



Effect of the type, size and concentration of solid lubricants on the tribological properties of the polymer PEEK

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ABSTRACT

Poly-ether-ether-ketone (PEEK) is a high-performance, temperature-resistance polymer that is finding an increasing range of applications. In order to even enhance PEEK's mechanical and tribological properties particles of different compositions, shapes and sizes are added into its matrix. PEEK has already been combined with many different particles; however, very rarely MoS₂ and WS₂ – two state-of-the-art solid-lubricants – were added into the PEEK matrix. Furthermore, a comprehensive tribological study combining MoS₂ and WS₂ particles of different sizes and concentrations has not yet been reported. In this investigation we looked at the effect of micro- and nanosized MoS₂ and WS₂ particles in PEEK on the dry-sliding tribological behaviour against stainless steel (100Cr6). A non-conventional technique, i.e., the sintering of dry-pressed compacts, was used to prepare the PEEK composites. The results show that all the particles, irrespective of their composition and size, reduce the friction (up to 30%); however, the nanoscale particles require a higher concentration to form an effective low-friction tribofilm. The formation of a tribofilm is necessary to reduce the wear of all the composites (up to 51%); this is strongly promoted by the addition of nano- or microparticles of both the MoS₂ and WS₂ materials. In addition, the hardness, which is greatly increased by the addition of all the particles, significantly improves the wear behaviour. The results of XPS analyses showed that the oxidation occurs during tribological sliding, which reduces particles beneficial wear behaviour effects.

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1. Introduction

Polymers are taking an increasingly important position in almost every branch of industry; they are being used as the main material for a final product or simply as a replacement for other materials (e.g., carbon steel, stainless steel, titanium, aluminium, magnesium, brass, bronze, etc.). High-performance engineering plastics are used for the most demanding applications, such as gears, sealing rings, bushings, bearings, valves, etc. These polymers are required to withstand harsh operating conditions with high loads, high temperatures and long operating hours. Often, these polymers are considered in non-lubricated applications, where either the operating conditions and environmental demands or the nature of the application itself (e.g., sterility) restricts the use of lubrications. For such applications, the polymer itself, albeit with some difficulty, fulfils the required demands, especially when

the application in question includes dry-sliding movements. Poly-ether-ether-ketone (PEEK) is a high-performance polymer with good mechanical and promising tribological properties. However, PEEK's coefficients of friction in dry-sliding contacts can reach values that are not desired for such conditions and can promote higher wear and premature material failure. Therefore different particles are added into PEEK's matrix in order to improve its tribological and mechanical properties [1–7]. In non-lubricated environments, the performance of the materials mostly depends on the properties of the interfaces and surfaces, and their ability to form anti-wear and low-shear boundary films that in lubricated conditions are usually provided by lubricants and additives. This can also be achieved by using appropriate self-lubricating particles, such as molybdenum disulphide (MoS₂) and tungsten disulphide (WS₂). These two solid lubricants are well known for their low-friction properties and wear resistance [8–11], but they have not yet been extensively used in polymer composites, especially in combination with PEEK.

A literature review of PEEK composites indicated the importance of the particle size, the concentration and the transfer-film

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formation of the surfaces of the counterparts [6,12–14]. Also, the formation of a transfer film on the counterpart surfaces is believed to be the key to a low coefficient of friction and wear rate [15–17]. However, this information is still missing for the suggested MoS₂ and WS₂ particles in PEEK. Therefore, in this study we have used two different sizes – nano and micro – of MoS₂ and WS₂ particles, and four different concentrations. With different tribological, mechanical, chemical and optical analyses we were able to thoroughly assess the effect of different solid lubricants (MoS₂/WS₂), their size and concentration on the properties of the PEEK. Special attention was given to the transfer-film formation with respect to the particle type, size and concentration.

2. Experimental

2.1. Materials and preparation

All the samples used in this investigation were based on a poly-ether-ether-ketone (PEEK) matrix obtained from the company Victrex (VICOTE® 704, d50=8.5 µm, Victrex plc., Thornton Cleveleys, UK). For the composite samples four different particles were used: microMoS₂, nanoMoS₂, microWS₂ and nanoWS₂. The micro-sized molybdenum disulphide powder was obtained from Sigma Aldrich (Sigma-Aldrich, St. Louis, USA). The average particle size was around 2 µm, with a purity of 99%. The nano-sized molybdenum disulphide powder was obtained from Graphene Supermarket (Graphene Supermarket Co., USA). The average particle size was ~90 nm, with a purity of 99%. The micro-sized tungsten disulphide powder was also obtained from Graphene Supermarket (Graphene Supermarket Co., USA). The particle size was in the range 0.4–1.0 µm, with a purity of 99%. The nano-sized tungsten disulphide particles were obtained from the company NanoMaterials (NanoMaterials, Ltd., Yavne, Israel) in a fullerene-like shape. The fullerene-like WS₂ had a uniform, symmetrical, spherical structure of 20–100 layers with a diameter of 30–70 nm. The particles were used in four different concentrations, i.e., 0.5 wt%, 1 wt%, 2 wt% and 5 wt%, and were compared to the pure PEEK sample.

