TRIBOLOGICAL BEHAVIOUR OF THE LOW AND HIGH VISCOSITY PEEK AT VARIOUS TESTING SCALES

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Abstract

Polyetheretherketone (PEEK) has become most attractive as a sliding bearing material in industrial applications, due to its excellent thermal stability, good friction and wear resistance. These properties promote the material to be used in so called high performance tribological applications. However, fundamental mechanisms governing friction and wear are not yet fully understood and neither is the influence of composition parameters. An important parameter is PEEK's viscosity during injection moulding which is heated up to semi-solid state, between its glass transition and melting temperature. It is not known to what extent the injection viscosity, related to the applied temperature profile, affects subsequent tribological features. This paper studies the friction and wear performance of low and high viscosity PEEK under dry reciprocating sliding contact. The tests were performed with small and large scale specimens under pin-on-plate and flat-on-flat configuration, respectively; to determine the transitions in tribological behaviour at different scales and to identify the applications limits. Tests were carried out at controlled atmosphere with 25 °C and a relative humidity of 50%. Parameters such as contact pressures and sliding speed were limited at 10 MPa and 20 mm/s, respectively; post mortem analyses were carried out by means of 2-D surface topography and optical microscopy. The results show that PEEK injected at high viscosity exhibits a tribological performance with a relatively high coefficient of friction and high wear rate compare to PEEK injected at low viscosity.

Keywords:
sliding bearing, polyetheretherketone PEEK, injection viscosity, friction and wear.

1 INTRODUCTION

Materials used in tribological applications are, for the most part, common materials used for general engineering applications. There are some materials designed specifically for bearings, ball-joints, sea-locks, crane guides and train boggies, characterised by high loads, low sliding velocities and large contact area. Polymers are used for dry sliding applications where the soft materials aid for self-lubricating properties. Polymers with such capabilities should be evaluated for precise tribological characteristics such as friction and wear rate.

The friction and wear rates are commonly obtained from small scale pin-on-disc, block-on-ring or flat-on-flat tests, using standard geometries. Small scale mechanical tests are preferred due there low cost or time and easy handling of the test specimens. These methods provide fundamental information about friction and wear mechanisms and are useful for preliminary material classification. However, the global characteristic of material in real scale is unknown unless the commencement of failure in real components.

Previous investigations shows [1] the selection material it's important than this require different factors or use a real criteria in the tribological characterization of the material under conditions that simulate practical functionality, such as contact geometries, contact pressures, environmental conditions, mechanical stiffness, etc. Consequently, these can give its tribological effect expressed on different geometries and on scales ranging from nanotribology up to teratribology [2].

For extrapolation of tests results towards real working conditions, Czichos [3] provided a scheme from 'field tests' over 'large-scale' simulation on real components to 'laboratory tests' where the same amount of energy concentration and thermal input is assumed. For polymers, the friction and wear properties change, due to transfer of wear debris and formation of a polymer film on the steel countersurface where the tribological performance may be altered. Thus special attention is needed testing polymers at different scales.

The present work is focuses on a comparative study of two different PEEK, low and high viscosity that are slid on a small-scale and large-scale tribotester under a certain contact pressure, sliding velocity and contact geometries. The effect of the manufacturing processing of those polymer low and high viscosity has influence on the tribological behaviour of the material. The relations between small-scale and large-scale test results are influenced by many factors. If the correlation between test configurations exists, they would provide more accurate design information. The steady-state sliding is influenced by transfer film formation and frictional heating. The results of the friction and wear performance is compared for PEEK, the high viscosity polymer exhibits a completely different tribological performance with a relatively high coefficient of friction and high wear rate compare to PEEK at low viscosity.

2 EXPERIMENTAL DETAILS

2.1 Test Materials

Polymers are used as wear materials, being commercially produced by injection moulding, extrusion are available in large samples. The samples are machined excluding molecular alignments effects at the surface due to the processing.

