

Wear Behavior of PEEK-Based Composites Designed for Water Lubrication Bearings

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ABSTRACT

The aim of this research was to study the tribological behavior and properties of antifriction polyetheretherketone (PEEK) based composites loaded with solid lubricant particles as well as carbon fibers under both dry friction and water lubrication conditions. The investigated tribological properties varied due to a change in the pattern of the formation and adherence of a transfer film (TF) on the steel counterpart surface. Under the water lubrication conditions, a wear rate increased for the composite loaded with both polytetrafluoroethylene (PTFE) and carbon fibers (CF) due to the suppression of the adherence of a transfer film on the steel counterpart by protruding carbon fibers above the sliding surface, followed by abrasive damage to both rubbed bodies. Therefore, they were not recommended for use in water lubrication bearings. Another composite, loaded with both PTFE and molybdenum disulfide (MoS₂) was suggested for using in such bearings according to the 'ball-on-disk' scheme under mixed dry friction/water lubrication conditions.

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1. INTRODUCTION

Bearings operated under water lubrication conditions have found wide application in shipbuilding due to their high both environmental friendliness and damping properties, as well as simple design and long-term service life [1–3].

During operation, such structural components are not always provided with the required lubrication. In some case, its deficiency or complete absence may take place [4]; so, it is of actuality to develop water lubrication bearings with high wear resistance both in the presence of a lubricating medium and under dry friction conditions.

Polyetheretherketone (PEEK) is a semi-crystalline polymer classified as high-performance thermoplastics (HPP). PEEK molecules are relatively rigid due to the presence of aromatic (benzene) rings. In addition, PEEK is inherently weakly reactive because of the delocalization of higher orbital electrons along the entire macromolecules [5]. As a result, PEEK is widely applied as a structural material for both rolling contact [6,7] and plain [8] bearings.

To improve the tribological properties of PEEK, various types of fillers are used: carbon fibers (CFs) and glass fibers (GFs); solid lubricant particles (polytetrafluoroethylene, PTFE), molybdenum disulfide (MoS_2), graphite (Gr); nanoparticles of inorganic materials (SiO_2 , Al_2O_3 , etc.), as well as carbon nanotubes (CNT). They are loaded both individually and in various combinations (fibers/solid lubricant; fibers/nanoparticles; solid lubricant/nanoparticles; fibers/solid lubricant/nanoparticles, etc.) [9–12]. However, regardless of the loaded filler types, the formation of a transfer film (TF) and its reliable adherence to the surface of a polymer composite or a metal counterpart is an invariable condition for enhancing wear resistance under the dry friction conditions [13].

Recently, interest has increased in studying the tribological properties of PEEK-based composites under water lubrication conditions [14, 15]. Particular features of such bearings are: i) water does not contribute to the formation of a lubricating film and prevents the TF formation/adherence; ii) water can exert a corrosive effect on metals [16]. As an instance, Z. Guan et al. [17] investigated the tribological properties of the PEEK/30%CF composite when sliding against stainless steel. It was shown that at an extremely low coefficient of friction (CoF) of ~ 0.03 , a wear rate (WR) were registered $1.66 \times 10^{-8} \text{ mm}^3/\text{Nm}$ for the composite, while it was equal to $6.40 \times 10^{-9} \text{ mm}^3/\text{Nm}$ for the steel counterpart.

According to [18], the PEEK/CF composites can wear out a counterpart that is significantly superior in hardness, in particular it took place for sintered silicon nitride ceramics under the water lubrication conditions.

C. Gao et al. [19] studied the effect of the addition of copper nanowires on the tribological properties of PEEK filled with 10 vol.% short SFs (SCF) and 8 vol.% Gr flakes. The latter were aimed at enhancing the TF formation under the water lubrication conditions. As a result, a composite loaded with 0.5 vol.% copper nanowires possessed the best tribological characteristics. According to the authors of [19], the addition of copper nanowires can promote the TF formation and adherence during the friction process. In addition, copper nanowires enable to 'heal' the sliding surfaces.

Despite the reported results of some previous studies, the TF formation mechanism remains incompletely understood for PEEK-based composites under water lubrication conditions. In addition, these tribological tests were predominantly carried out in dry tribological contacts, paying less attention to comparison with an aqueous lubricated environment. This limits the full understanding of the tribological processes that develop in water lubrication polymer bearings. Thus, the aim of this research was to study the tribological behavior and properties of antifriction wear-resistant PEEK-based composites filled with solid lubricant particles and CFs under both dry friction and water lubrication conditions.

