

Development and Study of Characterization of PEEK Composites under harsh Operating Condition with Addition of Fillers

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Abstract

Looking to the present industrial scenario, Light material is increasingly demands of Industry for different application. Especially in automobile sector the reduction in weight of vehicle is the challenge for inventors. As the weight of vehicle is proportional to engines performance. To make up this demand of light material, large work has been done on ceramic and its advancement. But due to its high cost it is only using for specific applications such as high-speed cars and airplanes. To overcome this problem inventor has started in search of alternative material that will be cheaper than ceramics and fulfill the industrial demands. Polymer material plays an important role being as cheaper and light material. Because of significant properties polymer material are increasingly used in different industry for different applications. As far as concern with machine wear is the measure parameter that decreased the machine performance, machine life as some of machine component get worn out due to continuous and heavy working under the harsh operating conditions such as at high temperature zone, heavy load or either at high speed. In such application ordinary polymer material are not suitable when it is to be used for harsh operating conditions. PEEK is the one of the solution amongst the polymer. With outstanding mechanical property, PEEK is helps in a replacement of new polymer material for number of application. In some application pure PEEK also lacks its physical as well as mechanical properties, therefore Advancement in the PEEK is the real challenge for scientist composite. This paper gives us outlook regarding development of new

polymer composite and study of its characterization under the harsh operating condition.

Keywords: *Light material, ceramic, Advancement in PEEK, High temperature, High velocity, PEEK (poly- ether-ether-keton), Wear, Coefficient of friction.*

INTRODUCTION

Tribology is the science and technology of interacting surfaces in relative motion of related subjects and practices. Sliding and rolling surfaces represent the key to much of our technological society.

Understanding of tribological principles is essentials for successful design of machine elements. When two normally flat surfaces are placed in a contact, surface roughness causes contact to occur at a discrete contact spots and an interfacial adhesion occurs. Materials coatings and surface treatments are used to control friction and wear. One of the most effective means of controlling friction and wear is by proper lubrication, which provides smooth running and satisfactory life of machine elements. Lubricants can be liquid, solid, gas. Present scenario works on solid lubricant as per demands from the industries. Some applications have complicate design and some application has to be installed in complicate placeless where periodically oiling requires for its better performance. In such cases

lubrication is also serious problem because of machine location and complicate design. Solid lubrication is the solution for such application. So now a day the more emphasize has been given to investigation of solid lubricants. Meanwhile the number of works has been done on metal matrix composites but to meet the light weight material industrial demands polymer matrix composite material is also the solution instead of metal matrix composite.

The aim of this work is to find and develop the better combination of polymer matrix solid lubricant for different application. PEEK, PTFE, and sub- micropartical of MoS₂ and Bronze are investigated in dry sliding conditions. Friction and wear experiments were conducted on pin-on-disc apparatus, using composite pins against polished EN8 steel counterparts, performed within the room temperature. With the addition of sub-micropartical, the frictional coefficient and wear rate of the composites were further reduced especially at high loading condition. In this

investigation, with insertion of Mos2 in PEEK improves friction and wear properties but also enhance the mechanical characteristic of PEEK.

The matrix PEEK is blended with different filler such as Bronze, Mos2 and PTFE with different proportion by it weight. The Polymer polyether- ether- ketone (PEEK) is a semi-crystalline, high performance engineering thermoplastic with a very good combination of thermal ($T_g = 143^\circ\text{C}$, $T_m = 343^\circ\text{C}$, continuous service temperature 250°C , heat distortion temperature often in excess of 300°C and better mechanical strength, modulus, toughness, resistance to creep, abrasion and good fatigue strength. It is injection as well as compression moldable polymer with good resistance to harsh chemical environment (Bijwe, J. et al, 2005) For last 15 years, it has been focussing of research for enhancing its tribo-potential in various ways and review articles updating the state of art of PEEK tribology have also appeared from time (Zhuang G.S. et al, 2007). Despite its good resistance to adhesive wear, it has serious drawback from a tribological point of view.

