

Some Studies on Preparation and Mechanical Characterization of Poly-aryl-ether-ketone (PAEK)/MWNT Polymer Nanocomposite (PNC)

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Abstract

It was proposed to create an advanced material with the requisite mechanical characteristics. Poly-aryl-ether-ketone (PAEK) was chosen as the polymeric matrix material for this investigation. It was intended to prepare various compositions with nano-filler added to enhance mechanical qualities. To enhance the desired property, multiwalled carbon nanotubes (MWNT) were intended to be used as nanofiller materials. The fillers ranged from 0, 0.3, 0.5, and 1 weight percent. In order to increase the dispersion of nanofiller material in the polymer matrix phase, the polymer nanocomposites (PNC) were synthesised using the melt mixing method. Additional mechanical characterisation procedures, such as tensile testing, impact testing, and flexural testing, will be used to assess PNC. At 0.5 and 1 weight percent of filler loading, MWNT considerably enhances mechanical characteristics. The mechanical properties of PAEK polymer are improved by the addition of MWNT. Moreover, the inclusion of MWNT significantly improves storage modulus.

Keywords: Poly-aryl-ether-ketone; Multiwalled carbon nanotubes; Polymer nanocomposites

1. Introduction

Phenylene rings connected by ether and carbonyl groups (ketone)-based oxygen bridges define PAEK polymers. The glass transition temperature and melting point of the polymer are mostly influenced by the ratio and order of ether to ketone. It also has an impact on the processing temperature and heat resistance. A higher glass transition temperature and melting point are produced by a polymer chain that is more stiff as ketone content increases. Temperatures used for processing can range from 350 to 430 °C [1]. This family of plastics includes, among others, PEK, PEEK, PEKK, PEEKK, and PEKEKK. A polymeric material can be modified to have specific qualities by blending it with another polymer, employing the right filler, or utilising a combination of the two. Focusing on the filler-matrix interactions and minimising the filler-filler interactions will improve the characteristics of nanofiller based polymer blends. As a result, the performance of the materials is best increased when the degree of intermolecular interactions between the polymer and filler is perfectly balanced. The advancement of technology for the blending of well-known polymer systems has accelerated recently [2].

Properties

The maximum working temperature for PAEK is 370 °C (698 °F), with a continuous running temperature of 250 °C (482 °F). It emits the least caustic and harmful gases when burned. Moreover, it produces less heat when burned, making it appropriate for use in interior aviation applications. Moreover, it has greater tensile strength of 85 MPa (12,300 psi), better Young's modulus of 4,100 MPa, and good overall chemical resistance (590,000 psi). At 23 °C (73 °F), its yield strength is 104 MPa (15,100 psi), and at 160 °C (320 °F), it is 37 MPa (5,400 psi). In an unnotched Izod impact test, it holds up [3].

Materials

2. In a polymer nanocomposite, the polymer poly-aryl-ether-ketone (PAEK) will serve as the matrix. As a filler, multi-walled carbon nanotubes will be used (MWNT). Under the brand name G-PAEK, PAEK powder was obtained from Gharda Chemicals in Bombay. It was a low viscosity, unreinforced powder. The propitiatory CVD technique is used to create the carbon nanotubes. This item can be used as a reinforcing filler in base polymers to create nano- and bio-nanocomposites. MWNT can be found as a black powder with an outer diameter of 10 to 30 nm, a length of 10

microns, and a purity of greater than 95% [4].

3. Material Preparation

In this work, PAEK polymer based nanocomposites were prepared by melt-mixing method. This method can provide uniform dispersion of filler in polymer. Various PAEK nanocomposites samples are prepared by variation in filler contents such as 0, 0.3, 0.5 and 1 wt% of MWNT. It is denoted by PCNTX; where P represents MWNT and X represents as PAEK, carbon nanotubes and filler weight percentage, respectively. The composites with sample codes are listed in table 1. PAEK blends with MWNT were prepared by melt-compounding using Coperion ZSK 26 (Germany) twin-screw extruder. Extrusion process carried out as rotational speed of 60 rpm with maximum barrel temperature 400 °C with 10 temperature zone and flow rate as 12 kg/hr. All blend components viz. polymer and fillers were pre-dry in vacuum oven at 120 °C for 6 hr. Before extrusion 0.2 % (2 gram) TNPP added as an antioxidant in each PAEK composite batch. Further polymer and required amount of filler mixed in Neoplast high speed mixer about 5 minutes.

