Development and optimization of high-performance PEEK/CF/ Nanosilica hybrid composites

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In the present study, tribological properties of PEEK/CF/nanosilica composites with distinct amounts of silica nanoparticles against steel were studied by using a block-on-ring tribometer followed by the characterizations of associated transfer films and polymer worn surfaces. The results demonstrate that the content of silica nanoparticles exerts an obvious influence on the friction and wear properties of PEEK/CF/nanosilica composites. Under low-load conditions, the friction coefficient and specific wear rate exhibit opposite dependence on the nanosilica content. The friction coefficient decreases with increasing nanofiller content, while the specific wear rate increases with enhancing nanosilica loading. When the load conditions were changed toward high values, the divergence of the tribological properties becomes insignificant, which show less dependence on the nanosilica loading. Taking into account the practical applications of such composites, the composite containing 2 wt.% silica nanoparticles can serve as an excellent candidate for manufacturing tribological components in the practical applications.

KEYWORDS
friction and wear performance, nanosilica content, PEEK/CF/nanosilica composites

1 | INTRODUCTION

Polyetheretherketone (PEEK) is one of the mostly used high-performance polymers for industrial applications, which exhibits excellent mechanical properties and thermal resistance.1-3 Its glass transition temperature and melting temperature are 150°C and 343°C, respectively.4 As a representative of high-performance polymers used as tribological materials for producing different components in mechanical and automotive engineering, for example, sliding bearing bushings, cages of high-precision ball bearings, polymer gears, etc., PEEK has been paid much attentions.5-8 It is generally accepted that a high-performance polymer-based tribomaterial consists of a high-performance polymer, such as PEEK, polyimide,9,10 polyphenylene sulfide,11 internal solid lubricants (polytetrafluoroethylene, graphite, molybdenum disulfide), and reinforcing fibers, for example, carbon fibers, glass fibers, and aramid fibers. The solid lubricants reduce the friction, while the reinforcing fibers enhance the wear resistance of the polymer composite.

Recently, the friction and wear properties of such tribocomposites have been successfully controlled by adding functional fillers at different length scales for further increase of their tribological properties, for example, submicro- and nano-sized particles.12-17 In the study of Zhang et al.,18 they studied the roles of low-loading nano-sized silica particles (1 vol.%) on the friction and wear behavior of short carbon fiber (SCF)/PTFE/graphite (micro-sized)-filled PEEK. It was found that the nanoparticles remarkably reduced the friction coefficient in the studied range of pressure (p) and velocity (v) conditions up to 7 MPa and 2 m/s. With respect to the wear performance, the wear resistance of the PEEK composite was greatly improved under high pv-conditions by addition of nanosilica. In a more recent study,19 Chang and co-workers...
investigated the impact of nanofiller content on the tribological properties of PEEK. It was reported that there was an optimum filler loading for the specific wear rate. More importantly, like other properties of polymer nanocomposites, homogeneous distribution of nanoparticles within the PEEK matrix and strong filler/matrix interphase is responsible for the improvement of the tribological performance. In summary, it can be concluded that addition of nano-sized inorganic particles can reduce the friction and wear of the polymer materials.

To guide the designing of PEEK components with high reliability, tailored friction, and wear performance for tribological applications, studies on the friction and wear mechanisms of PEEK composites have been the pursuit of researchers. Transfer films have been found to be a crucial factor in governing the tribological functionality of PEEK composites. As for the role of rigid particle fillers, it was revealed that the particles may slide or roll between the fiber and counterpart interface depending on the loading parameters. The movement pattern of the rigid particles may be closely related to the near surface properties of the mating polymer materials, which also showed great contribution to the tribological characteristics of polymer materials. In our recent study, it was revealed that the internal solid lubricants were no more than the indispensable component for ensuring superior tribological performance of polymer composites owing to the synergistic effect between the carbon fibers and the rigid particles. Meanwhile, such tribocomposites with high-carbon-fiber content exhibited excellent mechanical properties, which can broaden their applications. In this work, tribological properties of PEEK/CF/nanosilica composites with different nanosilica contents were investigated in a wide range of pressure and sliding velocity conditions and compared with those of a conventional PEEK-based tribocomposite, in which only carbon fibers and graphite particles were incorporated. In addition, the friction and wear mechanisms were characterized based on the comprehensive analysis of the polymer worn surfaces and transfer films formed on the steel counterpart. The aims of the present study are, on the one hand, to explore the optimum nanofiller loadings for the tribological performance of PEEK composites and, on the other hand, to reveal the dominant mechanisms in governing the tribological performance of the composites studied.