2. EXPERIMENTAL

2.1 Preparation of specimens

The '450PF' PEEK powder (Victrex, UK) with a particle size of $\sim 50 \mu\text{m}$ was used with the following fillers in the specified cases: i) 'F4-PN20' solid lubricating PTFE particles (Rufon, Russia) with a size of about $20 \mu\text{m}$, ii) MoS_2 ones (Climax Molybdenum, USA) with a size of $\sim 10 \mu\text{m}$, iii) chopped CFs (CCFs) (ZUKM, Russia) with a diameter of $6\text{--}8 \mu\text{m}$ and a length of $\sim 200 \mu\text{m}$ as a reinforcement.

The PEEK powder and the fillers were mixed in ethanol with a 'T 18 digital ULTRA-TURRAX' mixer (IKA Werke, Germany). Specimens were fabricated by hot pressing at a sintering temperature of $400 \text{ }^\circ\text{C}$ and a pressure of 10 MPa . Their compositions and designations are given in Table 1.

2.2 Test conditions

For the PEEK-based composites, both CoF levels and WR values were evaluated according to the ‘ball-on-disk’ scheme with a ‘CSEM CH-2000’ tribometer (CSEM, Switzerland). In addition to the dry friction conditions, distilled water was used as a lubrication medium. A GCr15 bearing steel ball 6 mm in diameter was used as a counterpart. The tests were carried out at a load of 10 N and a linear sliding speed of 0.4 m/s. A sliding distance was 3 km. The volumetric wear was assessed from wear track profiles using an ‘Alpha-Step IQ’ contact profilometer (KLA-Tencor, USA). The WR values were calculated according to Equation (1):

$$WR = \frac{V}{F \times L} \left(\frac{mm^3}{N} \times m \right) \quad (1)$$

where V is the worn material volume measured with the stylus profilometer (mm^3), F is the load (N), and L is the sliding distance (m).

The topography of the wear track surfaces was analyzed using a ‘Neophot 2’ optical microscope (Carl Zeiss, Jenna, Germany) equipped with a ‘Canon EOS 550D’ digital camera (Canon Inc., Japan).

Table 1. Designations of the specimens and their compositions.

Designations	Filler content, wt %			
	PEEK	PTFE	MoS ₂	CF
PEEK	100	–	–	–
PEEK/PTFE	90	10	–	–
PEEK/MoS ₂	90	–	10	–
PEEK/PTFE/MoS ₂ -A	89.5	10	0.5	–
PEEK/PTFE/MoS ₂ -B	85	10	5	–
PEEK/PTFE/MoS ₂ -C	80	10	10	–
PEEK/PTFE/CF	65	10	–	25
PEEK/CF	90	–	–	10

3. RESULTS AND DISCUSSION

The CoF versus sliding distance dependences under the dry friction conditions are presented in Fig. 1. It could be concluded that neat PEEK

was characterized by the maximum CoF level, which also corresponded to its high-frequency (HF) oscillation. For neat PEEK, the CoF kinetics was characterized by abrupt drops over the entire sliding distance, with a gradual increase from 0.2 up to 0.3. This fact testified to the wear process instability.

For the PEEK/MoS₂ composite, a pronounced low frequency (LF) CoF oscillation was observed at a distance of up to 1 km, then its value was fairly constant at about 0.23.

The PEEK/PTFE, PEEK/PTFE/MoS₂-A and PEEK/PTFE/CF composites had consistently low CoF levels (within 0.11) over the entire sliding distance.

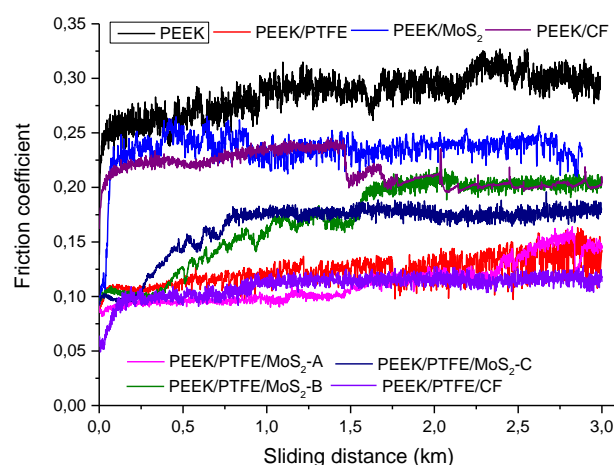


Fig. 1. The CoF versus sliding distance dependences for neat PEEK and its composites under the dry friction.

For the PEEK/CF composite, the mean CoF value gradually increased up to 0.24 (having a low HF oscillation level) till a distance of 1.5 km, then decreasing down to ~0.19.