PAEK and filler will melt-mixed in twin screw extruder then blend strands passed through blower for cooling and it palletize by Lunarmech palletizer [4]. The specifications of the twin screw extruder Coperion ZSK 26 are as follows: screw diameter as 26 mm while length to diameter ratio as 40:1. Injection-molded samples (according to ASTM standards) were prepared using ARBURG 320 °C ALLROUNDER injection-molding machine. The injection-molding parameters maintained for all the compositions were injection flow 40 cm/s, melt temperature 400 °C and divided into 5 zones, mold temperature 190 °C, injection time 5 second, holding time 10 second, and cooling time 20 second. The barrel diameter of injection molding machine is 30 mm [5]. After preparation of polymer nanocomposites, those composites are characterized by various techniques such as tensile testing, impact testing, and flexural testing. Result of mechanical tests recorded as the average of five repetitive observations.

Table 1. The sample codes and blends composition

Polymer Filler	Polymer Filler	Codes
PAEK (1000 grams)	-	PCNT0
PAEK (997 grams)	MWNT 0.3 wt% (3 grams)	PCNT0.3
PAEK (995 grams)	MWNT 0.5 wt% (5 grams)	PCNT0.5
PAEK (950 grams)	MWNT 1 wt% (10 grams)	PCNT1

4. Results and Discussion

Tensile properties

The mechanical behavior of blends is strongly decided by the contribution of each component, as well as by the morphology developed during compounding and the interfacial adhesion between the phases. It is well realized that elongation at break and toughness are the important tools to monitor the adhesion between phases whereas the tensile strength is related to the morphology, domain size and size homogeneity. To investigate the effect of multi walled carbon nanotubes filler on the mechanical properties of the PAEK and its nanocomposites, tensile testing has been carried out. The effects of blending sequence on the mechanical behavior of the blends have been investigated. The results are illustrated in table 2 given below.

Table 2. Tensile testing of PAEK/MWNT filler blends

Composition	Tensile Strength (MPa)	Modulus (MPa)	Elongation at break (%)
PCNT0	81	4200	13.8
PCNT0.3	98	5750	4.3
PCNT0.5	103	5405	4.9
PCNT1	99	5180	4.2

It can be seen that the PAEK/MWNT blends has much higher tensile strength than neat PAEK. As filler concentration increases affects favourable effect on tensile strength that is it also increases. When talking about composites, the usual trade between tensile strength and elongation is that one grows while the other decreases. Hence the elongation at break should be decrease and that is shown in table and plot [6]. From fig. 1 it can be seen that the neat PAEK has tensile strength of 81 MPa while by addition of 1 wt% MWNT, increases to 27.16 %. By addition of 1 wt% MWNT reduced elongation value from 13.8 % (neat PAEK) to 4.2 %. The incorporation of filler is indicating interference by the filler in the deformability or mobility of polymer. The interference is created through

physical interaction and immobilization of matrix by mechanical restraints [6]. MWNT does not allow polymer extend more, it becomes constraint in polymer linkages to move freely. As the filler concentration increases, material becoming more brittle and leads to brittle failure. By reduction in elongation, it shows poor adhesion between polymer and MWNT filler. Modulus of nanocomposites shows better results compared to unreinforced polymer. As the filler concentration increases leads to enhancement in elastic modulus. Fig. 2 shows that by addition of 1 wt% MWNT increases modulus of composite nearly by 23.33 % compared to neat PAEK polymer. After the addition of MWNT in polymer matrix, elastic modulus clearly increased due to rigidity of MWNT particles. This phenomenon mainly attributed to lower mobility of polymer in presence of MWNT [6].

