Development and Investigation of Mechanical Properties of PEEK Fine Particles Reinforced UHMWPE Composites

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Abstract
In the present work, novel Ultra-high molecular weight polyethylene (UHMWPE) composites reinforced with 3wt. %, 6wt. %, 9wt. %, 12wt. % and 15wt. % PEEK particles are fabricated by using hot compression moulding technique and mechanical properties are investigated. Experiments were conducted as per ASTM standards to evaluate the properties of tensile, flexural, impact, and microhardness. The results have revealed that the addition of PEEK to the UHMWPE reduced tensile strength and elongation at break, whereas the Young’s modulus of the composites was increased. The composite reinforced with 12wt. % PEEK has better flexural properties and greater impact strength when compared to other composites. The hardness of the UHMWPE composites was increased based on PEEK content. The morphologies of the fractured surface are analyzed under field emission scanning electron microscope. The results have indicated poor interfacial bonding between PEEK and UHMWPE.

Keywords: Polymer matrix composites, UHMWPE, PEEK, Mechanical properties, SEM.

Introduction
Ultra-high molecular weight polyethylene (UHMWPE) is an excellent polymer which possesses the highest molecular weight, with excellent properties such as high wear resistance, high impact strength, self-lubricating properties, low coefficient of friction, and nontoxic. Because of these excellent properties, UHMWPE has been widely used in acetabular components since Charmley recommended it for Total Hip Replacement (THR) [1]. However, its load carrying capacity is restricted because of its low hardness and young’s modulus, stiffness and easy creep under load [2] when compared to other engineering plastics such as PEEK. Therefore, the modification of UHMWPE to enhance its mechanical properties is currently a trending research topic. Cao et al. studied the mechanical and tribological performance of Basalt fibers filled UHMWPE composites [3]. Liu and Sinha studied the wear and compressive properties of PEEK filled UHMWPE [4]. Boon Peng Chang et al. studied the effects of Zeolite particles on the mechanical and tribological properties of UHMWPE composites [5] and many other studies have used different reinforcements merged in the UHMWPE matrix in order to enhance its mechanical and wear performance. This includes the addition of carbon fibre, hydroxyapatite, kaolin, Zirconium and others [6 – 9].

In literature, many studies have used various reinforcements incorporated into the UHMWPE matrix in order to enhance its mechanical properties and wear performance. This indicates that both the particulate and fiber reinforcement can improve the performance of UHMWPE composites and there has been an increasing number of studies reported on improving the performance of UHMWPE composites using both particle and fiber fillers. However, there has been very little research work done to enhance the performance of UHMWPE composites using organic PEEK hard particles. Several investigations have been carried out on the mechanical strength and wear properties of PEEK and its composites filled with fibers, and organic and inorganic fillers [10-11]. PEEK is one of the most important engineering plastics with outstanding performance in high mechanical properties such as strength, modulus, toughness, resistance to creep, abrasion and fatigue, high temperature resistance, good resistance to aggressive solvents, favourable processing capability, high wear resistance, self-lubrication etc. [12]. Therefore, the main objective of the research work is to develop novel polymer matrix composites using Ultra high molecular weight polyethylene as the matrix material with polyether ether ketone (PEEK) particles as the reinforcement material by varying weight percentage using compression molding technique. The different mechanical testings namely, tensile, flexural, impact and microhardness were conducted on the standard samples prepared. As a final point, the fracture surface of PEEK/UHMWPE composites is observed using SEM analysis.