CoF levels of both PEEK/PTFE/MoS₂-B and PEEK/PTFE/MoS₂-C composites rapidly enhanced from 0.10 up to 0.17 at distances of up to 1 km, which was accompanied by the pronounced HF oscillation (compared to neat PEEK and the PEEK/MoS₂ composite). After that, they were relatively stable (0.20 and 0.16, respectively).

Noted that, if the CoF level within 0.1 was taken as a characteristic of the antifriction properties of the studied composites, then the only PEEK/PTFE, PEEK/PTFE/MoS₂-A and PEEK/PTFE/CF ones fall under this definition.

Under the dry friction conditions, the PEEK/MoS₂ composite showed the highest WR value, while it decreased by ~3 times down to 4.1×10^{-6} mm³/N×m in distilled water. At the same time, the CoF levels remained equally high of >0.23 in both tests, while the average CoF value was even higher under the water lubrication conditions.

The PEEK/PTFE/MoS₂-A composite possessed the lowest WR value of 0.6×10^{-6} mm³/N×m under the dry friction conditions. Raising the MoS₂ content in the composites up to 5 and 10% (the PEEK/PTFE/MoS₂-B and PEEK/PTFE/MoS₂-C ones, respectively) gave rise to an increase in their WR values, but they significantly decreased in distilled water.

The lowest WR value of 1.7×10^{-6} mm³/N×m was observed for the PEEK/PTFE/MoS₂-B composite under the water lubrication conditions. Note that CoF levels reduced by 1.5–2.0 times for all MoS₂ and PTFE-containing composites (the PEEK/PTFE/MoS₂-A, PEEK/PTFE/MoS₂-B, PEEK/PTFE/MoS₂-C ones) under the water lubrication conditions compared to those in the dry friction case.

An extremely unexpected result was observed for the composites loaded with CFs. Under the dry friction conditions, the PEEK/PTFE/CF one had a low WR value of 2.5×10^{-6} mm³/N×m, which enhanced by more than 12 times under the water lubrication conditions with an increase in the CoF level by 1.5 times (compared to the dry friction case). For the PEEK/CF composite, a similar WR trend was observed, but the CoF level, on the contrary, decreased by 1.5 times compared to the dry friction conditions.

In general, it could be concluded that the water lubrication leveled out wear resistance of the composites relative to that for neat PEEK, with the exception of the CF-reinforced ones. Nevertheless, a clear correlation between the WR values and the CoF levels was not found.

Fig. 4 shows optical micrographs of the steel counterpart, while the wear track surfaces and their profiles after the tribological tests against the composites are presented in Fig. 5 for the dry friction conditions.

A TF formed on the counterpart surface during friction against the PEEK/PTFE composite (Fig. 4 (a)). It was quite thin (optically translucent) and fragmented, but evidently reflected the real contact area. Due to the low WR value, the wear track profile did not possess the shape of a semicircular valley (Fig. 5 (e)), and its topography could be characterized as smooth without pronounced furrows (Fig. 5 (a)).

After friction against the PEEK/PTFE/MoS₂-A composite, a TF was observed on the counterpart surface as well (Fig. 4 (b)). However, it was visually thinner and more uniform than that in the PEEK/PTFE case due to the lower WR value for the PEEK/PTFE/MoS₂-A composite (compared to the PEEK/PTFE one), since the TF actually formed from polymer debris.

Despite the difference in the WR values by a factor of two, the topographies of the wear track surfaces were visually similar for the PEEK/PTFE and PEEK/PTFE/MoS₂-A composites (Fig. 5, (a and b)). As mentioned above, both wear track profiles (Fig. 5, (e and f)) did not have the shape of a semicircular valley because of the low WR values.

After sliding against the composite with the higher MoS₂ content (namely, the PEEK/PTFE/MoS₂-B one, Fig. 4 (c)), two regions could be distinguished in the contact zone of the counterpart surface. The upper part was solid and occupied half of the contact area. It was characterized by a bright shade. In fact, this was the counterpart surface that did not experience a significant wear. On the bottom of the contact zone, a TF could be visually observed (Fig. 4 (c)), similar to the results of the tribological tests of the two previous composites. MoS₂ particles were evident on its wear track surface (Fig. 5 (c)), while its profile was smoother and appeared as a semicircular valley damaged by several furrows (Fig. 5 (g)). The reason was the fact that the composite was significantly inferior in wear resistance to both ones described above (namely, the PEEK/PTFE and PEEK/PTFE/MoS₂-A specimens).

