Tribological investigation of peek composite reinforced with nanometre sized potassium titanate powder

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Abstract. Tribological behaviour of poly ether ether ketone (PEEK) reinforced with (0-15 wt%) nanometre sized potassium titanate powder has been investigated on the pin on disk configuration. It has been found that the tribological properties of PEEK composite get enhanced with the increase in concentration of potassium titanate content. Tribological studies of the PEEK polymer-matrix composite shows that it forms a thin transfer film which impact the wear behaviour by reducing the coefficient of friction and wear rate. The worn surface is characterized using optical microscope and scanning electron microscope for analyzing the surface morphology. X-ray diffraction pattern and Fourier-transform infrared spectroscopy of the potassium titanate were also observed. The hardness value of the PEEK composite was obtained by durometer hardness test which found to be improved with concentration of potassium titanate. Moreover, the composite resulted in reduced wear rate and coefficient of friction with the addition of potassium titanate to the PEEK.

Keywords: Poly ether ether ketone, wear, transfer film, surface morphology, durometer hardness.

1. Introduction

Every mechanical system depends on the tribological behaviour, this friction property determines the product life cycle and the quality. Even a single step taken to reduce the wear behaviour of the material has a marked effect on the society. Polymeric materials are found to be the effective replacement of conventional materials (e.g. Aluminium, carbon steel, stainless steel) and various research have been carried to determine the most suited high-performance plastics. Most demanding applications like bearing, bushings, valves and sealings require high performance plastics. Such high performance plastics should be able to withstand higher temperature and higher loads for long operating hours. Poly Ether Ether Ketone (PEEK) is a new generation thermoplastic material which is semi crystalline in nature that offers the possibility of use at high service temperature [1]. PEEK has lower water absorption capacity than epoxies and they are highly preferred for matrix material as it possesses some excellent mechanical and thermal properties [2].

Even though PEEK possesses very good tribological property when compared with other polymers, its wear behaviour is somewhat high for pure peek material as its coefficient of friction is quite high. Addition of nano fillers particles with raw PEEK helps in reducing the wear rate and also the coefficient of friction under various sliding conditions[3]. Hence some weighted percentage of reinforcement material is added to raw peek to enhance its tribological properties. Solid lubrication materials have been used over a decade for achieving better tribological properties and these materials proved to have excellent synergistic effect when added with the polymers. Potassium titanate is also a self-lubricating material which provides a low material wear[4]. Potassium titanate is soft inorganic
material which improves strength and wear resistance of the material. Potassium titanate have a high melting point and excellent frictional stability which are some added values[5]. Wear behaviour of the potassium titanate compounds provide a gentle value which make them suitable for filler materials[6]. This self-lubricating material is known for their low friction property and wear resistance but they are not used extensively used in combination with PEEK polymer material.

Therefore, in this study, nano sized potassium titanate is reinforced with PEEK at different filler concentration. The present study is to investigate the tribological properties and mechanical properties of PEEK reinforced with the potassium titanate particles under dry sliding conditions at room temperature. At identical test condition unfilled PEEK were also investigated on friction and wear properties for comparison. Several characterization techniques were carried out for better understanding of the various properties. This study will provide guidance on understanding the effect of wear performance of PEEK and the function of PEEK filled with nano sized potassium titanate.

2. Experimental

2.1 Materials
For this investigation, we used commercially available Poly Ether Ether Ketone with average particle size of 50µm as the matrix material for composite preparation. The microsized potassium titanate powder has obtained from Travancore titanium products was used as filler material.

2.2 Sample preparation.
PEEK polymer matrix was blended with potassium titanate filler at a proportion of 0-15wt% which was clearly represented in the table.1 and they were ball milled for a duration of over 30minutes to get blended with each other. The samples were prepared under powder metallurgy process by compacting and sintering. The blended powder samples were compacted at room temperature by pressing at a pressure of 100psi with a dwell time of 180 seconds. The sample has been prepared in the shape of cylindrical pellets for a dimension of (10mm×10mm) diameter and height. Thus the green compact prepared by pressing was sintered initially to a temperature of 150 degree Celsius at a duration of 30 minutes and a dwell time 15minutes is maintained to prevent oxidation and then at the next 45minutes a temperature 310 degree Celsius had been reached and dwell time of 45minutes was maintained for developing a bond between the powder particles which was represented in the Figure.1. Then they were dried to room temperature and the finished composite sample had obtained.

