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Tribo-performance enhancement of PAEK composites using nano/micro-particles of metal chalcogenides

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ABSTRACT

While designing the ambitious advanced composites based on PAEK (50 wt%), glass fibers (30 wt %) and natural graphite (10 wt %), it was proposed to include one more solid lubricant (10 wt %) from the category of metal chalcogenides. Particles of MoS_2 and WS_2 in micron and nano-sizes were selected for inclusion in combination with natural graphite. The themes for investigations were; which one is the better performer amongst MoS_2 and WS_2, and nano-particles can impart beneficial effect or not; and the last how efficient would be these tribo-composites in terms of friction, wear resistance and PV_{limit} values. The composites were characterized in detail for physical, mechanical, thermal and tribological performance. The WS_2 proved more beneficial till moderate PV conditions but not for severe conditions as compared to MoS_2. The composites proved excellent tribo-materials in terms of low friction (~0.04), wear rate (2.2×10^{-16} MPa m/s) and PVsafe values (84 MPa m/s). The chemistry of the counterface surface was studied using X-ray photoelectron spectroscopy (XPS) and Raman spectroscopy (RS) while the morphology of the worn pins surface and counterface surface was observed using a combination of 3D profilometer and scanning electron microscopy.

1. Introduction

Polymers and composites are known to offer an excellent combination of tribo-performance in dry (oil-less) sliding conditions along with high specific strength, quiet operation due to very good damping capacity, ease of tailoring composites etc. Poor thermo-physical properties and thermal stability along with high-temperature sensitivity of performance properties are the major concerns about this special class of tribo-materials. Low PV_{limits} (P-pressure and V- velocity) is the main constraint for their utility for a wider spectrum of applications in severe operating conditions. The research efforts are hence continuously being focused on exploring newer speciality polymers to design the formulations of composites with right choices of combinations of fillers and fibers in right amounts. PAEK (Poly aryl ether ketone) is regarded as one of the most promising polymeric materials used for such advanced tribo-composites although less researched [1–10] as compared to its homologue, PEEK (poly ether ether ketone) [11–24]. PAEK composites have shown significantly high tribo-potential especially high PV values along with low friction coefficient (μ) and specific wear rate (K_0) as evident from authors' earlier publications and as summarized in Table 1 [1–4,6,7].

Chalcogenides or di-sulfides of metals such as Tungsten and Molybdenum are well-known SLs exploited in polymer composites, oils, greases etc. for further reducing the friction and wear [33]. Based on the type of short fibers (glass-fibers-SGF) and its optimized amount (30 wt %), the formulations are open for tailoring the combinations of more efficient solid lubricants such as Molybdenum di-sulfide MoS_2 and Tungsten di-sulfide (WS_2) in various sizes (micrometer and nano-meter) to enhance the tribo-potential further. Such comparative effects of size and types of metal chalcogenides as SLs are not available in the literature.

Gong et al. [28] and Zhang et al. [29] reported about the reduction in wear by adding both graphite and MoS_2 in PTFE. Holinski [30] also revealed that inclusion of MoS_2 in epoxy led to wear reduction. The μ for PEEK + MoS_2 and PEEK + WS_2 materials decreased by ≈ 30% compared to pure PEEK regardless of the particle type or size [25]. It was also found that μ of the Polyethene + MoS_2 and Epoxy + MoS_2 decreased 9% compared to those with Polyethene + PTFE and Epoxy + PTFE [26]. Polyimide (PI) reinforced by 15% carbon fibers and 5% MoS_2 lead to low μ~ 0.18 and low K_0 ~7.91×10^{-15} m^3/Nm [27]. Similarly, the WS_2 particles lowered the K_0 of the neat PEEK by 10%–60%, respectively [22].

Nano-particles (NPs) are known to enhance the performance of nano-composites (NCs) significantly. Addition of WS_2 and SiC NPs into

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Table 1
Tribo-performance of various PAEK composites at $PV_{safe\ limit}$ in severe operating condition.

Material composition (wt. %)	$PV_{safe\ limit}$ (MPa m/s)/ Vel. (m/s)	K_0 (10^{-16} m^3/Nm)	μ	Ref
PAEK+10CF+10G + 10N	72.2 (2.6)	5.54	0.050	[6]
PAEK+30GF + 15G + 5T	95.2 (3.4)	1.88	0.040	[7]
PAEK+30GF + 10G + 10N	96.6 (3.45)	5.45	0.048	[2]
PAEK+30GF + 10G + 10K	98 (3.5)	1.82	0.044	[3]
PAEK+30GF + 15TG + 5N	100.8 (3.6)	5.68	0.040	[4]

*GF- short glass fibers, G- Natural graphite, T- PTFE, N- hBN, CF- short carbon fibers, K- potassium titanate, TG-thermographite.

Table 1a
Raw material (micron size) manufacturer data for MoS_2 and WS_2 and NG.

Properties	Tungsten disulphide (W)	Molybdenum disulphide (M)	NG
Manufacturer	Alroko GmbH & co KG	Alroko GmbH & co KG	Graphite India Limited
Purity (%)	> 99	> 98.5	
Particle size (μm)	0.85–1.15	< 6	12–15

Table 1b
Suppliers' data of nanoparticles of MoS_2 and WS_2.