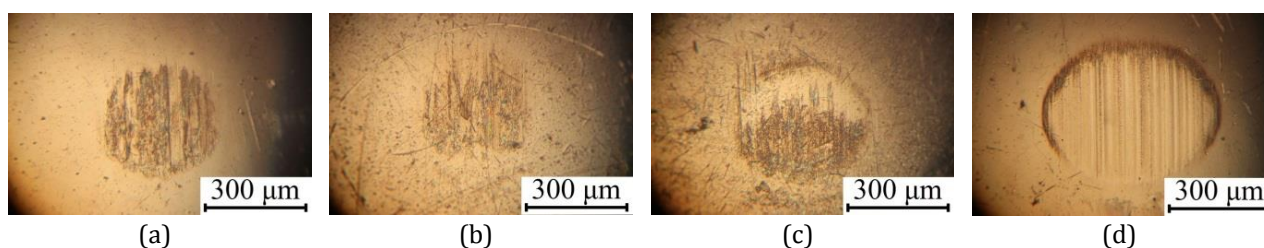


Fig. 4. The optical images of the steel counterpart surface after the tribological tests against the PEEK/PTFE (a), PEEK/PTFE/MoS₂-A (b), PEEK/PTFE/MoS₂-B (c) and PEEK/PTFE/CF(d) composites; dry friction.

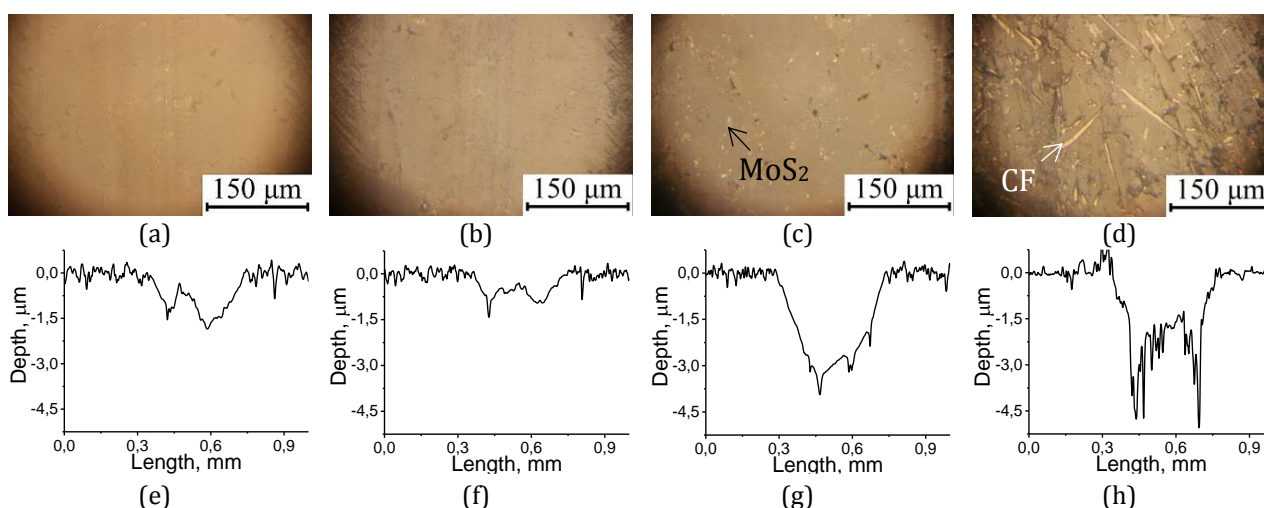


Fig. 5. The optical images of the wear track surfaces and their profiles in the PEEK/PTFE (a, e), PEEK/PTFE/MoS₂-A (b, f), PEEK/PTFE/MoS₂-B (c, g) and PEEK/PTFE/CF(d, h) composites; dry friction.

After the tribological test against the PEEK/PTFE/CF composite, a TF was found on the counterpart surface as a result of the debris adherence (Fig. 4 (d)), which was consistent with the increased WR value (Fig. 5 (h)). On the TF surface, a plowing effect of CFs, as constituents of the composite, was observed. At the same time, its WR value was comparable to that of the PEEK/PTFE/MoS₂-B composite (Fig. 4 (c)), although the CoF level was rather low of 0.11 (which was comparable to those of the other two PEEK/PTFE and PEEK/PTFE/MoS₂-A specimens, Fig. 1).