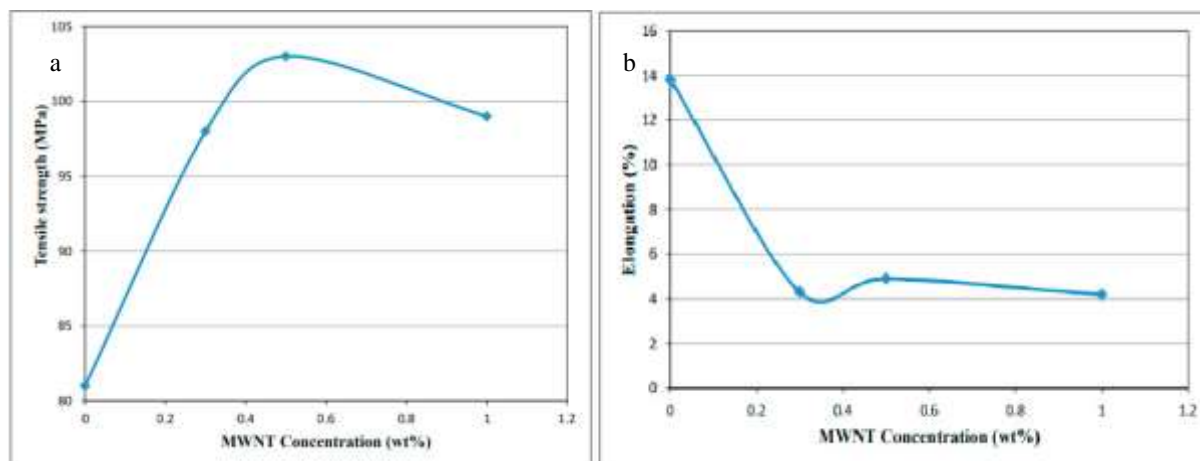


Fig. 1. (a) Semi-log plot of the tensile strength as a function of MWNT concentration; (b) Semi-log plot of the elongation as a function of MWNT concentration.

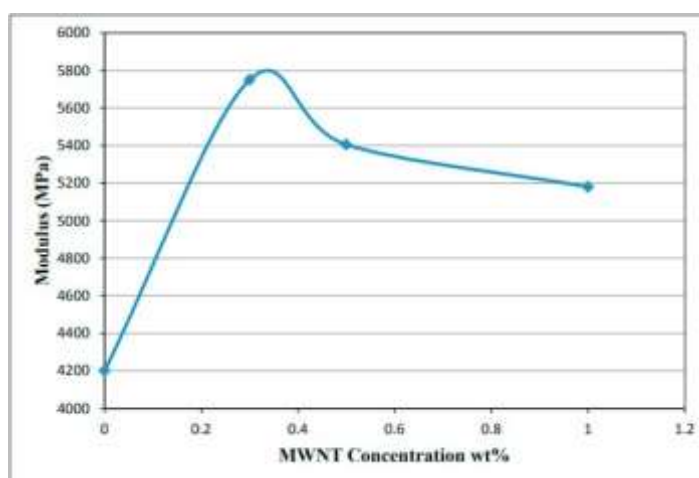


Fig. 2. Semi-log plot of the modulus as a function of MWNT concentration.

Impact properties

MWNT filled blends show inferior mechanical properties in the tensile tests exactly opposite trend is follow in Izod notched impact tests. It is also concluded that the elongation at break, as an indicator for the toughness of the materials, decreases dramatically when adding MWNT to PAEK. The results of impact tests are shown in table 3, which depicts impact strength or toughness decreases by the addition of MWNT. Both impact strength and elongation shows similar nature of toughness with MWNT filler.

