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Research Article

Investigation of Tribological behavior of PEEK and PEEK Composites at Elevated Temperature

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Abstract

*In the petroleum , chemical industry and coalmines where high temperature is often seen, special materials have to be used. The high wear rate at high temperature is a serious problem in large number of industrial applications such as elevated temperature compressor piston rings and bearings. 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. PEEK composites are expected to have excellent high-temperature tribological properties due to their outstanding mechanical and thermal performances above 180°C, where polytetrafluoroethylene (PTFE) composites will meet severe creep behaviors. To improve the tribological properties of high temperature resistance thermoplastic polyether-ether-ketone (PEEK) and PTFE, reinforced with short carbon fibre **(SFC)**, graphite flakes, and sub- micro particles of **MoS2** were investigated in dry sliding conditions. Friction and wear experiments were conducted on pin-on-disc apparatus, using composite pins against polished EN8 steel counterparts. Test performed within the p v-ranges at room temperature and elevated temperature (up to 300 °C). It was found that conventional fillers, i.e SCF and graphite flakes could effectively enhance both the wear resistance and load carrying capacity of base polymers. With the addition of sub-micro-particles, the frictional coefficient and wear rate of the composites were further reduced especially at elevated temperatures. In this investigation, with insertion of Mos2 and Bronze as metal solid lubricants improves friction and wear properties but also enhance the thermo mechanical characteristic of PEEK adhesive wear of PTFE/PEEK composites*

Keywords: *PEEK, PTFE compounding, wear, coefficient of friction; friction, elevated temperature.*

1. Introduction

Polymer-ether ether- ketone (PEEK) is a semi-crystalline, high performance engineering thermoplastic with a very good combination of thermal (Tg =143°C,Tm = 343°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 focus 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) . In spite of its fairly 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 Tg 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 a large number of 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 180°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 . Resistance 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

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ceramic composites because of its excellent reach. The objective of this research is to find friction and wear mechanisms of different fibers-filled PEEK composites at elevated temperatures. It is also expected that this work can be helpful to the use for bearing, compressor piston rings, impeller, etc.at elevated temperatures.

2. Experimental

2.1. Preparation of materials

Commercially available Polyetheretherketon (PEEK) of grade 450G fine powder with the average diameter of 100μm was supplied by Victrex. The polytetrafluroethylene (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 were prepared by compression as well as injection molding. First PEEK, PTFE, BRONZE AND MOS2 were mixed with different proportion for various batches with batch size100gm 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.

2.1.1 Compounding: The compounding of materials were done at Central Institute of Plastic Technology, (CIPET) Bhubaneswar, Orissa. Compounding was done before specimen preparation so that would form 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 were poured by stainless steel spoon slowly inside the Twin-screw extruder which was sated at temperature of 400°c and screw speed were kept 120 rpm,so that extruded material pallets 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 zone. The temperature profile were 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 in order to prevent hardening of PEEK material as its socking 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.

2.1.3 Selection of counterpart: the counterpart ie 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 ion 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.

2.2. 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 Banglore, 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 fine 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 evaluated the durability at elevated of material at elevated temperature, the pin was kept in the temperature controlled environment ie Pin holder or collet was kept inside the collet holder which is has provides heating device. The electrical heating device was controlled by microcontroller in various ranges. The variation in the temperature of collet is in the steady sate was less than ±5°c.during the test load values were selected from ranges 10N to 50N. and temperature were kept from 100c° to 250°c as glass transition temperature of PEEK is 143°c and melting temperature is 343°c as per standard data supplied by Victrex.Also another aim 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 sated 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. Therefor after the test the mass loss of the specimen was measured to calculate the specific were rate by the equations.

$$W = \frac{\Delta m}{\rho F n D} [\text{mm}^3/\text{ Nm}]$$

Fig.2.1 Wear and friction measuring test rig

Fig.2.2 Specimen obtained by injection molding

Table.2.1 Tribo-properties of Neat PEEK and PEEK composites at elevated temperature

Composition	P-v Mpa, m/s	Temperature of pin (°C)	Frictional force (N)	Coefficient of friction	wear (µm)
Neat PEEK	0.347,3.4	50	2.86	0.06	52
	0.347,3.4	100	2.49	0.08	131.42
	0.347,3.4	120	2.29	0.05	72.50
	0.347,3.4	150	2.48	0.05	84.75
	0.347,3.4	175	2.56	0.06	63.39
PEEK /PTFE/MOS2	0.347,3.4	50	3.0	0.063	10
	0.347,3.4	100	3.1	0.065	-18
	0.347,3.4	120	3.2	0.060	- 28
	0.347,3.4	150	3.0	0.060	-10
	0.347,3.4	175	3.0	0.060	18
PEEK /PTFE/BRONZE	0.347,3.4	50	2.6	0.05	-25
	0.347,3.4	100	2.9	0.06	-110
	0.347,3.4	120	2.8	0.06	-125
	0.347,3.4	150	3.0	0.06	-150
	0.347,3.4	175	3.0	0.07	-170