Polyetheretherketone (PEEK) is a tough semicrystalline thermoplastic polymer with attractive mechanical properties [4] and hence the polymer is currently finding use as matrix for high performance composites and in engineering applications. PEEK is an attractive bearing material as it is comparatively tough and fatigue resistant, even at elevated temperatures. At temperatures up to 250 °C the creep rate of PEEK is relatively low and thus it may be used to construct dimensionally stable components. PEEK represents a class of semicrystalline engineering thermoplastics with outstanding thermal and chemical resistance properties. Two types of PEEK were used as sliding material, for instance, PEEK-low viscosity and PEEK-high viscosity which are characterized from the melt process during the injection moulding. Mechanical properties of the tests materials are given in a table 1. The principal purpose of the injection unit for moulding a crystalline material is to deliver to the mold the necessary amount of a homogeneous melt (with no degraded material). The rules of construction of the injection unit are then dependent on the moulding material requirements in term of thermal behaviour and heat needed. The main point is to take into account of crystalline material has the thermal stability at melt temperature, to avoid the degradation of the material.

Table 1. Mechanical properties of the test material [5]

Material	Density (g/cm^3)	E-Modulus (MPa)	Thermal Expansion 10^{-4} (W/mK)	Melting Temperature (°C)
PEEK-Low viscosity	1.3	3700	0.6	340
PEEK-High viscosity	1.3	3500	0.6	340
100Cr_6	7.8	199000	0.15	1539

The counterfaces plate consist of steel plates 100 Cr_6 (DIN 1.3505) with hardness 147 HB, yield strength R_e= 1324 MPa, tensile strength R_m: 1640 MPa and chemical composition (wt %): C=0.93-1.05, Si= 0.15-0.35, Mn= 0.25-0.45, P< 0.025, S< 0.015, Cr= 1.35-1.60, Mo< 10, Al< 0.050, Cu< 0.30, O< 0.0015. Before each test, the surfaces were ground to an average surface roughness R_a= 0.20 µm measured parallel to the sliding direction. The measurements were conducted using a Surfascan 3D reference tester, manufactured by Hommel somicronic with a stylus S6T (radius 2 µm, angle 90°). R_a is calculated according to DIN EN ISO 4288 with an assessment length l_t = 4.00 mm and cut off λ_c = 0.80 mm for 0.1 µm < R_a ≤ 2 µm; l_t = 15.00 mm and λ_c = 2.50 mm for 2 µm < R_a ≤ 10 µm. Pior to each test the steel surfaces were cleaned with a cleaning solvent (petroleum ether) under ultrasonic vibration and then with acetone.

The test scheme and the samples were prepared in accordance with the large and small scale equipments. Parameters like normal stroke, sliding distance, velocity and normal load is given in Table 2. The applied test conditions on both large-scale and small-scale tests are summarized for understanding the difference in scale for both the tests.

Table 2. Tests conditions for small scale and large scale

Test parameter	Large-scale test	Small-scale test
Dimension of the polymer samples	40mm x 40mm x 40mm	4 mm x 4mm x 4mm
Dimension of the countersurface samples	200 mm x 80 mm x 19 mm	Flat disk: Ø: 24 mm h: 7.9 mm
Sliding stroke	80 mm	8 mm
Normal Load	16 kN	16 Kg
Ambient Temperature	25 °C	25 °C
Humidity	50%	50%

2.2 Test equipment

The *large-scale test-rig* (Figure 1a) flat on flat tribotester is used for investigating the effect of overload on large contact area related for practical applications of polymers as bearing materials. The test-rig is built on a fatigue rated load-frame with 200 kN capacity. The hydraulic actuator of this frame provides the reciprocating movement of the test specimens and the bed of the test frame is mounted with the load assembly. Two steel counter-faces (2) are mounted (bolt connection) on a central sliding block (1). This central sliding block is connected to the actuator of the load frame. The sliding block moves in the vertical direction and slides against the two specimens (5) placed in holders (4). The maximum normal load which can be applied on the friction specimens is 225 KN. The test material (5) and the holders (4) are held in (vertical) position by the reaction fork (3). Wear of the friction material is compensated by horizontal movement of the holders (4) with respect to the reaction fork (3). The reaction fork is constructed in such fashion that it can also hold the test medium. The normal load is applied by a piston in the horizontal position, pressing the polymer samples in contact with their countersurfaces under 10 MPa. The vertical displacement of the polymer specimens towards their counterface is continuously measured by displacement transducer. As measurements are influenced by thermal expansion (counteracting the wear signal), the real wear (material loss) is determined by weighing the polymer sample before and after testing and is compared to thickness reduction. The sliding temperature is measured by a K-type thermocouple positioned at 10 mm beneath the steel surface.