Experimental details
Materials
Compression molding grade UHMWPE (Relene UHM-2504) resin in powder form is used as matrix material and vestakeep 2000FP Poly Ether Ether Ketone fine powder is used as reinforcement material. UHMWPE was procured from Reliance Industries Ltd and vestakeep PEEK FP2000 was procured from Mumbai. The properties of UHMWPE resin and PEEK are shown in Table 1. Figure 1 shows the microscope image of compression moulded UHMWPE composite reinforced with 12 wt. % PEEK particles.
Mechanical properties
Tensile properties are estimated using computerized universal testing machine on tensile test specimens with a gauge length of 50mm and thickness of 3mm with a crosshead speed of 25mm/min performed at ambient temperature. Flexural properties of all composite samples are measured in the same UTM machine. In the three point loading system, the load is applied at a crosshead speed of 2mm/min and a span of 63mm maintained for all specimens. The size of the Izod impact test specimen is 65 x 13 x 3mm³. The impact strength Iₜ, expressed in KJ/m², is calculated using the following equation:

\[ Iₜ = \frac{E_I}{A} \text{ KJ/m}² \]

where \( E_I \) = Impact energy recorded in Kilo joules
\( A = \) Area of the sample in m²

In each group, two samples are tested, and the average value is reported. The microhardness of composites is measured using MITUTOYO - HM113 Vickers microhardness tester (Japan). A load of 0.50Kg for a time of 10s is applied. A minimum of five measurements are collected to calculate the microhardness value for each specimen.

Phase and fracture surface analysis
The Fourier transform infrared spectra of the PEEK/UHMWPE composites are recorded on a FTIR spectrophotometer. Scanning electron microscopy (SEM) is carried out to study the fractured surface of the composites after tensile test. Before scanning, the fractured surface of the composites are coated with gold/palladium (Au-Pd) using a vacuum sputter chamber.

Results and Discussion
The experimental results of mechanical tests are exemplified in Figures 3-9. These figures depict the mechanical properties in relationship with the filler content in wt. %.

Tensile properties
As per the standard ASTM D638, the tensile test is carried out using a computerized universal testing machine as shown in Figure 2 and the specimens after tensile test are shown in Figure 3. The tensile strength, young’s modulus and % elongation values of the PEEK/UHMWPE composites are shown in Figures 4, 5 and 6. In general, the ultimate tensile strength of the UHMWPE (As per manufacturers data UTS =29.37 MPa, indicating the highest value also due to the interruption in continuous phase of polymeric matrix with the increase in filler loading [13].

![Figure 1: Microscope image of UHMWPE composite](image)

### Table 1: Properties of UHMWPE and PEEK

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Properties</th>
<th>UHMWPE</th>
<th>PEEK</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Specific gravity</td>
<td>0.933 g/cm³</td>
<td>1.3 g/cm³</td>
</tr>
<tr>
<td>2</td>
<td>Glass transition temp</td>
<td>-110°C</td>
<td>143°C</td>
</tr>
<tr>
<td>3</td>
<td>Melting point</td>
<td>200 - 220°C</td>
<td>340°C</td>
</tr>
<tr>
<td>4</td>
<td>Tensile strength</td>
<td>48MPa</td>
<td>90-100MPa</td>
</tr>
<tr>
<td>5</td>
<td>Young’s modulus</td>
<td>0.69GPa</td>
<td>3.7GPa</td>
</tr>
<tr>
<td>6</td>
<td>Operating temp</td>
<td>&lt;100°C</td>
<td>250°C</td>
</tr>
</tbody>
</table>

### Composite preparation
The UHMWPE / PEEK composite designations are tabulated in Table 2. As per the table 2, Vestakeep PEEK particles are added to the UHMWPE powder at different loadings and mixed for 1 hour by a rotary ball mill at 300rpm to obtain uniform mixing. Before mixing, the container and balls are thoroughly cleaned using ethanol. The final resultant mixture of PEEK / UHMWPE powder is cold compressed in a compression molding machine for 10 minutes under a pressure of 100Kg/cm² to expel the air. The temperature is raised to 200°C to melt the UHMWPE powder and held constant for 1 hour.