Individual CFs were expectedly observed on the wear track surface of the PEEK/PTFE/CF composite (Fig. 5 (d)). At the same time, the central part of its profile was similar to an inverted valley (Fig. 5, h), additionally damaged by both small and large furrows. This topology was highly atypical, most likely caused by the abrasive action of CFs 'torn out' from the composite. Despite the development of these processes, the PEEK/PTFE/CF composite possessed low both CoF level and WR value. Previously, the authors interpreted this effect as a consequence of the tribological layer (TL) formation on the wear track surface when studying antifriction composites based on polyetherimide (PEI) [20,21].

The above differences in the kinetics of the changes in both CoF levels and WR values for the dry friction and water lubrication conditions manifested themselves in the variations in the topography of the wear track surfaces (Figs. 6 and 7). Noted that the visual difference was more characteristic of the steel counterpart surface (Fig. 6).

After friction against the PEEK/PTFE composite, the contact zone on the counterpart surface had a complex structure (Fig. 6 (a)). In its central part, there was a TF damaged by longitudinal grooves (oriented along the sliding direction), while a thin TF having a brown tint was observed on the periphery (above and below the TF). At the same time, any deep furrows were not found on the wear track surface (Fig. 7 (a)), and its profile had the shape of a trapezoid (but not a semicircular valley, Fig. 7 (f)). This corresponded well to the low CoF level (due to the presence of the water lubrication medium). At the same time, the WR value was high (slightly less than that for neat PEEK). Thus, the presence of PTFE solid lubricant particles in distilled water led to rising the WR value

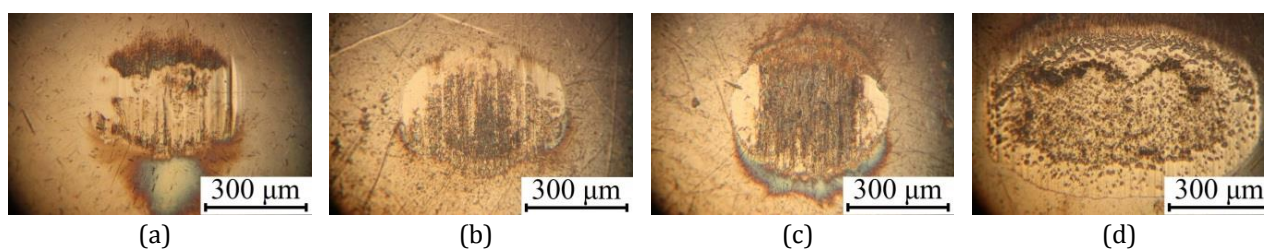


Fig. 6. The optical images of steel counterpart surface after testing against PEEK/PTFE (a), PEEK/PTFE/MoS₂-A (b), PEEK/PTFE/MoS₂-B (c) и PEEK/PTFE/CF(d) composites; water lubrication.

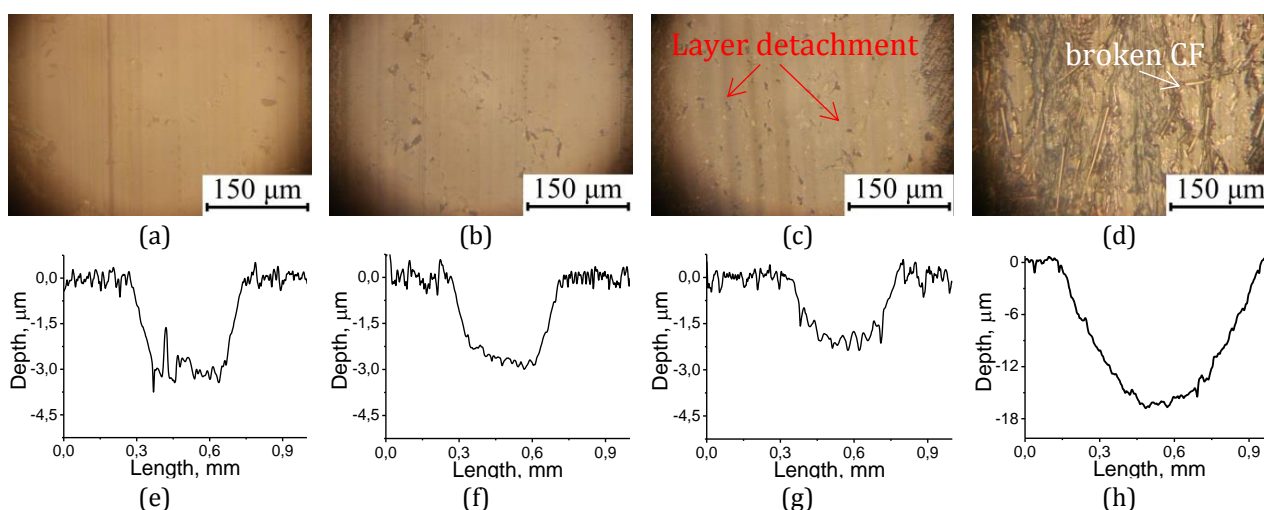


Fig. 7. The optical images of wear track surfaces and their profiles in the PEEK/PTFE (a, e), PEEK/PTFE/MoS₂-A (b,f), PEEK/PTFE/MoS₂-B (c,g) и PEEK/PTFE/CF(d,h) composites; water lubrication.