Table 3. Izod notched impact test of PAEK nanocomposites

Composition	Bonding Energy (J)	Impact Strength (J/m)
PCNT0	0.1864	45
PCNT0.3	0.1633	37
PCNT0.5	0.1143	37
PCNT1	0.1035	37

Figure 3 exhibits that the addition of MWNT by 1 wt% results in reduction in impact strength around 32 %. Impact strength decreased with filler addition because of the MWNT agglomeration in PCNT1 polymer nanocomposite, due to this the toughness of PNC is reduces [7]. The mechanical properties of composites generally depend on many factors such as aspect ratio of filler, degree of dispersion of filler and adhesion between matrix and filler. Due to poor adhesion between phases, insufficient load transfer to fillers occurs. Also by presence of filler aggregates impact properties deteriorates. To recover this problem we have to use the modifiers in MWNT. Rigid particles of filler might create stress concentrated points leads to reduction in deformability of matrix. Also filler agglomerates acts as stress concentrated areas and decreased the impact strength of composite [7].

Flexural testing

To investigate the effect of MWNT reinforcement on flexural properties, flexural testing is carried out. Flexural testing results taken as average of minimum of three specimens per category in a three point bend configuration. The results of flexural strength and flexural modulus of given composites are shown in table 4.

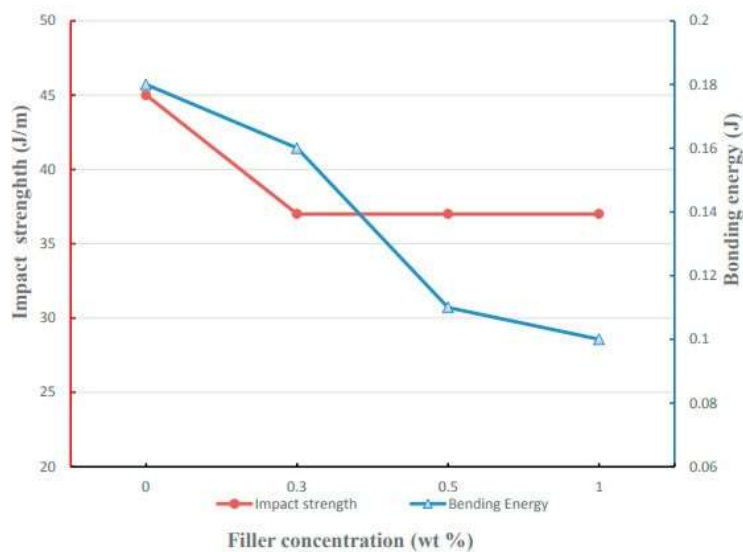


Fig. 3. Impact properties of PAEK/CNT nanocomposite.

Table 4 clearly depicts flexural properties (strength and modulus) increases first and then decreases up to some amount with increasing percentage of MWNT. Same trend is observed for tensile properties also. The addition of MWNT increases the flexural modulus of neat PAEK from 3600 to 3800, improved approximately equal to 6 % and at the same time its flexural strength from 172 to 179, improved approximately equal to 4 %. Fig.4 shows data for PAEK/MWNT nanocomposite specimens tested in flexural. Fig.4 (a) shows the relation between wt% of MWNT and flexural strength. Fig.4 (b) shows the relation between wt% of MWNT and flexural modulus. 0 wt% shows highest load bearing capacity and while testing in bending mode specimens doesn't break till 12 mm elongation. Addition of MWNT reduces its load bearing capacity and increases brittle nature in material. PAEK/MWNT 1 wt% shows lowest flexural properties among all composites, with increasing MWNT wt% it only deteriorates flexural properties [8]. It means that MWNT are not dispersed uniformly and needed some modifier.

