# Mechanical and thermal properties of PEEK/ Polyphosphazene coated MWCNT

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***Abstract-*** *In the present investigation and effort has been made to prepare nanocomposite based on Polyether ether ketone (PEEK) and also to study the effect of polyphosphazene coated multiwall carbon nanotubes (MWCNT) on the mechanical and thermal properties. Nano composite has been prepared by melt mixing process with the help of co-rotating twin screw extruder. The results of mechanical properties such as tensile strength, flexural strength, impact and hardness have remarkable increased with the incorporation of MWCNT's in the PEEK matrix. The enhancement in mechanical properties may be attributed to excellent interfacial adhesion between reinforcement and the polymer matrix. Heat deflection temperature (HDT) has increased with the addition of Polyphosphazene coated MWCNT in to matrix.*

***Keywords-*** MWCNT, Polyphosphazene, Nanocomposite, Mechanical Properties, HDT.

## I. INTRODUCTION

Single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) have been extensively investigated because of their special mechanical properties as well as their excellent thermal and electrical properties [1-6], since the discovery of carbon nanotubes by Iijima [7]. In the present scenerio, it is worth believing that SWCNTs, MWCNTs, nanoclay support nanotubes [8] and carbon nanofillers (CNFs) can be used as fillers in polymer matrix providing a noble path to composites with several enhanced properties [9-18]. The influence of this incorporation on mechanical, thermal and electrical properties have been studied for many kinds of polymers such as poly ether ether ketone (PEEK) [19], polystyrene (PS) [18], Poly methyl methyl acrylate (PMMA) [21, 22], polypropylene (PP) [23] etc. These studies have demonstrated a drastic enhancement in tensile strength, tensile modulus, thermal and electrical conductivity.

PEEK is an high performance engineering polymer having a high glass transition and melt temperature, excellent mechanical properties and good solvent and abrasion resistance, Sandle et al. [24], studied CNFs reinforced PEEK composites with improved mechanical properties. The addition of CNFs also improve the elongation flow properties in the PEEK matrix [25]. Mishra et al. have studied PEEK composites reinforced with zirconia nanofillers [26]. However, this high performance thermoplastics would not satisfy all the mechanical and thermal requirement in pristin form, therefore, it should be reinforced with various reinforcing agent such as inorganic nano particles/ MWCNTs or different type of fibers in order to become suitable for mechanical and thermal applications. It is well established fact that a good dispersion state of MWCNTs in polymer matrix and a strong interfacial bonding between MWCNTs and the polymer matrix could significantly enhance the mechanical and thermal properties.

In the present study, an effort has been made to modify MWCNTs by polyphosphazene to obtain satisfy dispersion of MWCNTs in PEEK matrix and strong interfacial bonding between MWCNTs in PEEK. The influence of loading of modified MWCNTs in PEEK matrix has also been investigated. The interaction between MWCNTs and polyphosphazene have been characterize fully. The significantly improved mechanical and thermal properties have been correlated to the largely enhanced interfacial bonding between MWCNTs and PEEK, and remarkably enhanced dispersion of MWCNTs in PEEK matrix.

## II. MATERIALS AND METHODS

### 2.1. Materials

PEEK (grade Ketaspire 820 NT) purchased from solvay chemicals alpheretta(USA) is used as metrix materials. The multiwalled carbon nano tubes (MWCNT) and polyphosphazene (PPH) were synthesises by DMSRDE , Kanpur. MWCNT having a diameter 2-4 nm nad 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 PPH 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 $90^0$C then they obtained residues is heated up to $25^0$C and then leave it to cool as a result we get PPH coated MWCNT.

### 2.3 Dispersion of inorganic polymer coated MWCNT'$_S$

Before incorporation of PPH 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 polyphosphazene coated MWCNT's at in order to induce an efficient dispersion of nanotubes.

### 2.4 Preparation of organic/inorganic polymer Nanocomposite

Before mixing, the organic polymer (PEEK) was dried under vacuum at $80^0$C for at least 12 hrs. Then after organic polymer and inorganic polymer with nanoclay (polyphosphazene coated MWCNT) were blended in twin screw extruder ( at RPM 144) at temperature processing condition given in Table 2.1 for PEEK blend with inorganic polymer as per formulation Table 2.2. Testing specimens were prepared using injection moulding machine at temperature 340°C for PEEK & PEEK/PEI blend with inorganic polymer / nanoclay.

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

| Heating Zone | PEEK |
|---|---|
| Zone 1 | 340 °C |
| Zone 2 | 366 °C |
| Zone 3 | 385°C |
| Zone 4 | 386°C |
| Zone 5 | 390°C |
| Die | 398°C |

Table – 2.2 Formulation of the nanocomposite based on PEEK/PPH coated MWCNT blend

| Sr. No. | PEEK (gm) | PPH coated MWCNT (%) |
|---|---|---|
| 1. | 500 | 0.1 |
| 2. | 500 | 0.3 |
| 3. | 500 | 0.5 |

### 2.5 Testing and characterization

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 ASTMD 792, Tensile properties were determined using dumbbell shaped specimen as per ASTMD 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 PEEK/ polyphosphazene 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.

## III. RESULTS AND DISCUSSION

### 3.1 Mechanical and Thermal Properties

The variation of hardness were increased with the addition of filler , which is at 0.3% and has shown in Table 3.1. Tensile strength, tensile modulus and elongation % value have mentioned in Table 3.2, It is clearly found that tensile properties are increased with the addition of polyphosphazene coated MWCNT up to 0.3 wt% of nano filler in to the PEEK. Flexural modulus (shown in Table 3.3) increased at 3 wt % loading as compared to virgin PEEK. Impact strength also increased as other Tensile and flexural properties increased. The thermal properties HDT also increasing with the addition of filler in to matrix. The increased in mechanical properties in depends on the dispersion of volume fraction of filler and interaction of PEEK and nanofiller interaction. The increase in properties is mainly based on better interface, which transfer the stress from one phase to another phase.

