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Development of high performance poly (ether-ketone) composites based on novel processing technique

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A B S T R A C T

Homogeneous mixing of ingredients by efficient method is necessary for desired performance of a composite. In this work, selected solid lubricants and powder of poly ether ketone (PEK) were mixed by probe-sonication method and then composites were developed incorporating short fibers during extrusion process followed by injection molding. Another batch of composites was developed by conventional mechanical powder mixing method instead of sonication. The developed composites were evaluated for physical, thermo-physical, mechanical and tribological properties (in two types of wear modes viz. adhesive and abrasive). The influence of mixing technique on all performance properties was extensively studied including SEM and EDAX studies on the surfaces of worn composites. It was concluded that the probe-sonication technique proved beneficial for improving tribological properties significantly, but not for all mechanical properties. The extent of influence depended on type of solid lubricants, fibers and operating parameters during tribo-evaluation.

1. Introduction

It is a well-accepted fact that performance of a polymer composite (filled with fibers, particles or both) depends on various factors such as; type of matrix and fillers and fibers; their amounts, sizes, aspect ratios, combinations; their orientations with respect to loading direction, and fiber/filler–matrix interface apart from processing technology (extrusion, injection molding, compression molding). A lot is reported about the performance influencing parameters such as type of polymer matrix or its molecular weight [1,2], fillers- their sizes, shapes, aspect ratios, amounts, and interface with the matrix [3,4], fiber/fabrics-type and amount [5–7], techniques for manufacturing prepregs (by hand lay up or use of film or impregnation or vacuum bag process) [8].

In the case of nano-composites extent of dispersion of nano-fillers in the matrix plays a crucial role on the performance of composites. The selected nano-fillers or even micro-fillers may not give the desired properties due to aggregation, tendency to sedimentation, contamination, moisture absorption, etc. Hence, the composites manufacturing technique should be judiciously selected to eliminate or reduce the shortcomings in order to get the desired range of performance properties. Amongst above-mentioned aspects, processing technology or influence of process of dispersion of ingredients (i.e. mixing process of fillers in the matrix) in particular, on the performance of composites is least researched [9–12]. As per general practice during extrusion of composites, fibers are fed from the side feeder while powders or granules of polymer matrix and micro-fillers (which may or may not be mixed evenly by simple mechanical mixing before pouring in the hopper) are fed from the main feeder. It is of interest to examine if the proper premixing of fillers and polymer powders also plays a role on performance properties of the final product.

In the present work, the potential of probe sonication for the homogeneous mixing of solid lubricants in the matrix and its influence of performance properties is explored by developing short carbon/glass fiber reinforced composites based of poly ether ketone (PEK). The solid lubricants were added in the composites to reduce the coefficient of friction (μ) and wear while fibers were added as a reinforcing and wear reducing additive. In this regard two series of composites were developed based on PEK and short fibers (either carbon or glass) and solid lubricants. First series contained carbon fibers and a solid lubricant (SL) viz. hexa-boron nitride (hBN) (also known as white graphite) while the second series contained glass fibers and two SLs viz. natural graphite (NG) or thermo-graphite (TG) in combination with hBN. Graphite and hBN

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have similar hexagonal crystal structure. hBN shows higher thermal conductivity (TC), thermal and thermo-oxidative stability as compared to graphite (Table 1) [11,13].

PEK is a high performance engineering specialty polymer with excellent thermal and mechanical properties (Table 2) along with very good resistance to solvents and chemicals. It is less investigated polymer as compared to poly ether-ether-ketone (PEEK) for development of composites. In recent studies it proved to be excellent matrix for developing composites with significantly superior tribo-properties (very low friction, wear and high *PV* (pressure–velocity) limit [14–16].

In the present work, the potential of probe sonication for homogeneous mixing of solid lubricants (as discussed above) in the PEK matrix is explored and results are presented in subsequent sections.

2. Experimental details

2.1. Materials-selection and procurement

Polymer matrix polyether ketone (PEK) (Grade G-PAEK 1100P) was supplied by Gharda Chemicals Ltd, Mumbai and properties are shown in Table 2. Short fibers of carbon (with average fiber length of 6 mm and diameter of 7–8 μm) and glass (average diameter of 10 μm and length of 3–4 mm) were selected as reinforcements.

