modulus, MPa (PEI Tensile Modulus – 3406.43 MPa)
PEI, gm	Filler %	PCS coated MWCNT		
500	0.1	118.72	6.7	3789.23
500	0.3	128.64	6.5	3976.91
500	0.5	123.56	6.8	3826.23

Table 3.3: Flexural Strength & Modulus of PEI with Inorganic polymer coated MWCNT composite

Composition		Flexural strength, MPa (PEI Flexural Strength – 163.24 MPa)	Flexural Modulus, MPa (PEI Flexural Modulus – 3428.12 MPa)
PEI, gm	Filler %	PCS coated MWCNT	
500	0.1	173.24	3634.46
500	0.3	180.67	3999.03
500	0.5	177.25	3739.72

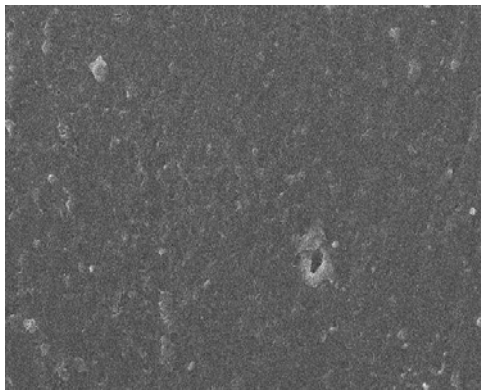
Table 3.4: Izod Impact & HDT of PEI with Inorganic polymer coated MWCNT composite

Composition		Izod Impact, J/m (PEI Izod Impact – 51.0 J/m)	Heat Deflection Temperature (HDT), Deg C (PEI HDT – 206 Deg C)
PEI, gm	Filler %	PCS coated MWCNT	
500	0.1	62	212.6
500	0.3	71	218.6
500	0.5	60	214.3

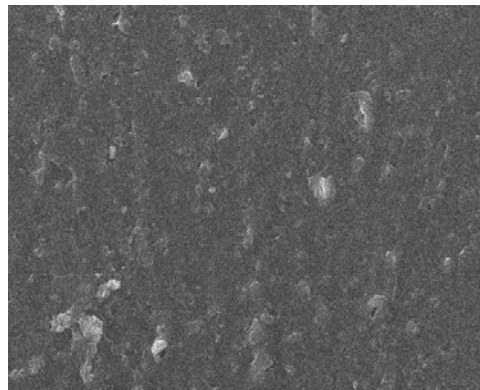
3.2 Morphological properties

SEM micrographs were shown in Figure 3.1 (a, b, c, d) demonstrate that excellent dispersion of PCS coated MWCNTs have been found at 0.3 wt % loading of PCS coated MWCNTs in PEI matrix. It is also obvious from scanning electron microscopy (SEM) micrograph at 0.3 wt % loading of PCS coated MWCNTs has uniform and homogeneous

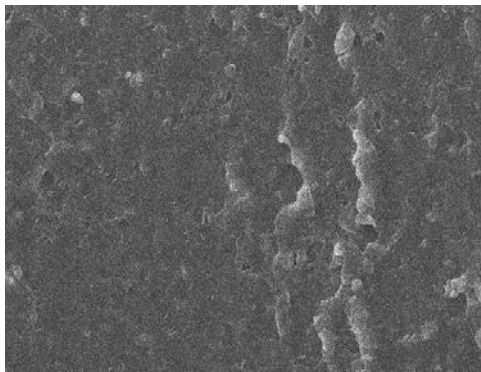
dispersion over the entire polymer matrix because of their significant in enhancement in mechanical properties and HDT.