The coefficient of friction μ is determined by the ratio of horizontal forced and the applied normal load and wear rates are determined from weight measurements before and after testing. The later values are compared to dimensional measurements of thickness reduction (large-scale).

The total friction force (F_{FR}) is measured by the force transducer. The coefficient of friction (μ) is calculated from the measured friction force (F_{FR}) and the normal force (F_N) according to equation 1, where in the factor of two is used because the friction force is the aggregate of the two friction specimens. From the results of every logged cycle the maximum of the coefficient of friction (static coefficient of friction) and the coefficient of friction at the first pass through the centre of the stroke (dynamic coefficient of friction) are calculated. In the flat on flat sliding the measurements were made for the tangential friction force (FT) for a given a Normal load (FN). The

ratio of FT/FN forces defined as the coefficient of friction (μ), which has two components the static (μstat) and a dynamic friction (μdyn). From the literature it is obvious that the static friction co-efficient is higher than the dynamic co-efficient of friction. The values of friction force in the beginning of sliding will be considered for calculating the static coefficient of friction

$$\mu = \frac{F_{FR}}{2 \cdot F_N} \qquad (1)$$

The *small-scale tests* (Figure 1b) were carried out under a linear reciprocating sliding contact using the UMT (CETR) tribometer. The test samples were PEEK pins of low and high viscosity under a Hertzian contact pressure of 10 MPa at a sliding velocity of 20 mm/s. The counterparts were 100 Cr_6 steel disks. Wear rates of the polymer samples are determined by weight loss before and after the sliding test. The evolution of coefficient of friction through time was recorded.

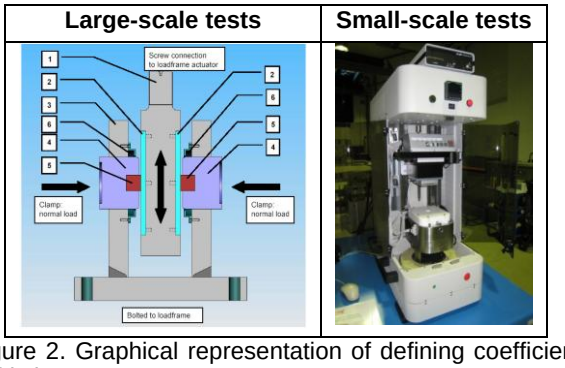

Figure 2. Graphical representation of defining coefficient of friction.

2.3 Data logging and calculations

All signals are logged for 8 seconds out of every minute in 'block'-files with a sample frequency of 200 Hz. At the start of the test the central sliding block is at the centre of the stroke and at first it moves upwards. The velocity of the linear reciprocating motion is kept constant.

From the results of every logged cycle the static and dynamic coefficient of friction are calculated. The static coefficient of friction is defined as the maximum of the absolute value of COF in t_{s1} (interval of 2 second after zero cross) whereas dynamic coefficient of friction is defined as the average of the absolute value of coefficient of friction at mid of stroke in t_{s2} (interval of 4 seconds), see figure 2.

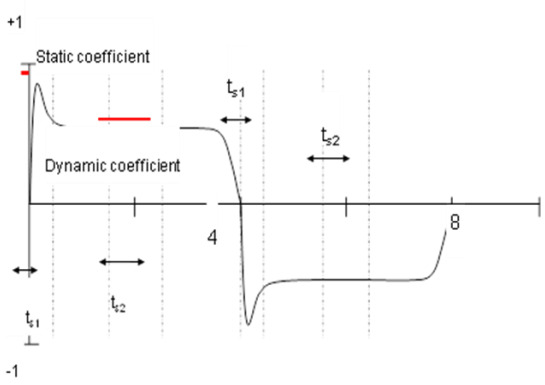

Figure 2. Graphical representation of defining coefficient of friction.

3. RESULTS AND DISCUSSION

3.1 Friction measurements

The small-scale and large-scale test results for coefficient of friction and wear rates are summarised in Table 3 against the applied normal and sliding velocity. From table 3 can be seen that the coefficient of friction for large-scale testing is lower for both low and high viscosity PEEK on comparing with small-scale testing. The difference in frictional properties might be attributed by parameters such as, real contact area of a polymer contact which increases during deformation, generated heat per unit of macroscopic scales. Additionally, it has been already reported that increase in real contact area and decreased mechanical strength has great influences in the friction force and this applies for high performance polymers such as polyimides (PIs), polyetehretherketone (PEEK) or polyphenylene sulphide [6]. Moreover, the tribological behaviour is also affected by many parameters, such as thermal heating, contact conditions and geometry [7].