Two sizes of hexa boron nitride (hBN) particles were selected for developing solid lubricated composites. Micro-sized hBN (MK-hBN-150) (average particle size 1.5 μm) procured from M.K. Impex, Canada was used while developing carbon fiber reinforced composites (CFRC), while another type (PW Grade) (with average particle size of 3–15 μm) from Zibo Jonye Ceramic Technologies Co., China was used in glass fiber reinforced composites (GFRC). Along with hBN, two grades of graphites were used (individually) in GFRC. The first one thermo-graphite (TG) (Timrex Graphite C-therm-011-Patented product) [18] was procured from Imerys Graphite & Carbon Switzerland Ltd. (Properties shown in Table 3) and natural graphite (NG), was procured from a local supplier with a density of 2.23 g/cc. SEM micrographs of particles of natural graphite and thermo-graphite (TG) are shown in Fig. 1.

2.2. Development of composites

Mixing of solid lubricants with PEK powder was done by two ways; viz. sonication (designated as S) and mechanical mixing-non-sonication (designated as NS).

- Sonication method – SLs and PEK powder were sonicated using ChromeTech probe sonicator with a gradual addition of SLs first followed by addition of PEK in sonication medium (acetone). Sonication was carried out in a step by step process with pulse duration of 10 min. After sonication the samples were dried in vacuum oven at 70 °C to remove the acetone. This mixture was used for further processing during extrusion, followed by injection molding.
- Non-sonication method – The SLs and PEK powder were mixed in a high speed mixer for 5 min. This mixture was used for extrusion followed by injection molding.

Coperion Twin Screw Extruder ZSK 26 having L/D 40:1 was used and mixture of PEK and SL was fed through main feeder. Short fibers of glass or carbon were fed from side feeder. The processing temperature was in the range 380–410 °C. The specimens for mechanical and tribo testing of PEK composites were then injection molded at 400–420 °C on Arburg 60 t machine.

In all, six composites were developed and the detailed compositions along with designations are given in Table 4.

Table 1
Characteristic properties of solid lubricants [11,13].

S.L.	Thermal stability in oxidizing atmosphere (0 C)	Thermal conductivity (W/m K)	Cost	Moisture if required for lubricity
Graphite	800	140–500‖; 3–10⊥	Low	Yes (not desirable feature)
hBN	1000–1200	600‖[a]; 30⊥[a]	High (approximately 50 times)	No (desirable feature)

[a] ‖ – parallel to planes/layers; ⊥ perpendicular to planes.

Table 2
Properties of polyether ketone (PEK) [17].

Properties	PEK
Density	1.30
Glass transition temperature T_g (°C)	152
Melting temperature T_m (°C)	373
Processing temperature (°C)	400
Crystallinity (%)	36
Tensile modulus (GPa)	4.0
Tensile strength (MPa)	105
Tensile elongation (%)	15
Impact strength (J/m)	55
Limiting oxygen index (%)	40
Estimated relative temperature index (°C)	280
Heat distortion temperature HDT (°C)	167

Table 3
Physical properties of thermo-graphite [18].

Properties	Thermo-graphite (TG)
Purity	
• Ash	0.18%
• Moisture	0.06%
Density	
• Bulk	0.13 g/cc
• Tap	0.18 g/cc
Specific surface area	25.9 m^2/g

2.3. Characterization of composites

2.3.1. Mechanical properties

The physical and mechanical strength characterization of various composites was done according to ASTM standards (Tables 5a and b).

2.3.2. Thermal conductivity (TC)

Thermal conductivity of few selected composites from series II was measured at room temperature, in-plane by Netzsch TCT416 instrument and through-plane by Netzsch Laserflash LFA447 instrument.

2.3.3. Tribological studies

The composites were evaluated for adhesive and abrasive wear performance as follows.

2.3.3.1. Adhesive wear studies. Sliding wear tests in adhesive wear mode were performed using a pin on disk configuration on a tribometer, supplied by CETR, USA (Fig. 2a). Cylindrical pin of 6.4 mm in diameter and length 4 mm was cut from a tensile bar. It was slid against a mild steel disk (Ra 0.1–0.2 μm) under constant