3. Result and Discussion

The comparative study of PEEK and PEEK/PTFE/Mos2 were studied from table 1. It was observer that wear rate at 50°C and at 175°C for pure PEEK was found to be 52 micron to 63 micron. This indicates that for pure PEEK due to increasing the temperature it thermo mechanical properties decrease which leads to increase the wear rate because of softening of material further increasing the temperature the wear rate falls down drastically and archived the value 63 micron at. 175°C. But with addition of mos2, it was found that ware rate is very less as compare to pure PEEK. Wear value drastically enhanced by mos2 addition. It was also observed that transfer film formed very soon with increasing of temperature as compared to pure PEEK material. The negative values of wear till continued up to temperature 150°C with increasing temperature the value of wear increased when temperature goes beyond the 150 °C. It was also observed that with addition of BRONZE in PEEK from table 2 and table 3 shows that bronze is more effective than the mos2. It was observed that with same operating parameters BRONZE show good lubrication properties. The wear was tremendously falls with increasing temperature than the Mos2.

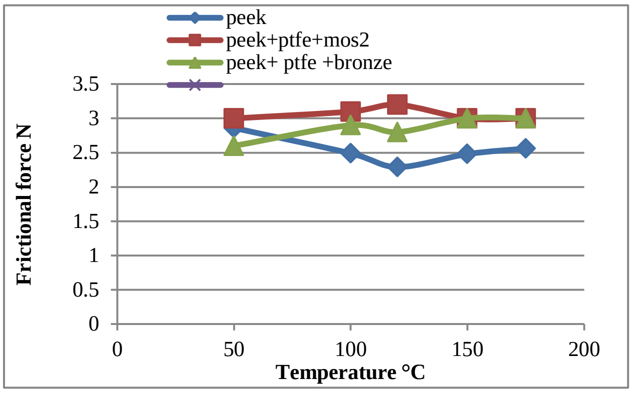

Fig.3.1 Effects of temperature on Frictional force for PEEK and PEEK composites at 1Kg load and 3.4m/s velocity

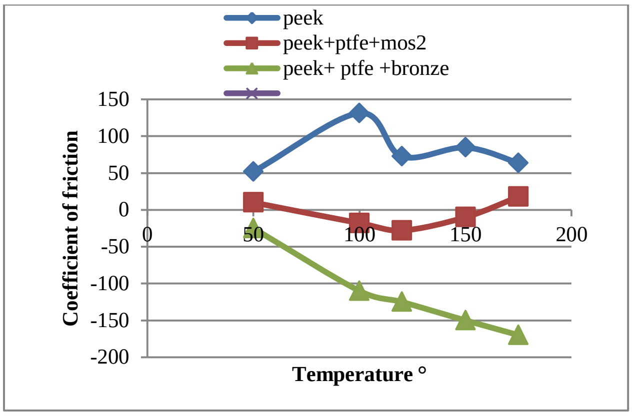

Fig.3.2 Effects of temperature on coefficient of friction for PEEK and PEEK composites for 1Kg load and 3.4m/

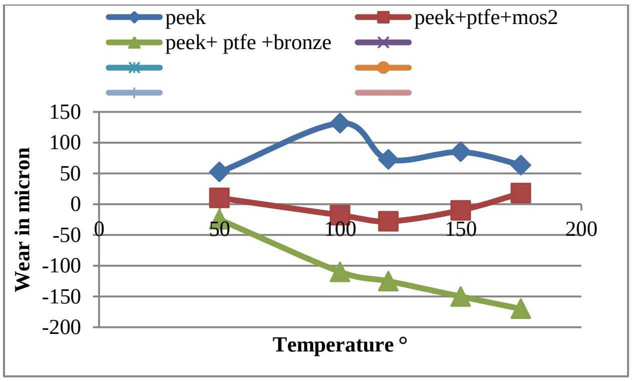

Fig 3.3. Effects of temperature on wear peek and peek composites for 1kg load and 3.4m/s velocity

Conclusion

The tribological properties of peek and peek composites filled with ptfe, mos2 and bronze were systematically studied under different operating condition at low temperature as well as at elevated temperatures. From the result the following conclusions are drawn. In comparative study of peek &peek/ptfe/mos2. It was observer that wears of peek composites decreases at high temperature as compare 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 compare to pure peek and mos2 filled peek composites.

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