It is known to exhibit a coefficient of friction as high as 0.7 under some operating conditions, which limits its

utility as an anti- friction material. Another potential problem with this material, is its scuffing behavior (Feng, Xin, et al, 2008). Development and tribo-evolution of various blends of PEEK to enhance its T_g with some high temperature polymer such as poly ethamide (Lal, B.S. et al, 2007), thermo tropic liquid crystal polymer (Friedrich, J.K. et al, 2005) and PTFE (Breidt, C. et al, 2004) have also been tried. Very low coefficient of friction (as low as 0.1) without scuffing at high pressure velocity and very low in the range of 10-6/Nm. The high wear rate at high temperature is a serious problem in many industrial applications such as elevated temperature compressor piston rings and bearings (Taktak S et al, 2006).

Meanwhile, to meet the combination of light weight and high strength demands, polymer-based materials are increasingly applied in many industries. However, at temperatures above 180°C is a challenge for most of the polymer composites (West, G.H. et al, 1973) , PEEK composites are expected to have excellent high-temperature tribological properties due to their outstanding mechanical and thermal performances above 240°C , where polytetrafluoroethylene (PTFE) composites will meet severe creep behaviors However, high friction

coefficient and big (Lu and Friedrich et al, 2005) wear rate of pure PEEK limit its wider use .

Resistive and compressive strength of the polymer composites and result in enhanced wear resistance. Among numerous inorganic fillers, Bronze has been found to be a promising structural reinforce for polymers, metals, and ceramic composites because of its excellent reach. The objective of this research is to find friction and wear mechanisms of different filled PEEK composites at harsh operating condition. It is also expected that this work can be helpful to the use for bearing, compressor piston rings, impeller, etc.

EXPERIMENTAL

Preparation of materials commercially available Polyetheretherketon (PEEK) of grade 450G fine powder with the average diameter of 100µm was supplied by Victrex. The polytetrafluoroethylene (PTFE) powder with the diameter smaller than 60µm was provided by PCEE Textile Kanpur Bronze powder with 10% tin was supplied by Pometon India Ltd. Molybdenum disulfide powder of diameter 100µm also supplied by Vishal Pharmachem Mumbai. The composite was prepared by compression as well as injection molding. First PEEK, PTFE,

BRONZE AND MOS₂ were mixed with different proportion for various batches with batch size 100gm for compression molding and 15 gm. For injection molding. For accurate weighing digital weighing balance are used with accuracy 0.0001gm for uniform mixing were done by compounding of raw materials.

Compounding:

The compounding of materials was done at Central Institute of Plastic Technology, (CIPET) Bhubaneswar, Orissa. Compounding was done before specimen preparation so that it forms uniform mixture or homogeneous mixture. For compounding of raw material Twin screw extruder are used during compounding of material firstly the PEEK, PTFE, BRONZE and MOS₂ were weighed on digital weighing machine with weight measuring accuracy 0.0001gm. This compounding was done by compression molding, the PEEK, PTFE, MOS₂ and BRONZE which all were in powder form were mixed proportionally in china crucible and steered by china spoon. The prepared mixture was poured by stainless steel spoon slowly inside the Twin- screw extruder which was seated at temperature of 400°C and screw speed were kept 120 rpm, so that extruded material pellets form during extrusion would be extruded and

that would be handled properly. The extruded pallets were undergoing water bath so that they can cool down soon and could not be broken into small pallets which would be difficult to handle.