S No	Properties	Tungsten disulphide	Molybdenum disulphide
1	Manufacturer	Nano-Amor (USA)	Lower friction (M K Impex)
2	Particle size (nm)	90	90
3	Surface Area (m^2/g)	30	35
4	Density (g/cm^3)	7.5	5.06
5	Purity (%)	> 99	> 99
6	Crystal cell	nearly spherical	nearly spherical
7	Mol. Wt. (amu)	248	160.08
8	Melting Point (^{0} C)	1250	1185
9	Boiling Point (^{0} C)	N/A	450
10	Colour	Silver grey	blue-silver grey

CF reinforced PEEK resulted in ultra-low μ (0.021) [31]. Hou et al. [32] incorporated IF-WS_2 (inorganic fullerene-like WS_2) into PEEK coatings and the μ decreased by ~70%. Relatively low loadings, (2.5–5 wt.%) of IF-WS_2 NPs proved efficient SLs. Wang et al. [32] investigated the tribo-properties of PEEK NCs filled with NPs of SiC, SiO_2, Si_3N_4 or ZrO_2 and found that the μ decreased sharply from ~0.38 for neat PEEK to 0.2–0.3.

This paper reports on the tribo-investigations of a combination of micro and nano particles of MoS_2 and WS_2 in PAEK reinforced with SGF and solid lubricated with particles of graphite. Thus, the composites containing a combination of two SLs (micro and nano-sized particles) were designed to understand the role of MoS_2 and WS_2, which in general, exhibit an entirely different environmental dependence with respect to its frictional properties. Therefore, tribo-chemical analysis of transfer films was also performed in order to reveal the friction and wear mechanisms.

2. Experimental

2.1. Materials

The composites prepared in this investigation were based on a powdery polymer viz. Poly aryl ether ketone (PAEK) with a chemical structure already described [1] having an average diameter lesser than 100 μm (GPAEK 1200P, Gharda chemicals limited, Mumbai, India). The selection of 30 wt % short glass fibers (SGF) (supplied by Nippon Electric Glass, Malaysia having a diameter around 10.7 μm and the length in the range of 3–4 mm) into PAEK matrix as a reinforcement was based on significant enhancement in wear resistance reported in earlier work [5]. The natural graphite (NG) particles (12–15 μm) supplied by Graphite India Ltd. Nasik, India were selected as a primary solid lubricant (SL) for developing a series of proposed composites. The two transition-metal dichalcogenides viz. Molybdenum disulphide (MoS_2 -designated as M) and Tungsten disulphide (WS_2 -designated as W) each in two different sizes (micrometer and nanometer) were selected as secondary SLs. Their details are listed in Table 1(a) and (b).

The morphology of these micro and nanoparticles and their sizes are illustrated in Fig. 1 in the form of micrographs Scanning electron microscopy (SEM) and Transmission electron micrographs (TEM).

2.2. Composite preparation

The details of developed composites are shown in Table 2. First four composites are the theme composites while the fifth one C_{GF} containing only glass fibers (30 wt%) and no solid lubricants was developed to understand the extent of the effect of SLs on mechanical and tribological performance. Since 10% NPs cannot be added due to the possibility of agglomeration, only 3% of MPs were replaced with 3% of NPs. In order to achieve uniform de-agglomeration of nano-particles (NPs), probe sonicator was used whose details are described in the earlier paper [1]. NPs of MoS_2 or WS_2 in Acetone medium were ultra-sonicated for 30 min to achieve homogeneous nano-suspension followed by mixing fine particles of PAEK in a beaker with stirring magnetically. The suspension (mixture of PAEK and NPs) was dried using vacuum oven. The dried mixture and graphite was rigorously mixed in a high-speed mixer at a speed of 1200 rpm for 5 min followed by inclusion of 0.02% of TNPP (Tris nonyl phenyl phosphate-anti-oxidant) in order to prevent possible oxidation during processing. Afterwards, the mixture was subjected to compounding in a twin-screw extruder whose details are given elsewhere [2] with a temperature profile from 380 °C to 420 °C and screw speed of 30 rpm. The extruded material was dried followed by granulation and then placed in a main feeder and SGF in a side feeder (feed rate of 3.5 kg/hr.). Compounded granules were subjected to injection molding and the process of manufacturing composites is illustrated in Fig. 2. The injection pressure was 1200 bar following by holding pressure 1000 bar for 10 s under the holding pressure of 85–180 MPa. The mold temperature was kept at 200 °C. The material in the cavity was cooled and solidified for 10 s.

2.3. Performance evaluation

2.3.1. Physical properties

The density of all composites was measured as per ASTM D792 and details of the process are given in earlier paper [1].

2.3.2. Mechanical strength

Mechanical properties such as tensile, flexural and compressive strength were measured as per ASTM standards while Vickers micro-hardness and scratch hardness values were calculated as per ASTM E92-82 and ASTM G171-03 methods and details are given in Refs. [3,4].

2.3.3. Thermo gravimetric analysis (TGA)

The thermal stability of the composites was measured by thermo gravimetric analysis (TGA Q50 thermobalance, TA Instruments Ltd.) at a heating rate of 20 °C/min. Prior to the testing, samples were dried

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