Tribology International 117 (2018) 225–235

Contents lists available at ScienceDirect

Tribology International

journal homepage: www.elsevier.com/locate/triboint

Effect of nano-sized TiO_2 addition on tribological behaviour of poly ether ether ketone composite

Abdulaziz Kurdi [a,b], Hongjian Wang [a], Li Chang [a,*]

[a] Centre for Advanced Materials Technology, School of Aerospace, Mechanical and Mechatronic Engineering, The University of Sydney, Sydney, NSW 2006, Australia

[b] The King Abdulaziz City for Science and Technology, The National Center for Building System, P O Box 6086, Riyadh 11442, Saudi Arabia

ARTICLE INFO

Keywords:
Nanoindentation
TFL
TiO_2 nanoparticles
Water lubrication

ABSTRACT

Tribological behaviour of poly ether-ether ketone (PEEK) loaded with different amount of titanium oxide (TiO_2) nanoparticles were investigated by pin-on-disk test followed by the characterization of associated transfer film layer (TFL) with the help of nanoindentation. PEEK loaded with 5% TiO_2 nanoparticles exhibit lowest specific wear rate in both dry and water lubricated conditions mainly due to the formation of characteristic transfer film layers and water lubrication, respectively. Intrinsic molecular structure of PEEK synergise tribological responses when reinforced with nano-particles. The inbuilt nature of PEEK in contact with water cause matrix relaxation which in turn overshadows the benefit of nanoparticles addition beyond 5% in PEEK composite and overall outcome is the inferior wear-rate of PEEK composites compared to dry tests condition.

1. Introduction

Over the past decades, engineering polymers have been progressively used for tribological applications in a number of industries like physical, chemical, automotive and aerospace. These tribological applications have pushed the research towards the development of high performance polymers [1-6]. The properties obligatory for tribology related applications include sustaining elevated service temperature, superior chemical, mechanical and wear resistance as well as outstanding cohesive strength. With these in agenda, poly ether-ether ketone (PEEK) is regarded as one of the most capable polymer for such applications due to its inherent semicrystalline thermoplastic nature with excellent mechanical and chemical resistance [7-10]. The mechanical and tribological performance of general polymers by itself, like PEEK, can be improved by the addition of fillers in such polymers in various forms such as fibres, particle, whiskers or a combination of those [4]. However, the combination of polymer and filler is not straight forward, as not only types of fillers but also types of polymer have profound effect on overall tribological behaviours of resultant polymer composite. As a rule of thumb, smaller the particles/fibres used as fillers, better is the wear resistance of polymer composites as evident in literature [5-7]. Considering this facts, some nano-metric inorganic fillers have the ability to reduce friction and wear of polymer composites. In the case of PEEK reinforced with small amount inorganic nano-particles, such as silicon nitride (Si_3N_4) [11], silicon dioxide (SiO_2) [12], goethite (α-FeOOH) nanoparticles [13], zirconium dioxide (ZrO_2) [14], titanium dioxide (TiO_2) [15-16] contribute towards lowering coefficient of friction as well as wear rate under different sliding conditions [11-16]. This improvement in wear resistance is usually attributed towards the formation of transfer film layers (TFLs) [7,17] which is a mixture of materials from sliding and counterparts incorporated with nanoparticles in it [5,17-18]. Specific wear rates of nano-filler reinforced polymer composites attain a nominal value with escalating filler content, thought it was reported that, excessive nano-filler content results filler agglomeration that might overthrow the benefits. Additionally, homogeneous distribution of nano-filler and a good filler-matrix interaction has further benefit for property improvements as analogues of metal matrix composite [19].