Table 4. Flexural properties of PAEK and PAEK/MWNT nanocomposites

Composition	Flexural Strength (MPa)	Flexural Modulus (MPa)
PCNT0	172	3600
PCNT0.3	178	3800
PCNT0.5	179	3743
PCNT1	178	3800

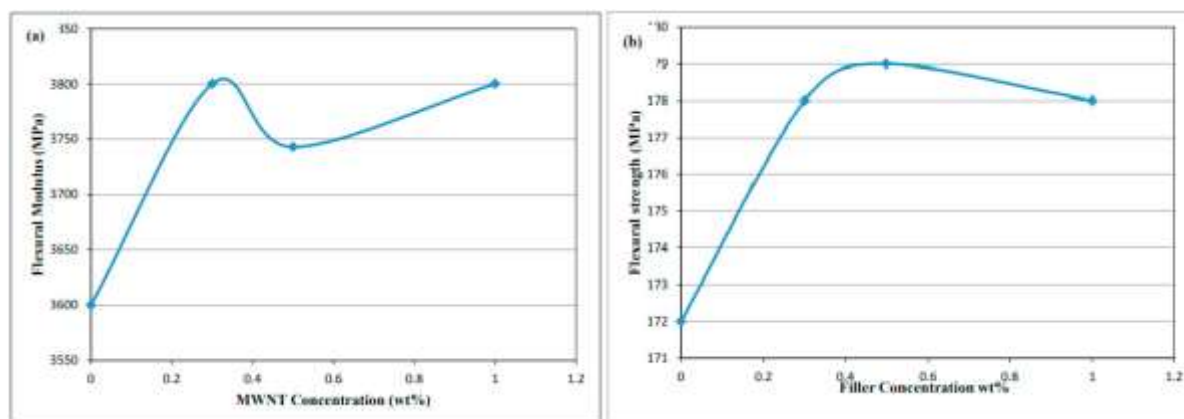


Fig. 4. (a) Flexural properties of PAEK/MWNT nanocomposites flexural strength; (b) flexural modulus by varying filler content

5. Conclusions

A new class of innovative materials (nanocomposites) with superior mechanical, thermal, electrical, and barrier properties is produced using polymer matrices based on multiwall carbon nanotubes (MWNTs), which can replace many conventional materials in engineering applications. Since MWNT has the highest basal plane elastic modulus, it is anticipated to be the best high strength filler. The mechanical characteristics of PAEK polymer tend to dramatically increase when MWNT is added. At 1 wt% MWNT, tensile strength increases by about 27%, elongation drops by about 60%, impact strength practically stays constant, flexural strength declines by about 4%, and flexural modulus decreases by about 5%. This can be attributable to two main factors. The first is that MWNT has a substantially higher elastic modulus than steel, which makes them extremely resistant. The second factor was inadequate matrix-filler adhesion since the majority of the load is often handled by stiff filler. However, MWNT has a significant beneficial impact on storage modulus, increasing it by around 37, 27, and 24% at 0.3, 0.5, and 1 wt%, respectively, compared to neat PAEK at 33°C.

References

- [1] J. P. Agrawal, "Composite materials", Popular science (1990), pp. 01-08.
- [2] Kurtz S.M., and Devine J.N., "Review: PEEK biomaterials in trauma, orthopaedic, and spinal implants", Biomaterials 28 (2007), pp. 4845–4869.
- [3] Gharda chem., "Ultra High Performance Polymer", Revision 2010.
- [4] Lisa M. Hall, Arthi Jayaraman et al., "Molecular theories of polymer nanocomposites", Current Opinion in Solid State and Materials Science 14 (2010), pp. 38–48.
- [5] Changru Rong, Gang Ma et al., "Effect of carbon nanotubes on the mechanical properties and crystallization behaviour of poly (ether ether ketone)", Composites Science and Technology (2010), pp. 380–386.
- [6] Toshio Ogasawara, Terumasa Tsuda et al., "Stress–strain behaviour of multi-walled carbon nanotube/PEEK composites", Composites Science and Technology (2011), pp. 73–78.
- [7] Chong-Tao Han et al., "Mechanical properties and bioactivity of high-performance poly (etheretherketone)/carbon nanotubes/bioactive glass biomaterials", J Polymers (2013), pp. 20-203
- [8] D.R. Paul, and L.M. Robeson, "Polymer nanotechnology: Nanocomposites", Polymer 49 (2008), pp. 3187-3204.