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

| Composition | | Density, g/cc (PEEK density – 1.28 g/cc) | Rockwell Hardness, (M Scale) (PEEK Hardness – 96) |
|---|---|---|---|
| PEEK, gm | Filler % | PPH coated MWCNT | |
| 500 | 0.1 | 1.29 | 100 |
| 500 | 0.3 | 1.29 | 102 |
| 500 | 0.5 | 1.3 | 98 |

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

| Composition | | Tensile strength, MPa (PEEK Tensile Strength – 93.42 MPa) | Tensile Elongation, % (PEEK Tensile Elongation – 5.6%) | Tensile Modulus, MPa (PEEK Tensile Modulus – 3498 MPa) |
|---|---|---|---|---|
| PEEK, gm | Filler % | PPH coated MWCNT | | |
| 500 | 0.1 | 102.98 | 5.4 | 3793.28 |
| 500 | 0.3 | 107.18 | 5.3 | 3957.45 |
| 500 | 0.5 | 104.89 | 5.5 | 3879.26 |

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

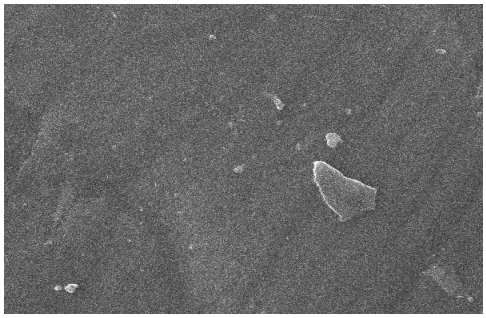
| Composition | | Flexural strength, MPa (PEEK Flexural Strength – 144.82 MPa) | Flexural Modulus, MPa (PEEK Flexural Modulus – 3698.42 MPa) |
|---|---|---|---|
| PEEK, gm | Filler % | PPH coated MWCNT | |
| 500 | 0.1 | 152.14 | 3789.43 |
| 500 | 0.3 | 159.24 | 3891.56 |
| 500 | 0.5 | 156.18 | 3816.42 |

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

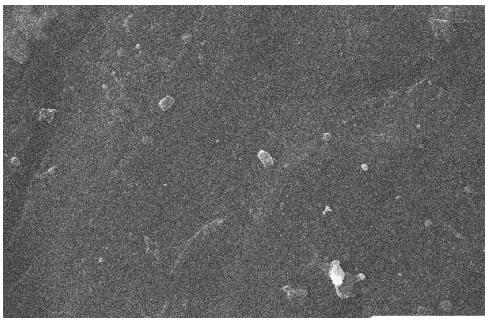
| Composition | | Izod Impact, J/m (PEEK Izod Impact – 90.2 J/m) | Heat Deflection Temperature (HDT), Deg C (PEEK HDT – 156.3 Deg C) |
|---|---|---|---|
| PEEK, gm | Filler % | PPH coated MWCNT | PPH coated MWCNT |
| 500 | 0.1 | 96 | 158.6 |
| 500 | 0.3 | 100 | 164.2 |
| 500 | 0.5 | 97 | 163.9 |

### 3.2 Morphological properties

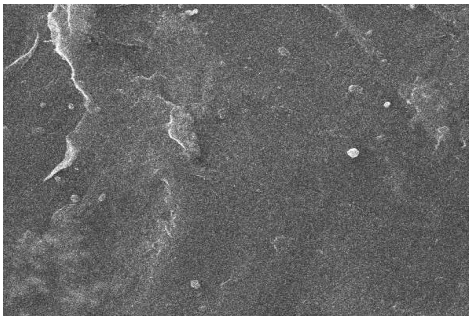
SEM micrographs were shown in Figure 3.1 (a, b, c, d) for the virgin PEEK 0.1, 0.3 & 0.5% PPH coated MWCNT fillers in PEEK matrix. The scanning micrograph shows the homogeneously dispersion of filler in to polymer matrix. It is due to the coating of PPH in to MWCNT due to this elastomeric phase improved the dispersion in to polymer matrix.



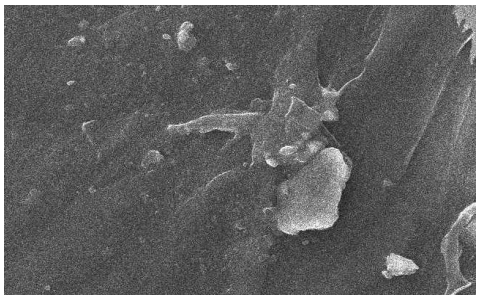
a) PEEK virgin



b) 0.1% filler in PEEK



c) 0.3% filler in PEEK



d) 0.5% filler in PEEK

Fig 3.1 Morphological properties of PEEK/ PPH coated MWCNT

## IV. CONCLUSIONS

Various compositing of PEEK/ polyphosphazene coated MWCNT were developed with twin screw extruder and properties like mechanical, thermal and morphological were

studied. Mechanical and thermal properties of the nanocomposites were increased with the increasing the loading of nano clay in to polymer matrix up to 0.3 %. This was also confirmed through SEM analysis. The thermal properties also shown the significant improvement with the incorporation of polyphosphazene coated MWCNT due to better interface and dispersion of nano clay.

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