Figure 1. Sintering cycle
2.3. Tribological test
The tribological tests were carried out on pin on disc tribometer at dry sliding conditions. The test was conducted for duration of 30 minutes at a constant sliding speed of 0.5 m/s for a varying load between 10-30N which was enough to ensure a steady state coefficient of friction of the tested sample. The test was conducted at controlled humidity and at laboratory environment, without any fluid lubricant. Before installing the specimen on the pin on disc apparatus for each test, the flat ended polymer pins and the steel discs were cleaned with cotton dipped in ethanol. For every test condition, the applied load was noted and also the mass loss was calculated to determine the specific wear rate. On each sample the tests were performed to ensure a relevant evaluation and presented values represent the steady state coefficient of friction for each sample.

Table.1. Composition of PEEK and Potassium Titanate

| Serial no | PEEK | Potassium Titanate |
|-----------|------|--------------------|
| 1         | 100  | -                  |
| 2         | 95   | 5                  |
| 3         | 90   | 10                 |
| 4         | 85   | 15                 |

2.4. Characterization and analysis
On foregoing to tribological testing, all the powder samples i.e., both the matrix and filler were characterized using X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) for fetching necessary data about the samples for confirming their authentic nature. The potassium titanate filler was also characterized using Laser Scattering Particle Size Distribution Analyzer LA-950 to ascertain the size of the particle. The shore hardness of all the samples were measured on durometer instrument using shore D type indenters at a loading force 4536 gram for an indentation loading time of 10 seconds. The average of three measurements for every single composition is presented. The density of PEEK filled samples was measured by using Archimedes principle using water as the immersing medium. The surface topography of the wear scars and wear debris were observed with optical microscopy and scanning electron microscopy.

3. Results

3.1. Particle size values
As through the literature surveys the particle size of filler materials have direct impact in enhancing the tribological performance and so here the potassium titanate which we used as the reinforcing agent has been ball milled for a duration 120 minutes at a speed of 400 rpm using tungsten carbide balls. The particle size of the filler material was analyzed using Laser Scattering Particle Size Distribution Analyzer. The size of filler material as received and after the ball milling were analyzed. The mean particle size of filler material after ball milling was reduced to 80 nm which initially was in the average size of 1.10 µm. The resultant values shown in Figure.2 are found to be nominal which was intent to enhance the tribological performance.

3.2. X-ray diffraction analysis
A crystallographic study was conducted on the potassium titanate powder using X-ray diffractometer. The crystallographic structure was determined by XRD pattern. The distance between peaks were calculated for comparing the data with the ASTM XRD diffraction data cards to verify the crystal structure. The peaks 310 and 311 indicates the presence of potassium (K) and TiO2 which have good similar matches with standard pattern. The resultant XRD pattern of the potassium titanate and the
calculated spacing distance between the peaks was found to be similar with the ASTM data. The resultant XRD of potassium titanate is graphically represented in figure.3.

3.3 Fourier-transform infrared spectroscopy
Fourier-transform infrared spectroscopy was used for identifying the organic component present in the potassium titanate to determine its authentic nature. The resultant characteristic IR spectra confirms with the spectroscopic signature of potassium titanate by showing very similar absorption bands. The resultant IR spectra shows relatively similar peaks between the band region of (1400-800) cm$^{-1}$ which confirms the authentic nature of potassium titanate. The obtained resultant IR spectra is graphically represented in Figure 4.

![Figure 2. Average particle size of potassium titanate before ball milling and after ball milling](image1)

![Figure 3. XRD pattern of potassium titanate](image2)
3.4. Hardness of PEEK composite

Figure 5 shows the shore hardness value of PEEK composite with potassium titanate as the filler material. It is seen that the highest hardness values are obtained for 15wt% potassium titanate, the composite with 5wt% of filler material shows the hardness values which slightly resembles close to plain PEEK values. However, the resultant values exhibit that the hardness of the composite increases with addition of the potassium titanate to PEEK.