<table>
<thead>
<tr>
<th>Designation</th>
<th>PEEK (wt. %)</th>
<th>UHMWPE (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3</td>
<td>97</td>
</tr>
<tr>
<td>2</td>
<td>6</td>
<td>94</td>
</tr>
<tr>
<td>3</td>
<td>9</td>
<td>91</td>
</tr>
<tr>
<td>4</td>
<td>12</td>
<td>88</td>
</tr>
<tr>
<td>5</td>
<td>15</td>
<td>85</td>
</tr>
</tbody>
</table>

After that 50Kg/cm² pressure is maintained until the total mass comes back to the room temperature. The test specimens are cut to suitable dimensions from the molded composite sheets and used for mechanical tests.

Table 2: Designation of UHMWPE composites
Sole and Ball [14] stated that rigid fillers reveal deleterious effect on tensile property and ductility. In general, UHMWPE has relatively low young modulus and yield strength [15]. However, from Figure 5, Young’s modulus of composites increased with increase in hard particles reinforcement. This is due to the enhancement in stiffness of the composite specimens with the addition of PEEK reinforcement which is having higher stiffness than the weak resin matrix [16-17]. Young’s modulus of UHMWPE composites is in the range of 0.768 – 1.098 GPa and the designation 5 (15 wt. %) has the maximum of Young’s modulus of 1.098 GPa.

This behaviour may possibly lead to the conclusion that tensile modulus may depend on the filler content rather than reinforcement-matrix adhesion and also higher stiffness of the reinforcing particles than the matrix material. Owing to this, overall stiffness of the composite specimens is increased and thus tensile modulus enhanced. From Figure 6, % elongation of UHMWPE composites decreases with increase in hard particles reinforcement. The decrease in % elongation at break is due to the interruption of the chain mobility of the UHMWPE by the PEEK reinforcement; the plastic deformation of the composites is also decreased. It can be seen that the % elongation decreases rapidly from Designation 1 to 2 and then slowly decreases with an increase in wt. % of PEEK particles.

**Figure 2:** Tensile testing machine

**Figure 3:** UHMWPE composite samples after testing

**Figure 4:** Tensile strength of UHMWPE Composites

**Figure 5:** Young’s modulus of UHMWPE composites

**Figure 6:** % Elongation of UHMWPE composites

**Flexural properties**

The experimental results of flexural properties of UHMWPE composites are displayed in Figures 7 and 8. As per the standard ASTM D790, the flexural test is carried out using the same UTM as shown in Figure 2. No breakage of samples observed during the test. Figure 7 shows a steady increase in flexural strength with increase in filler content upto 12wt. %.
By further increasing the PEEK content for 15wt. % flexural strength value decreases. Flexural strength of UHMWPE composites is in the range of 24.26 – 29.56GPa and the designation 4 (12wt. %) has the maximum of flexural strength of 29.56 MPa. The possibility of raise in flexural strength at 12 wt. % PEEK content for UHMWPE/PEEK blends might be due to less entanglement between polymer chains as the amount of UHMWPE is reduced. Therefore, the restriction of chain movement is reduced and a higher stress is needed to allow the slippage of polymer chains before the rupture happens.

From Figure 8, Flexural modulus of composites increases with increase in hard particles reinforcement. It is well known that the modulus of a reinforced system depends on the properties of the reinforcement and matrix; the modulus of PEEK is higher than that of the UHMWPE matrix, and the flexural moduli of reinforced composites are higher than that of UHMWPE due to higher loading of PEEK. Flexural modulus of UHMWPE composites is in the range of 1236.64 – 1373.59MPa and the designation 5 (15 wt. %) has the maximum of flexural modulus of 1373.59MPa.