The WR value of the PEEK/PTFE/MoS₂-A composite was similar to that for the PEEK/PTFE one (Fig. 3), as was the CoF level (Fig. 2). No pronounced grooves were observed on the wear track surface, (Fig. 7, b), and it could be also characterized as trapezoidal (Fig. 7, f). So, the presence of the small amount (0.5%) of MoS₂ did not cause a pronounced change in the tribological behavior of the PTFE-filled PEEK-based composite under the water lubrication conditions in contrast to the dry friction case.

Increasing the MoS₂ content up to 5% did not drastically change the TF appearance on the steel counterpart (Fig. 6 (c)). However, the wear track surface on the PEEK/PTFE/MoS₂-B composite turned out to be covered with longitudinal stripes (oriented along the sliding direction, Fig. 7 (c)). The latter could result from rolling debris on the wear track surface of the composite. The presence of MoS₂ as an active element could additionally contribute to this phenomenon. In this case, the lowest WR value (Fig. 3) was observed, much like it was the CoF level (Fig. 2). Noted that the wear track profile had a more familiar shape of a semicircular valley (Fig. 7 (g)),

whose smoothness was disturbed only by the aforementioned bands. Of all the studied PEEK-based composites, this particular specimen showed the lowest WR value under the water lubrication conditions.

The maximum WR value was observed after friction against the PEEK/PTFE/CF composite (Fig. 3). In this case, a large oval-shaped TF was found on the counterpart surface (Fig. 6 (d)). It possessed a porous structure and did not look like a continuous one. Nevertheless, there were no longitudinal grooves caused by the cutting/ploughing action of CFs (protruding above the wear track surface, Fig. 7 (d)), but the 'adhered' TF could cover (decorate) the worn surface of the steel counterpart. The high WR value of the composite was accompanied by the formation of a deep wear track, which had a 'classic' shape of a semicircular valley, to a certain extent repeating that of the steel counterpart (Fig. 7, (h)). Most likely, the sharp CoF rising at the sliding distance of 1.0–1.5 km, shown in Fig. 2, was caused by the TF adhering on the counterpart surface, which made it difficult to slide even under the water lubrication conditions.

In order to generalize the obtained results, the WR versus CoF dependences were plotted for both dry friction and water lubrication conditions (Figs. 8 and 9). In the dry friction case (Fig. 8), the best tribological properties were achieved by loading PEEK with PTFE (shown by the red arrow), namely for the PEEK/PTFE, PEEK/PTFE/CF, and PEEK/PTFE/MoS₂ composites (with the MoS₂ content within 0.5 wt.%). Its rising up to 5 and 10% was accompanied by enhancing the CoF level.

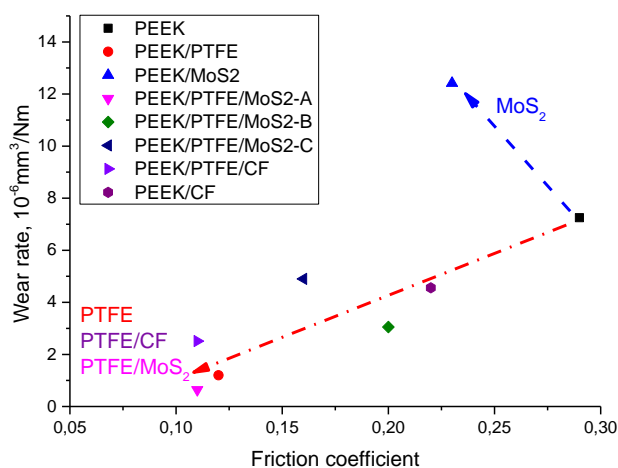


Fig. 8. The WR versus CoF dependence for the dry friction conditions.

The PEEK/10MoS₂ composite was characterized by the worst tribological properties (indicated by the blue arrow). This meant that, as a monofiller, it did not play the role of a solid lubricant (CFs could not be considered a solid lubricant filler as well). This result could not be considered as unexpected data, since the efficiency of loading PEEK with PTFE as an antifriction component was reported by many authors, and the TF formation was called the mechanism for improving the tribological properties [22–25].