Table 3. Coefficients of friction and wear rates of tests condition

10 Mpa - 20 mm/s						
	Larg -Scale Test				Small-Scale Test	
	μ (-)	W [10^{-6} mm^3/Nm]			μ (-)	W [10^{-6} mm^3/Nm]
PEEK-LV	0.38	8.16E-06		PEEK-LV	0.45	15.10E-06
PEEK-HV	0.43	11.63E-06		PEEK-HV	0.46	10.90E-06

From the figure 3 (a) and (b) can be observed general tendencies of coefficient of friction for PEEK- low and high viscosity at sliding velocity of 20 mm/s in large-scale testing; the high viscosity PEEK has relatively higher coefficient of friction. The surface behaviour as well as the integrity of the polymer bulk properties are important because the materials becomes highly viscous due to the intrensic property. Plasticisation helps to have transfer layer thus having appropriate self lubricating property of the polymer surface that contributes to low friction and acceptable wear rates as long as the deformation is controlled by reinforcing structure.

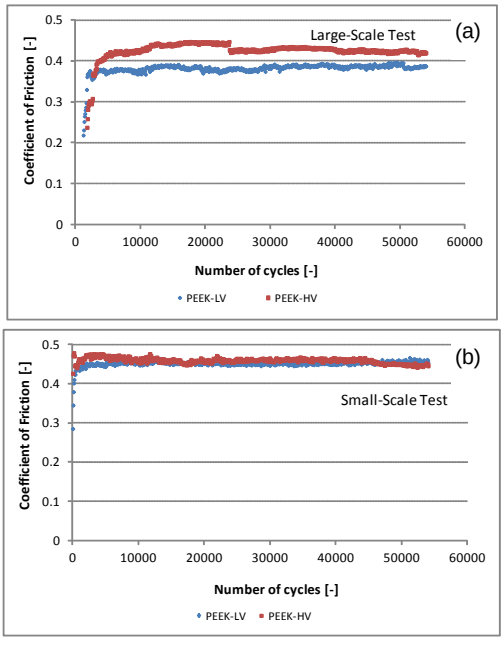

Figure 3. Comparison of on-line measurements of the coefficient of friction between (a) large-scale and (b) small-scale test at a contact pressure of 10 MPa and sliding velocity of 20 mm/s.

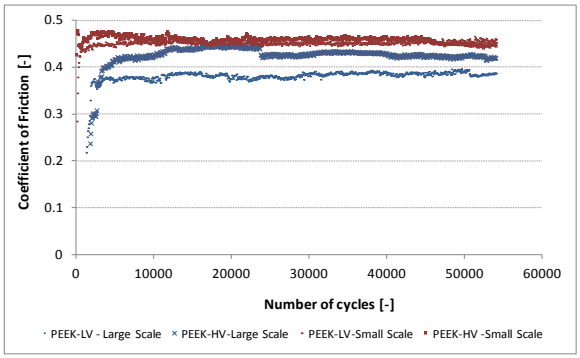

Figure 4. Comparison of on-line measurements of the coefficient of friction all together large-scale and small-scale test at a contact pressure of 10 MPa, and sliding velocity of 20 mm/s.

The PEEK- low viscosity and high viscosity were slid against steel counterfaces over the large scale, investigating the evolution of the friction as shown in Figure 4. After a transient behaviour during the running-in, the coefficient of friction stabilizes. Steady-state condition is constant for the PEEK-low viscosity but for the PEEK-high viscosity is not the same, there is a decrease in friction coefficient towards a steady-state. Zsidai et al [7] obtained under 100 N normal load, the coefficient of friction rises from 0.2 towards 0.3 on smooth surfaces while it frequently stabilises at 0.2 on rough roughness. Stable friction is attributed to the formation of a thin transfer film on the countersurface, see in the section 3.2 microscopy optical.

3.2 Wear rates measurements

The wear rate is determined from the weight or dimensional measurements. Figure 5 illustrates the wear rate at the small-scale is higher than large-scale. The large-scale wear samples have the lowest edge effects and stress concentrations. Additionally, the moveability of generated wear debris into the contact zone is less effective. Likewise, it is possible to determine the real wear lifetime of polymer components in laboratory scale within conditions implied by practice. Although its results are more close to practice, a large scale test rig demands high dimensions, weight, stiffness and the manipulation of the test pieces is more difficult and the heat sink for samples at large scale is larger and has more space to transfer material between the countersurface and the polymer material.