3.5. Friction and wear

The concentration of added potassium titanate influenced the wear rate of the PEEK and it’s composite. Among all the tested samples reduced wear behaviour was observed for 15wt% concentration of the filler material. Pure PEEK exhibited highest coefficient of friction which reached
the values of 0.4. Interestingly, the lowest coefficient of friction was also obtained at a concentration of 5wt% and 10wt% with slighter difference. When compared with pure PEEK, the coefficient of friction showed a decreasing trend with the increase in addition of potassium titanate as represented in figure 6. The effect of increasing the load showed a modest increase in the value of coefficient of friction but still the values are lower compared with pure PEEK composite. This indicates that nanometre sized potassium titanate is more effective at improving the friction reduction ability of the composite at a higher load.

![Figure 6](image)

**Figure 6.** Coefficient of friction values corresponding to the varying loads (a) pure PEEK (b) 5wt% potassium titanate filled PEEK (c) 10 wt% potassium titanate filled PEEK (d) 15wt% potassium titanate filled PEEK.

### 3.6 Analysis of worn surface

Worn surface of the pure PEEK and its polymer composite were investigated by using optical microscopy for characterizing the wear mechanism and to quantify the wear model. The worn surface
of the pure PEEK shows wide scratches and the wide scratches will lead to the deterioration of the material by causing removal of material from the surface is shown in figure 7. The wide scratches depend on amount of material transferred to the counterpart and adhered back to polymer surface. Wear scar show parallel grooves caused by ploughing of material from the surface. By observing the worn surface of the polymers of different concentration the wear scars got reduced with increase in the concentration of potassium titanate.

![Image](image1)

**Figure 7.** Analysis of worn surface using optical microscopy:
(a), (b) Abrasion wear marks across the surface
(c), (d), (e) Peak and valleys caused by ploughing of material in the worn surface
(f) Shows wear debris on the surface

The SEM observation of the worn surface after wear testing is shown in the figure 8. It can be observed that the surface of the wear scar is basically smooth with the little traces of wear and the distribution of the particles was also observed in the SEM micrographs. The worn surface is smooth with small debris particles distributed over the wear region. It clearly reveals the traces of wear caused by abrasion and plastic deformation over the surface. The sample with lower content of potassium titanate showed distinct traces of wear parallel to sliding direction and with increase in the concentration of potassium titanate a smoother surface with reduced marks are observed.

![Image](image2)
Figure 8. Analysis of worn surface using scanning electron microscope (a, b) shows the distribution of particles (c, d) shows smooth worn surface with wear debris distributed across the surface (e) parallel wear tracks (f) show speaks and valley caused by ploughing on the surface.

4. Discussion
The incorporation of potassium titanate into PEEK lowered the average wear rate and causes a significant improvement in the tribological characteristics compared to the pure PEEK, while the concentration of the potassium titanate differ the values of average wear rate. The incorporation of 15wt% of potassium titanate with PEEK gives a lower friction coefficient and reduced wear rate. Also, the composite incorporated with 5wt% and 10wt% potassium titanate showed better values of friction coefficient which are closest to 15wt% of filled composite. The poor quality of transfer film formation resulted in worse wear behaviour of pure PEEK whereas in case of potassium titanate filled PEEK composite resulted in a uniform transfer film during friction process and that makes the potassium titanate filled PEEK composite to experience lower friction coefficient when compared with pure PEEK.

5. Conclusions
The following conclusion can be drawn from the above resulted details, the PEEK composite when filled with nano sized potassium titanate reduces the coefficient of friction and wear rate of PEEK and from the obtained results it clear that with the increase in the concentration of potassium titanate, the wear behaviour of the PEEK composite gets enhanced.
On examining the surface morphology of the worn surface, the thin uniform transfer film formed during the friction process helps in enhancing the wear resistance of the material by having a strong influence in reducing the material wear at the time of friction process. This explains why nano sized potassium titanate results in decreased coefficient of friction and wear coefficient.

The hardness of the PEEK composite was also resulted in higher values with the increase in concentration of potassium titanate. From the observed results it can be concluded with the increase in concentration of potassium titanate as filler in the PEEK composite the wear resistance of the composite was greatly improved.

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