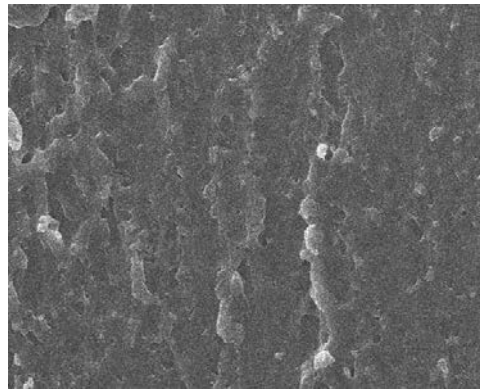
a) PEI virgin



b) 0.1% filler in PEI



c) 0.3% filler in PEI



d) 0.5% filler in PEI

Fig 3.1 Morphological properties of PEI/ PCS coated MWCNT

4.0 Conclusions

It can be concluded that at 0.3 wt % PCS coated MWCNTs are capable of enhancing the mechanical properties as well as heat deflection temperature (HDT) of developed nanocomposites. Scanning electron microscopy (SEM) results demonstrate that there is excellent dispersion of PCS coated MWCNTs over the entire polymer matrix which results in an appreciable enhancement in mechanical properties and HDT.

References

- [1] Iijima S, Helical microtubes of graphite carbon. Nature 1991: 354 (6348) ; 56-8.
- [2] Ajayan PM, Nanotubes from carbon. Chem Rev 1999(7): 1787-99.
- [3] Zhang WD, Shen L, Phang by simple melt compounding. Macromolecules 2004: 37 (2): 256-9.

- [4] Liu TX, Phang IY, Shen L, Chow SY, Zhang WD, Morphology and mechanical properties of multiwalled carbon nanotubes reinforced nylon 6 composites. *Macromolecules* 2004; 37(19): 7214-22.
- [5] R.Ravikumar, NR Schadler LS, Zhao YP, Wei BQ, Ajayan PM. Synthesis and characterization of thickness aligned carbon nanotubes-polymer composites films. *Chem Mater* 2005; 17; 974-83.
- [6] Du FM, Scogna RC, Zhou W, Brand S, Fisher JE, Winey KI. Nanotube networks in polymer nanocomposites: rheological and electrical conductivity. *Macromolecules* 2004 :37 (24): 9048-55.
- [7] Lau KT, Lu M, Lam CK, C Cheung HY, Sheng FL, Li HL. Thermal and mechanical properties of of single-walled carbon nanotubes bundle reinforced epoxy nanocomposites: the role of solvent for nanotubes dispersion. *Composites Sci Tech* 2005, 65 (5): 719-25.
- [8] Satapathy BK, Weidisch R, Potschke P, Janke A, Crack toughness behavior of MWCNTs/polycarbonate nanocomposites. *Macromol rapid commun* 2005: 26 (15):1246-52.
- [9] Potschke P, Bhattacharya AR, Janke A, Goering H. Composite of polycarbonate with multiwalled carbon nanoyubes produced by melt mixing. *ACS Symp Ser* 2005: 898: 148-63.
- [10] Zhang WD, Phang IY, Liu TX, Growth of carbon nanotubes on clay: unique nanostructured filler for high- performance polymer nanocomposites. *ADV Mater* 2006, 18(1):73-7.
- [11] Park C, Quanten Z, Watson KA, Crookes RE, Smith J, Lowether SE, et al, Dispersion of single carbon nanotubes by in-situ polymerization under sonication. *Chem Phys Lett* 2002: 364(3): 303-8.
- [12] Besancon BM, Green PF. Polystyrene-based on single carbon nanotubes nanocomposites thin films dynamics of structural instabilities. *Macromolecules* 2005: 38 (1):110-15.
- [13] Kashiwagi T, Grukle E, Hilding J, Harris R, Award W, Douglass J. Thermal degradation and flammability properties of polypropylene/carbon nanotubes composites. *Macromole Rapid Commun* 2002, 23(13): 761-5.
- [14] Ogasawara T, Ishida Y, Ishikawa T, Yokota R, Characterization of multiwall carbon nanotubes/phenylethylene terminated polyamide composites. *Composites, Part A* 2004: 35(1):67-74.
- [15] Wilson D, Stenzenberger HD, Hergenrother PM, *Polimide*. London: Chapman & Hall: 1990.
- [16] Morgan AB, Gilman JW, Jackson CL. Charecterization of the dispersion of clay in a polyetherimide nanocomposites. *Macromolecule* 2001: 34(8):2735-8.
- [17] Hirsch A, Vostrowsky O, Functionalization of carbon nanotubes. *Top Curr Chem* 2005: 245:193-237.
- [18] Balasubramanian K, Burghard M. chemically functionalized carbon nanotubes. *Small* 2004: 1:180-92.