The long thread like pallets was collected and then it would be again cut into small granules by using pallet cutter machine. These small granules were collected in polybag and coded it for further specimen process. Same procedure was used for all three types of combination and all were packed in polybag and bags were coded with code S1, S2, & S3. Before the compression molding the prepared granules were dried in hot electric furnace about 1hr at 80°C in order to remove moisture present in it. The dried granules then underwent compression molding with pressure 120MPa and temperature 400°C as the processed based material PEEK whose melting temperature is 343°C and when it goes compounding its processing temperature required would be up to 400°C so that it can be processed properly. The compression test was carried out near about for 3hrs. With the help of compression molding sheet of dimension 100mm×100mm×6mm with weight of 75g was prepared. The sample specimens were cut into small pins of dimension 6mm×6mm×30mm. These samples were

grind for surface finishing on fine grinder as well as various fine grade emery papers were used for perfect flat surface so that zero clearance were maintain between pin and disc.

In such a way 03 pins with code S1, S2 & S3 were ready for testing on Tribo meter TR-20 for elevated temperature test. 2.1.2 Compounding by micro compounder: The specimen for mechanical testing as well as tribo-testing made by injection molding. This micro compounding facility available at CIPET Bhubaneswar. For micro compounding same all raw material like PEEK, PTFE, BRONZE and Mos2 weighted with digital weighing balance 15gm per batch, contains all four-powder material with different percentage.

In micro compounder the processing temperature for PEEK were kept at 400°C. The batch of 15gm was poured step by step and allows melting 30 minute so that complete homogeneous mixture would be formed. During processing of mixture, the processing temperature are kept 400°C. This temperature are divided into six temperature zones. The temperature profile was kept 390°C, 350°C, 341° front side and 340°C, 350°C, 392°C for rear side for proper mixing and melting step by step. The maximum axial torque or axial

forces are selected up to 8000N. For processing of PEEK material maximum force value were required 4037N. This force value increasing in ascending order from top side to the molten mixture were collected in gun which is also maintain at processing temperature 400°C to prevent hardening of PEEK material as its soaking period is very small. The collected molten mixture was injected into die with help of injection molding machine and sample were prepared for mechanical as well as tribological test.

Selection of counterpart:

The counterpart i.e. disc is selected with consideration of application like air compressor. The generally the cylinder liner for air compressor or cylinder is made up by stainless steel or grey cast iron with this reference the counterparts also selected as made up of same material. So, the disc material was selected steel with grade EN38 and grey cast iron with dimension Ø165mmx8mm thickness made ready for test. After that disc hardness was checked by Brinell hardness Tester and surface roughness 0.4µm was checked at Vishal Engineering.

Wear Test:

The prepared samples were used for tribological test for elevated temperature at

P. Dr. V. V Patil College of Engineering Ahamednagar. Maharashtra. The Wear was performed on a pin-on disc apparatus according to ASTM D2538 and ASTM D2396. The test rig was supplied by DUCOM Instrument Bangalore, shown in fig 3.1.4. Initially the calculations were done before test. Specially the wear tests were conducted for non-lubricating reciprocating compressor piston ring.

The basic aim was that to minimize wear rate and find better material for piston rings of non-lubricating reciprocating compressor. The Specimen pin (4×4×30 mm³) was run against the polished steel disc of grade EN-38 with an initial surface roughness of 0.4 µ. With contact pressure ranges from 1 to 4MPa. The value of contact pressure were selected with no lubricating air compressor application which works at working pressure at 4MPa. and sliding velocity were selected in the range from 1.8m/s to 3.4m/s.

To evaluate the durability at elevated of material at elevated temperature, the pin was kept in the temperature-controlled environment i.e. Pin holder or collet was kept inside the collet holder which has provides heating device. The electrical heating device was controlled by microcontroller in various ranges.

The variation in the temperature of collate is in the steady state was less than $\pm 5^{\circ}\text{C}$. during the test load values were selected from ranges 10N to 50N. and temperature were kept from 100°C to 250°C as glass transition temperature of PEEK is 143°C and melting temperature is 343°C as per standard data supplied by Victrix's another aims for keeping the temperature from 100°C to 250°C has taken specific application of ATLAS CAPCO non-lubricating reciprocating air compressor.