In most tribological applications, TFLs play an important role in load-transfer and dictate the overall wear process and thus a reliable material data for TFL is beneficial [6,20]. Therefore, TFL characteristics are a signature identity for any set of polymer/fillers. Some filler affect the development of TFL positively and there are number of filler which have no such effect and thus enhanced wear instead of lowering it [20]. Sometimes, incorporation of fibrous/whisker reinforcement in addition to nano-fillers with polymer matrix could be beneficial in that respect. Wear of components in tribological application not only depends on base materials but also on fabrication technique and critical reinforcement content which in turn facilitates the formation of TFLs. It was reported

* Corresponding author.
E-mail address: li.chang@sydney.edu.au (L. Chang).

http://dx.doi.org/10.1016/j.triboint.2017.09.002
Received 11 May 2017; Received in revised form 26 August 2017; Accepted 1 September 2017
Available online 11 September 2017

that, once the hybrid nano-composites are full of nanoparticles along with traditional tribo-fillers, the elements were more efficacious in the formation of long-lasting TFLs on steel counterpart associated with desirable tribological properties during sliding [7]. Thus TFL efficiency factor is an important term to be analysed for better understanding of tribological behaviour of modified polymer composite as explained in the previous communications [7,20]. In this regard, brittle-ductile transition of polymers with increasing temperatures sometimes plays a vital role to develop TFLs [9]. The enhanced role of nano-filler addition over micro/macro-filler towards better TFLs formation is well documented in literature [21-24,37,38]. In general, thanks to their larger surface-volume ratio, nanoparticles tend to offer benefit with much low volume fraction than microparticles. In case of micro/macro-filler reinforced composites, the TFLs fragmented easily as the wear-debris are relatively larger as shape and size of wear debris are very much related to the shape/nature of nano-fillers itself. Finally, for nanoparticles, the abrasiveness of the hard particulate fillers decreases remarkably as the result of the reduction in their angularity.

Considering mechanical properties of high performance PEEK, which is now used as sealing materials in container for water cooling in nuclear power plant [21], tribological behaviours of PEEK in water lubricant condition is a major concern. Most of the work available in literature focus on the tribological aspects of high performance polymers under dry conditions with a few reports involve water lubricated conditions [13, 25-29]. As reported by Gao et al. [13], addition of nanoparticles in PEEK matrix enhance the formation of transfer layer, due to dehydration reaction and improve the overall tribological performance of PEEK composite. Thus, more scientific investigation is foreseen in this area for better understanding tribological aspects in water lubricated conditions. During the presence of water in pin-on-disk tests, a number of phenomena takes place like absorption of water, removal of heat at contact and interference during TFLs formation. These aspects affect the friction and wear behaviour which is otherwise not seen in dry condition tests. Water absorption by the free volume of amorphous polymer reduce materials' strength and modulus of elasticity which in turn favours the breakage of polymer macro-molecules chains. In addition, swelling cause differential expansion followed by plasticization of polymer surfaces during rubbing action [28].

Therefore, the aim of this work is to study the tribological and mechanical properties of PEEK reinforced with titanium dioxide (TiO_2) nano-particles under dry and water lubricated sliding conditions at room temperature. Special attention has been paid on the characterization of TFLs formed under different conditions, as well as their effect on the wear performance of polymeric specimens.

2. Materials and methodology

Commercially available PEEK polymer was used as matrix materials (Victrex, UK) and inorganic titanium dioxide (TiO_2) (Kronos 2310) with average diameter of 300 nm was used as fillers. Density of PEEK and TiO_2 was 1.3 g/cm^3 and 3.9 g/cm^3 respectively. PEEK and PEEK with different percentage of TiO_2 were prepared by a twin screw extruder in Arburg all-rounder injection moulding machine at around 400 °C. Four samples were prepared, namely, pure PEEK, PEEK with 5% TiO_2, PEEK with 10% TiO_2 and PEEK with 15% TiO_2. All TiO_2 content was in weight percentage. Dimensions of all specimens were 4 mm × 4 mm x 12 mm for pin-on-disk testing under two different slide conditions: dry condition and water lubricated condition at room temperature. All polymer pin surfaces were polished against a rotating disk covered with polishing cloth without any lubrication. Just before the tests, the samples were pre-heated at 50 °C for 3 h to get rid of surface moistures. Pin-on-disk tests were performed by using NANOVEA (MT/60/NI) tribometer for a period of 24 h under 5 N normal load and 0.1 m/s sliding speed. A carbon steel disk (German standard 100Cr6) was used as counter body with hardness of about 13 GPa. The internal diameter of the disk was 25 mm and external diameter of 42 mm with surface roughness (Ra) of about 220 nm, as measured by non-contact profilometer (STIL, France). The steel disk was also polished against polishing cloth with 0.1 μm alumina slurry. Specific wear rate was calculated by using equation (1) [30]:

$$W_s = \frac{\Delta m}{\rho \times F_N \times L} \left[\frac{mm^3}{N \times m}\right] \quad (1)$$

Where Δm was mass loss of specimens' during the test, ρ was density, F_N was applied normal force and L was total sliding distance. Each individual tests were triplicated under identical test conditions and average values were used for result analyses. After the sliding tests, worn surfaces of both test coupons as well as steel counterbody were examined by Carl Zeiss field emission scanning electron microscope (FE-SEM). Nano-indentation wear performed with the help of Hysitron triboindentation instrument (Hysitron Inc., USA) with a diamond Berkovich indenter to investigate the hardness of tattered surfaces as well as to characterize TFL with following parameters: peak/maximum indentation load of 3 mN with loading and unloading rate of 0.3 mN/s and a holding time of 5 s at the peak loads. Each indentation test was carried out at 40 straight points over the total sliding distance of about 600 m. Thickness of TFL was estimated by using Equation (2) [7,30]:

$$h_f = h_t - h_s = h_f\left(1 - \frac{h_s}{h_t}\right) = h_t\left(1 - \sqrt{\frac{(H_C - H_f)}{(H_s - H_f)}}\right) \quad (2)$$

Where h_t was total indentation depth, h_f was film thickness, h_s was indentation depth in substrate, H_c was composite hardness of film/substrate system, H_f and H_s was intrinsic hardness of film and substrate, respectively. In addition, transfer film efficiency factor (λ) was determined by using Equation (3) [26]:

$$\lambda = \frac{t}{R_a} \quad (3)$$

where t = $(h_f)_{average}$ was average thickness of TFL determined by nano-indentation and Ra was surface roughness of steel counterpart. Details of equations formulation as well as the concept of TFL were reported in our previous communications [7]. Mechanical properties of the samples were carried out by performing tensile tests on an Instron 5567 instrument at 5 mm/min crosshead speed according to ASTM D638-99 standard [31] by using type IV specimen. Each test was performed on at least five samples for each material composition under ambient temperature. Both longitudinal and lateral strains were monitored via clip-on extensometers against 50 mm and 25 mm gauge length, respectively. 0.2% offset strain was used for the definition of yield. Fracture tests were carried out according to ASTM D5045-99 standard. Sample thickness (B) and width (W) was 6 mm and 30 mm, respectively. A sharp crack between 0.45 W and 0.55 W was introduced by tapping a fresh razor blade at the notch tip. Equation (4) [31] was satisfied for valid plane strain toughness (K_{IC} or G_{IC}) measurements. At least four specimens were tested for each composition with a loading rate of 1 mm/min.

$$B, a, \ (W - a) \geq 2.5\left(K_{IC}/\sigma_y\right)^2 \quad (4)$$

K_{IC} was obtained according to equation (5) [21]:

$$K_{IC} = \frac{P}{B\sqrt{W}} \ f(a/W) \quad (5)$$

where P is applied load. Fourier-transform infrared spectroscopy (FTIR) of the PEEK samples before and after pin-on-disk sliding test at dry and wet condition were recorded using an Agilent 4300 spectrometer at room temperature. Four scans were collected for each sample in the wavelength range between 4000 and 650 cm^{-1}.