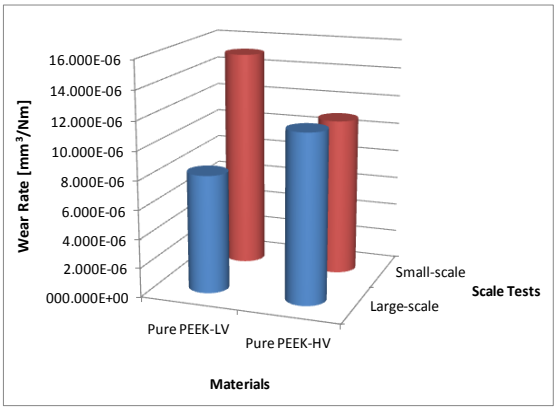

Figure 5.Comparison of the wear rate of the material measured by the material loss at large-scale and small-scale test with contact pressure of 10 MPa and sliding velocity of 20 mm/s.

3.3 Wear Mechanisms

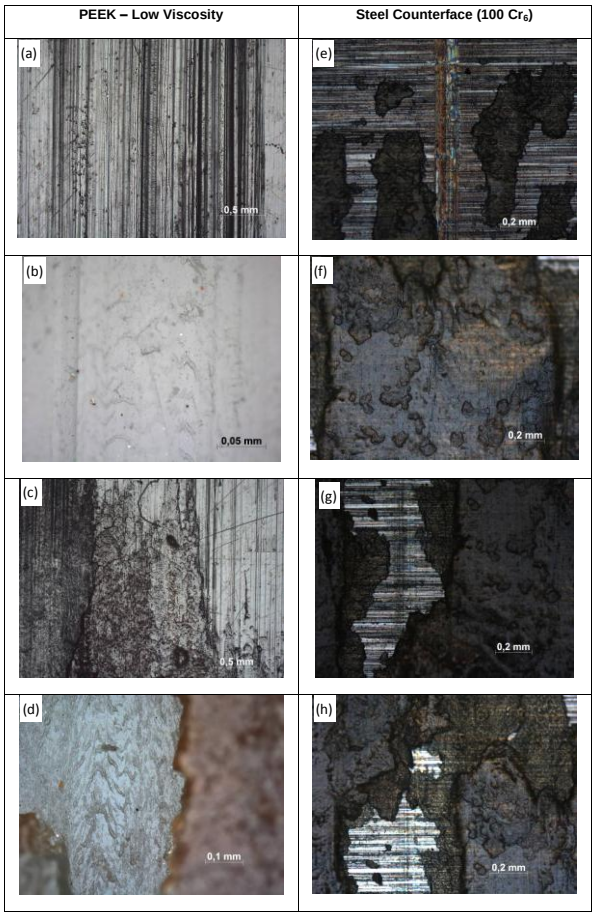

Figure 6. Optical Microscopy of Pure PEEK-low viscosity and steel counterface after sliding distance under contact pressure of 10 MPa and sliding velocity of 20 mm/s.

The results obtained from the optical microscopy of the countersurface and PEEK- low viscosity are presented in the Figure 6 for the large-scale test at a contact pressure of 10 MPa and sliding velocity of 20 mm/s thus evidencing different wear process. Figure 6 (a) and (b) shows a combination of adhesive, abrasive and cohesive failures leading to debris detachment in the polymer. In previous investigation [8] was founded that interfacial bond between the polymer and the transferred film is stronger than the cohesive strength. The deposition of PEEK is uniformly distributed over the sliding area. In the figure 6 (c), (d), (e) and (g) a layer in the polymer surface can be observed, these layers are called third-body layers or films and may be composed of wear debris from either polymer surface or steel surface. When wear debris from one surface coats the counterface, the film is called transfer films or layer. Transfer films are always thick and discontinuous with debris particles progressively growing around fixed nucleus or containing agglomerated plasticized polymer, reducing the performance through the vibration within the sliding interface, contributing to stick-slip Figure 6 (b).