The generally discharge temperature of compressed air vary from ambient to 70°C as compressor run continuously long time. Also, similarly all parameter like sliding velocity, load and temperature parameter were selected on basis of considering same application. During the test first specimen with code S1 were holed in a temperature environment and initial temperature was seated at 100°C , load 10 N and sliding velocity 1.8m/s. as input parameter for wear and frictional force measurement. During the test, the frictional coefficient was recorded and calculated by a ratio between the tangential force and normal load. This is also monitored by placing

load cell transducers. The test temperature was monitored by an iron-constantan thermocouple positioned in the hole of collet where pin specimen was fitted.

The reduction in height of the specimen was measured by a displacement transducer, could be used to characterize the wear process. However, the reliability of this measurement was affected by the possible thermal expansion of the sliding counterparts. Therefore, after the test the mass loss of the specimen was measured to calculate the specific wear rate by the equations.



Fig. 1 Micro compounder



Fig. 2 Temperature profile for PEEK composites



Fig. 3 PEEK70%wt+PTFE15%wt+BRONZE15%wt



Fig. 4 Injection molding machine



Fig.5 Specimen obtained by injection molding



Fig. 6 Wear and friction measuring test rig TR-20

RESULT AND DISCUSSION

This section discussed about the outcome from the experimental work. The Behavior of PEEK and PEEK composite at ambient temperature has been studied under variable load condition and discussed in this section from the experimental data. Table shows the variation in frictional

force, frictional coefficient and specific wear against the time for the neat PEEK and PEEK composites. The wear resistance greatly enhanced by the addition of fillers and observed that without reinforcements of filler material, the base polymer material PEEK is easily removed

by hard asperities of the metallic counterpart, resulting high wear loss. When the PEEK is modified with PTFE, MOS2 and BRONZE, a polymeric film can be transferred to the EN8 metal disc which is a counterpart that prevents point contact between the asperities of two

meeting objects and result less wear as compare to pure PEEK. The tribological behavior for PEEK and PEEK composites are studied under the standard normal condition at different loading condition are discussed as below.

Table 1: Tribological properties of PEEK composite at 2kg load

T (Min)	WEAR			Coeff. Of Friction			Frictional force		
	P1	P2	P3	P1	P2	P3	P1	P2	P3
0	9.07	13.58	-4.94	0.16	0.189	0.13	3.28	3.78	2.66
10	23.2	20.80	11.55	0.22	0.226	0.24	4.55	4.52	4.96
20	34.7	26.50	17.75	0.23	0.227	0.23	4.58	4.55	4.66
30	42.6	27.69	24.63	0.25	0.26	0.22	5.06	5.20	4.58
40	50.5	34.83	28.87	0.23	0.264	0.22	4.66	5.28	4.45
50	59.1	38.77	33.12	0.26	0.263	0.24	5.27	5.26	4.89
60	68.7	41.95	36.68	0.26	0.277	0.23	5.34	5.55	4.66
70	78.2	44.35	40.71	0.28	0.288	0.23	5.64	5.76	4.60
80	85.8	48.31	41.46	0.26	0.285	0.22	5.24	5.70	4.59
90	92.4	52.21	45.03	0.28	0.293	0.22	5.70	5.86	4.51
100	100	55.81	46.07	0.28	0.309	0.22	5.79	6.19	4.41
110	108	59.86	47.64	0.28	0.236	0.22	5.78	4.73	4.54
120	112	62.27	50.02	0.27	0.241	0.22	5.56	4.83	4.50
130	117	64.35	50.57	0.29	0.24	0.23	5.93	4.80	4.46
140	122	65.48	52.00	0.30	0.266	0.22	6.08	5.33	4.54
150	126	66.78	55.49	0.29	0.293	0.22	5.84	5.86	4.57

160	132	68.81	57.51	0.30	0.313	0.23	6.11	6.27	4.63
170	135	71.27	58.82	0.30	0.257	0.23	6.07	5.14	4.68
180	139	73.71	61.19	0.32	0.291	0.23	6.44	5.83	4.61