The graph shows a steady decrease in impact strength with increase in PEEK content upto 9 wt. %. However, there is an increase in impact strength for further increase in filler content (12 wt. %). This is possibly attributed to the fact that the reinforcing particles absorb more potential energy as their content increases in the matrix material. This may be attributed to the continuity of the UHMWPE matrix which when broken produces defaults at the interfaces. Conversely on further filler loading to 15 wt. % there is a decrease in impact strength. This may be due to the weak interfacial interaction between the filler material and matrix for higher filler content that is beyond 15 wt. %. In their study Cao et al. [3] reported that the impact strength of the UHMWPE composites significantly decreased as the weight percentage of the fibre was increased. Similar behaviour of the composite specimens was also observed by Haque [18] that is impact strength decreases for higher filler loading.

Impact strength
The experimental results of impact strength of PEEK reinforced UHMWPE composites are displayed in Figure 9. The graph shows a steady increase in microhardness with increase in filler content, indicating 9.75HV with 15 wt. % of PEEK addition. The main reason is that the strength and hardness of the PEEK is much higher than that of the UHMWPE. When the UHMWPE composites are compressed, the load is mostly supported initially by the reinforce material PEEK. So, the hardness of the UHMWPE composites is increased. From the results, it is found that the hardness value is in proportion to the amount of PEEK particles added. Yemei Liu et al. [4] also stated that the incorporation of PEEK powder into UHMWPE has shown better surface hardness. But, if the filler content is increased too high, the molecular continuity of the UHMWPE resin is broken and the defaults like the micro-fractures increased. So, the strength and hardness of the UHMWPE composites is no longer increased or even decreased with the increase of the filler content [3].
Fourier transform infrared analysis of the UHMWPE composites is acquired on a Nicolet iS10 (Thermo scientific) spectrometer using 2.5 – 25 mm wavelength radiation. The spectral region is spanned from 4000 to 500 cm⁻¹. Various characteristic bands 1462.06 cm⁻¹, 2922 cm⁻¹ and 2850 cm⁻¹ are present in FT-IR spectra of UHMWPE and the band at 1472.3 cm⁻¹ corresponding to aromatic ring structure confirms the presence of PEEK in the composite as shown in Figure 11. From the spectra absence of any degradation is observed.

Fracture surface analysis

Naturally, the polymer composites are non-conductive. Therefore, the test sample’s fractured surface is coated with gold/palladium (Au-Pd) layer to increase the conductivity of the material. Figures 12(a) and 12(b) show the SEM images of fractured cross-section of 9wt. % and 12wt. % UHMWPE composites. UHMWPE has smooth, long carbon backbone molecules, while PEEK has bulky molecules which result in difficulties in chain to chain movement. From Figures 12(a) and 12(b), it is observed that with PEEK inclusions more interfacial spots are formed between the UHMWPE matrix and PEEK particles, which can act as initiation points for the material to deform or fracture. This indicates that the tensile strength decreases with an increase in the concentration of PEEK particles. When the UHMWPE is reinforced with PEEK, the continuity of chain mobility is disrupted resulting in difficulties in chain to chain movement with the defaults forming weak points. These can be the initiation points for the materials to form and grow micro cracks which will result in deformation or fracture. This results in increased brittleness of the UHMWPE composites. It is observed that the main fracture mechanism occurred through de-bonding of PEEK from the UHMWPE matrix, suggesting a poor interfacial interaction of PEEK with UHMWPE.

Conclusion

Experimental investigation of tensile properties, flexural properties, impact strength and hardness of compression molded UHMWPE composites reinforced with different weight percentage of PEEK were carried out as per ASTM standard. The following conclusions were drawn based on the present work:

- The tensile strength and elongation at the break of UHMWPE composites was found to decrease with the addition of PEEK loadings, while the Young’s modulus was observed to increase.
- The 12 wt. % PEEK reinforced composites exhibited the maximum flexural and impact strength.
- The hardness of the composites improves with an increase in filler content of PEEK. The hardness of the UHMWPE composites was increased proportionally to the PEEK loading.
- The SEM images show poor interfacial bonding between PEEK and UHMWPE, with cup like depressions. This indicates that interface failure is the reason for the failure of the composite.

References