As can be seen in Figure 6 (c), PEEK-low viscosity has material removal from a surface via plastic deformation during abrasion, these occur by several deformation modes which include ploughing. The ploughing occurs in a series of grooves as results of the plastic flow of the softer material. The ploughing process also causes surface and subsurface plastic deformation and may

contribute to the nucleation of surface and subsurface cracks.

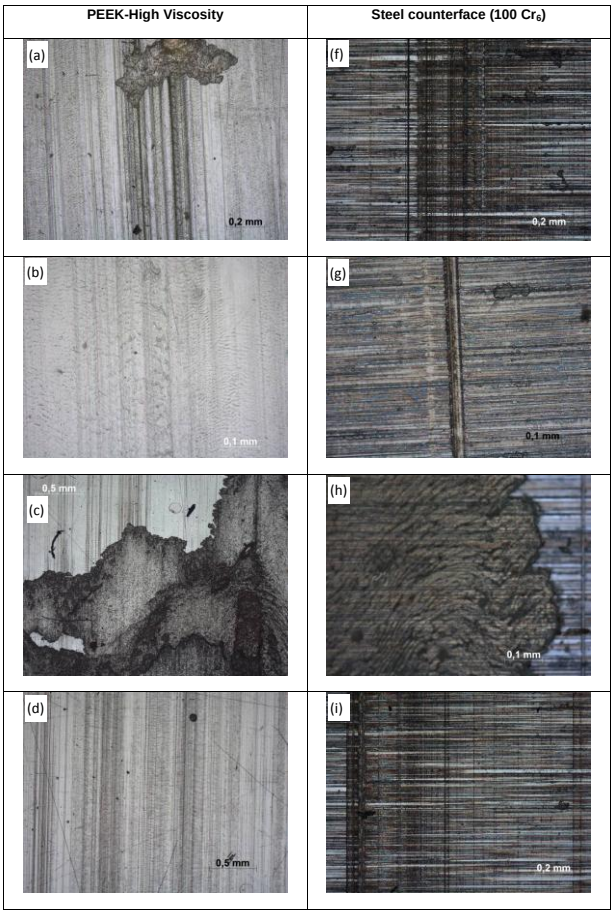

Figure 7. Optical Microscopy of PEEK-high viscosity and steel counterface after de sliding distance under contact pressure of 10 MPa and sliding velocity of 20 mm/s.

PEEK- high viscosity surfaces are examined by means of optical microscopy in figure 7. The presence of polymer on the steel counterface can be observed as a film. It is vastly found on the middle of the contact zone, see figure 7 (c) and (a). Abrasive wear patterns parallel to the sliding direction can be seen in the polymer; figure 7 (d). The re-adhered wear debris film polymer (from the polymer to the steel and from the steel to the polymer) is observed in the steel counterface having a beige colour, see Figure 7 (h) and (j). The appearance of the PEEK-high viscosity sliding surface is different from its original look, by having a dull aspect change after sliding.

It is worth nothing that the PEEK-high viscosity is more prone to be adhered to the steel counterface thus presenting higher stick-slip phenomenon. The figure 7 (e) shows stick-slip characteristics on the PEEK-high viscosity surface.

The main differences in transfer layer and polymer surface aspects in large-scale behaviour are thus attributed to mechanical effects such as wear debris circulation in the interface and the high or low viscosity of the polymer.

Creep and visco-elastic deformation influence under the contact conditions should be further elucidated for having a better understanding of how the friction and wear could be altered on macro and micro scales.

4. CONCLUSIONS

In order to select a material for a given bearing applications, the consultation of tribological literature or technical data is not enough. Friction and wear properties depend on the layout of the tribological system with specific contact geometry, normal load and sliding velocity. In conventional tribotesting, small-tests are mainly used because of their cost and time effectiveness and the ease of handling little samples.

Large-scale and small-scale polyeterheretherketone (PEEK) specimens have been worn for investigating the friction and wear behaviour. The large-scale test has shown lower coefficient of friction and wear than small-scale. Nevertheless, the manufacturing process of the polymer has significant influence. The wear behaviour of the test samples with small and large contact areas is significantly different for PEEK-low viscosity, with lower specific wear for large scales due to the edge effects and transfer layer formation. Furthermore, the differences depending on the testing scale can be attributed to contact stress concentrations and the limited wear debris mobility within large contact areas, promoting a homogeneous film formation onto the polymer surface.

5. ACKNOWLEDGEMENTS

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