Table 2: Tribological properties of PEEK and PEEK composite at 4kg load

Time Min.	WEAR (mm ³)			Coeff. Of Friction			Frictional force (N)		
	P1	P2	P3	P1	P2	P3	P1	P2	P3
0	2.22	6	0.7	0.22	0.22	0.17	8.64	8.75	6.81
10	2	5	0.6	0.23	0.28	0.27	9.35	11.30	10.7
20	7	5	7.9	0.26	0.30	0.28	10.3	12.33	11.4
30	25	3	16	0.30	0.31	0.3	12	12.67	11.7
40	38	3	24	0.30	0.32	0.3	12.2	13.06	11.8
50	50	2	32	0.29	0.34	0.29	11.9	13.60	11.9
60	60	9	43.4	0.29	0.34	0.31	11.6	13.66	12.5
70	71	1	53	0.27	0.34	0.3	11	13.6	12.3
80	81	8	64	0.29	0.34	0.3	11.7	13.6	12.0
90	94	14	78.	0.28	0.33	0.3	11.4	13.6	12.3
100	100	21	93	0.29	0.33	0.3	11.8	13.4	12.3
110	110	27	108	0.3	0.33	0.3	12.0	13.3	12.3
120	120	34	127	0.28	0.32	0.3	11.3	13	12.2
130	130	40	143	0.28	0.33	0.3	11.3	13.3	12.2
140	135	46	160	0.27	0.33	0.3	11.1	13.3	12.3
150	14	52	178	0.30	0.33	0.3	12.0	13.3	12.3

160	150	58	200	0.3	0.32	0.2	12.0	13.1	11.7
170	155	64	209	0.29	0.32	0.2	11.9	13.1	10.3
180	160	70	215	0.3	0.33	0.25	12.0	13.2	10.0

Table 3: Tribological properties of PEEK and PEEK composite at 8kg load

Min	WEAR (Mm ³)			Coeff. Of Friction			Frictional force (N)		
	P1	P2	P3	P1	P2	P3	P1	P2	P3
0	3.66	9.2	26	0.18	0.22	0.15	15	18	11
10	12.2	2.7	29	0.19	0.21	0.22	15	17	17
20	37	2.8	57	0.19	0.24	0.22	15	20	18
30	53	5.6	92	0.2	0.25	0.29	16	20	24
40	71	18	117	0.2	0.25	0.27	17	20	21
50	105	35	151	0.2	0.26	0.27	18	20	21
60	149	54.3	171	0.2	0.25	0.27	19	20	22
70	193	74	193	0.2	0.24	0.30	20	20	24
80	223	99	210	0.2	0.24	0.29	20	19	23
90	259	122	231	0.3	0.23	0.32	24	18	25
100	298	147	256	0.2	0.23	0.32	23	18	26
110	315	172	278	0.2	0.23	0.30	23	19	24
120	338	201	303	0.3	0.23	0.30	23	18	24
130	386	226	327	0.3	0.23	0.31	23	19	25
140	413	251	348	0.2	0.23	0.30	23	19	24
150	453	280	368	0.3	0.22	0.30	24	18	24

160	480	307	389	0.3	0.22	0.29	23	18	24
170	515	335	415	0.3	0.22	0.31	25	18	25
180	546	362	437	0.3	0.21	0.30	24	17	24

CONCLUSIONS

The tribological properties of peek and peek composites filled with ptfe, mos2 and bronze were systematically studied under Harsh operating condition. Especially the heavy load and high temperature were preferred in its performance analysis. From the result the following conclusions are drawn. In comparative study of peek & peek/ptfe/mos2. It was observed that wears of peek composites decreases at high temperature as compared to neat peek with addition of mos2 as filler material. It was also clear that mos2 filled peek shows good lubricity quality than pure peek. With addition of bronze in peek cleared that bronze filled peek composites showed more effective than the mos2. This result cleared that bronze can be act as good solid lubricants at low as well as at high as compared to pure peek and mos2 filled peek composites.

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