The effect of the fillers changed markedly under the water lubrication conditions (Fig. 9). In particular, loading with 10% MoS₂ was not accompanied by a decrease in the WR values compared to neat PEEK, but the CoF level noticeably increased (the blue arrow). On the other hand, loading with CFs, both as a monofiller and in combination with 10 wt.% PTFE (the black arrow) resulted in the multiple increase in the WR values (at the CoF levels commensurate with that for neat PEEK).

In contrast to the dry friction case, the greatest tribological properties were shown by two and three-component PEEK/PTFE composites, in which the MoS₂ contents differed up to 20 times (shown by the red arrow in Fig. 9) under the water lubrication conditions. The TF formation mechanism was suppressed by the presence of the aqueous medium. For this reason, in terms of the combination of the WR/CoF properties (compared to the dry friction conditions), neat PEEK did not differ so much even from the composites loaded with PTFE and PTFE/MoS₂.

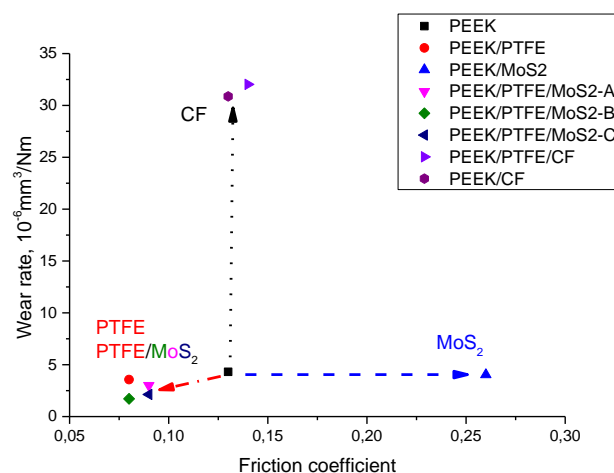


Fig. 9. The WR versus CoF dependence for the water lubrication conditions.

Fig. 10 (a) shows an optical images of the counterpart surface after friction against the PEEK/10MoS₂ composite. Unlike the PTFE-containing ones (PEEK/PTFE, PEEK/PTFE/MoS₂-A and PEEK/PTFE/MoS₂-B presented in Fig. 6 (a, b and c), respectively, the counterpart surface was not smooth, but covered with a TF damaged by longitudinal microgrooves. It was they, which prevented the facilitated sliding of the tribological contact parts (CoF>0.25). However, the process was not accompanied by a noticeable increase in the WR values (<4.5×10⁻⁶ mm³/N×m) due to the presence of distilled water, although longitudinal grooves were observed on the wear track surfaces of these composites (Fig. 10 (c and e)).

Thereby, the TF formation and adherence on the counterpart surface affected the CoF levels without worsening wear resistance under the water lubrication conditions. Nevertheless, in the cases of friction against the CF-containing PEEK-based composites, the presence of reinforcing fibers prevented the ‘protective’ TFs from being

adhered on the steel counterpart surface (CFs protruding above the wear track surfaces of the composites, in fact, continuously scraped it). Additionally, water lubrication prevented the TF from adhering to the wear track surface of the PEEK/PTFE/CF composite. Due to the presence of water lubrication, the CoF level was identical for neat PEEK, as well as both PEEK/CF and PEEK/PTFE/CF composites. However, CFs gradually abraded the steel counterpart (Fig. 10 (b)), which in turn began to act identically on the composite surface (Fig. 10 (d)). In this case, the WR value increased numerously (Fig. 3), and debris could be fixed on the counterpart surface as a TF (Fig. 6 (d)). Thus, the presence of the aqueous medium mainly decreased the CoF level. On the other hand, it prevented the 'protective' TF from adhering to the steel counterpart.