The samples were prepared by dry pressing the powder, followed by sintering. This procedure proved to be both time and cost efficient, and also showed a high potential for producing homogenous composite materials with a high flexibility of both the material and the processing parameters [18,19]. First, the pure PEEK powder and the different concentrations of particles were ultrasonically and magnetically mixed in ethanol for 5 min to achieve a homogenous suspension. The suspensions were dried (80–83 °C) to remove the ethanol and the powder was pressed in a disc-like shape with a diameter of 15 mm at a pressure of 100 MPa (PW 10, Paul-Otto Weber GmbH, Remshalden, Germany). The samples were inserted into a tube furnace (IJS, Slovenia) and heated at a temperature of 300 °C for 60 min, with a heating and cooling rate of 5 °C/min. Disc-like composite samples were embedded in universal embedding resin (Technovit 4071, Heraeus Kulzer GmbH, Germany) and, prior to the testing, were polished to a surface roughness of $Ra=0.030 \pm 0.005 \mu\text{m}$ (T8000, Hommelwerke GmbH, Schwenninger, Germany). The compositions of the samples and their denotations are presented in Table 1.

For the counterpart material used in our tribological tests a DIN 100Cr6 stainless steel was chosen, since it is a common material used for various mechanical components (bearings, gears, etc.) and it also commonly used in other tribological investigations. The counterpart sample was a flat-end cylinder with a diameter of 3 mm (Tinex, trgovska družba d.o.o., Šenčur, Slovenia). Before the tribological testing the cylinder surfaces were polished

Table 1

Compositions and denotations of the samples.

Particle type	Concentration, wt%	Denotation
Pure PEEK	–	PEEK
MicroMoS ₂	0.5; 1; 2; 5	M0.5; M1; M2; M5
NanoMoS ₂	0.5; 1; 2; 5	NanoM0.5; nanoM1; nanoM2; nanoM5
MicroWS ₂	0.5; 1; 2; 5	W0.5; W1; W2; W5
NanoWS ₂	0.5; 1; 2; 5	NanoW0.5; nanoW1; nanoW2; nanoW5

to the surface roughness $Ra=0.030 \pm 0.005 \mu\text{m}$ (T8000, Hommelwerke GmbH, Schwenninger, Germany).

2.2. Tribological tests

Since the test conditions can also have a major effect on the tribological properties [20,21] only one set of test conditions was used in order to focus only on the concentration, size and particles type. The test parameters were determined with preliminary tests and were kept the same throughout entire investigation, including our other work [18,19,22]. The tribological tests were performed on a CETR tribological tester (UMT-2, now Bruker, CA, USA) in a pin-on-disc configuration. The upper specimen (i.e., the pin) was a steel cylinder and the lower specimen (i.e., the disc) was a polymer sample. Tribological tests were conducted in a reciprocating sliding regime, with a stroke of 5 mm. The upper cylinder was loaded with a pressure of 1 MPa and the sliding velocity was 0.05 m/s. The test duration was 7 h with an overall sliding distance of 1260 m. The test duration was long enough to ensure a steady-state coefficient of friction for all the tested samples. All the tribological sliding tests were conducted in a dry-sliding contact (without additional lubrication) at 20 °C, $RH=30 \pm 5\%$.

The tests on each sample were performed at least four times to ensure a relevant statistical evaluation. The presented values for the coefficient of friction show an average value of the steady-state coefficients of friction for each sample, with the corresponding standard deviations.

2.3. Characterisation and analyses

Prior to the tribological testing, all the samples were characterised in terms of the particle distribution in the PEEK matrix and the hardness. The distribution of particles was evaluated with mapping-mode energy-dispersive X-ray spectroscopy (EDS). A scanning electron microscope (SEM; JEOL Ltd., Tokyo, Japan) combined with a light-element Si energy-dispersive X-ray detector (beryllium-window type EDS; Oxford Inst., Abingdon, UK), operated at an accelerating voltage of 20 kV was used for the evaluation.

The Vickers hardnesses of all the samples were measured on a Miniload 2 hardness tester (Leitz Miniload, Wild Leitz GmbH, Wetzlar, Germany). The hardness was measured with a standard Vickers indenter, with a load of 491 mN for an indentation loading time of 15 s. The average value of 20 measurements and their standard deviation are presented.

The wear rates and the corresponding 2D cross-sections were measured with white-light optical interferometry (Contour GT-K0, Bruker, Billerica, Massachusetts). The graphs show the average values of the normalised wear rates with the corresponding standard deviations. The worn surfaces were inspected with an optical microscope (Eclipse LV-150, Nikon, Tokyo, Japan) using 25× magnification and with a scanning electron microscope (SEM; JEOL JSM-T330A, JEOL Ltd., Tokyo, Japan) operated with an accelerating voltage of 10 kV and equipped with an Inca Energy data-processing unit (Oxford Inst., Analytical Ltd., Abingdon, UK). Representative worn surfaces are presented.

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