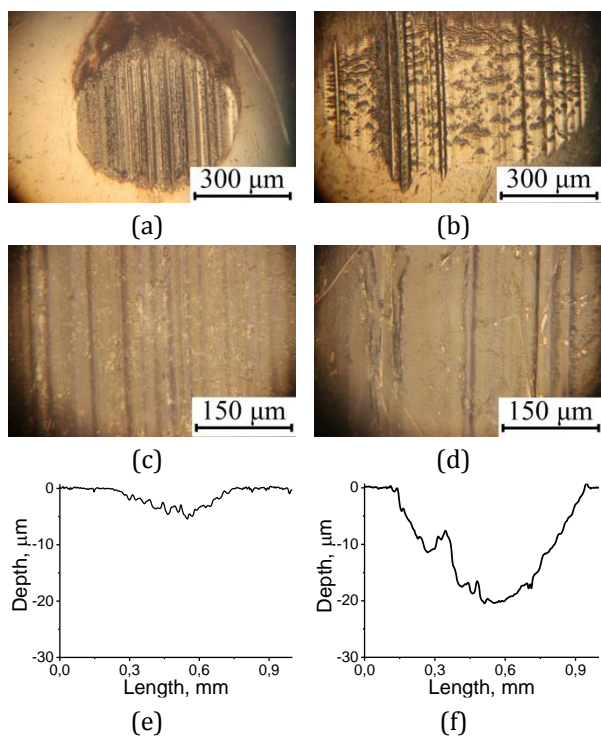


Fig. 10. The optical images of the wear track surfaces (a, b) and their profiles on the steel counterpart after the tribological tests against the PEEK/MoS₂ (a, c, e) and PEEK/CF (b, d, f) composites; water lubrication.

Previously, however, the opposite effect was shown after loading PEEK with CFs, which enhanced WR values under aqueous lubrication conditions. For example, Z. Guan et al. [17] reported an extremely low WR value of $1.66 \times 10^{-8} \text{ mm}^3/\text{N}\cdot\text{m}$ for the highly-loaded PEEK/30%CF composite when sliding against stainless steel. According to the authors of [17], such a result was determined by the

test scheme, which was referred to a planar tribological contact. In this case, debris, due to their retention in the contact zone, as well as a lower specific pressure on the friction surfaces, could act as a third body, protecting the polymer composite from wear. In addition, the formation mechanism for a CF-reinforced TL could serve as an effective way to improve the tribological properties [20,21].

It should be noted finally that, in addition to PEEK, PEI is a promising material for some tribological applications. Based on the results obtained in this research, the combination of the fillers was used that provided the best wear resistance for both applied conditions of the tribological tests: 10 wt.% PTFE and 0.5 wt.% MoS₂ (the PEI/PTFE/MoS₂-A composite). Considering these data, the WR values are presented in Fig. 11.

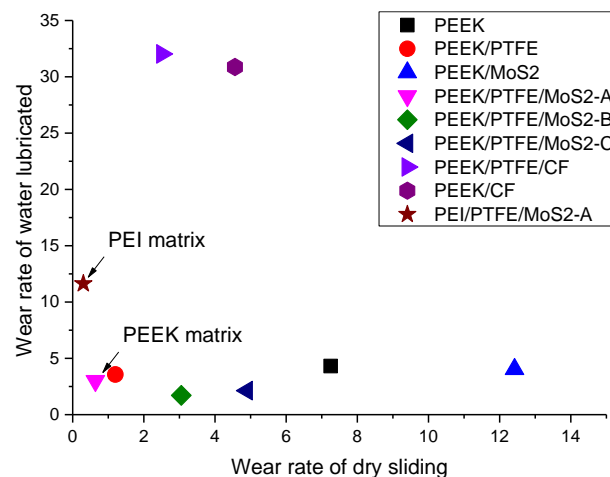


Fig. 11. Generalization of the obtained results taking into account the PEI/PTFE/MoS₂ composite.

The PEEK/PTFE/MoS₂-A and PEI/PTFE/MoS₂-A composites showed comparable wear resistance under the dry friction case. However, the PEI/PTFE/MoS₂-A specimen possessed the WR value higher by 3.9 times than that of the PEEK/PTFE/MoS₂-A one under the water lubrication conditions. By analogy with the above results, this effect should be interpreted from the standpoint of the TF formation and adherence on the steel counterpart surface. Some patterns in the TF formation on the PI/PTFE/MoS₂ composite can be found in [26] for the dry friction conditions. So, it is recommended to use the PEEK/PTFE/MoS₂-A composite in bearings operated under both dry friction and water lubrication conditions.

4. CONCLUSIONS

As a result of the study, the following conclusions were drawn:

1. The tribological properties of the PEEK-based composites with solid lubricating particles under both dry and water lubrication friction conditions varied due to the change in the pattern of the TF formation and adherence on the steel counterpart surface.
2. Under the water lubrication conditions, the WR multiple increase for the PEEK/PTFE/CF composite was due to the suppression of the TF adherence on the steel counterpart by protruding CFs above the sliding surface, followed by abrasive damage to both bodies. Therefore, they are not recommended for use in water lubrication bearings.
3. The PEEK/PTFE/MoS₂ is suggested for using in such bearings according to the 'ball-on-disk' scheme under mixed dry friction/water lubrication conditions.

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