2 | MATERIALS AND METHODOLOGY

2.1 | Materials and samples preparation

VESTAKEEP 2000G from Evonik Industries, Germany, was chosen as the polymer matrix due to its excellent mechanical properties and thermal resistance. Chopped carbon fibers (Sigrafil C30) provided by SGL Group, Germany, graphite powder (RGC 39A) from Superior Graphite, Sweden, and nano-sized silica (Aerosil R9200) from Evonik Industries, Germany, were utilized as reinforcing fillers. Their specifications according to the manufacturers are given in Table 1. In addition, morphology images of the as-received fillers are shown in Figure 1.

The nanosilica content in the composites was varied from 1 to 10 wt.%. The designations and detailed formulations of the composites are introduced in Table 2. Traditional PEEK tribocomposite (PEEK-Tr.) without nanosilica was used as the reference material. The combinations of PEEK and different kinds of fillers were chosen based on the experimental results of our recent study, which studied the influence of inorganic particles on the tribological performance of polybutylene terephthalate (PBT) composites. The composites were prepared on a co-rotated twin-screw extruder (ZSE 18 MAXX, Leistritz Extrusionstechnik GmbH, Germany) by using a multistep compounding approach. In a first step, a PEEK masterbatch filled with 20 wt-% silica nanoparticles was prepared. Afterward, it was diluted to the target concentrations by mixing with pure PEEK. The final compounding of the composites was succeeded by feeding this pure PEEK/PEEK masterbatch mixture and chopped carbon fibers in the main feeder of the twin-screw extruder. The temperature of the heating zones was set to 120°C, 370°C, 395°C, 395°C, 395°C, 395°C, 395°C, 395°C, and 395°C from the hopper to the nozzle. The screw speed and throughout were chosen as 100 rpm and 2.5 kg/h, respectively. In comparison, the traditional PEEK composite filled with chopped carbon fibers and graphite particles was prepared by feeding pure PEEK and chopped carbon fibers in the main feeder followed by feeding the graphite particles through a side feeder under same processing conditions. This multistep compounding process can lead to a high dispersion and distribution quality of the particulate fillers according to early studies.

After compounding of the designed composites, they were injection-molded to sheets with a dimension of 50 mm × 50 mm × 4 mm by using an injection molding machine (Engel victory 200/80 spex, ENGEL Austria GmbH, Austria), from which the mechanical and tribological specimens were milled. During injection molding, the cylinder temperature was kept at 385°C, 395°C, 395°C, 395°C, and 395°C in different zones. The mold temperature was chosen as 195°C. The processing chain from the raw materials to the testing specimens is shown in Figure 2. After injection molding of all the composites, the density of each composite was determined according to Archimedes’ principle by using a precision balance (Kern ABT 220-50M, Germany). The results are shown in Table 3. The density of the composites was used to calculate the specific wear rate of the studied composites.

| Filler designation | Trade name | Particle size | Fiber diameter | Fiber Length | Density |
|--------------------|------------|---------------|----------------|--------------|---------|
| CF                 | Sigrafil C30 | —             | 7 μm          | 6 mm         | 1.8 g/cm³ |
| Graphite           | RGC 39A    | 18-22 μm (d90)| —             | —            | 2.25 g/cm³ |
| Nanosilica         | Aerosil R9200 | 12 nm         | —             | —            | 2 g/cm³  |
2.2 | Mechanical tests

In order to reveal the mechanical properties of the composites, tensile tests were performed on a Zwick universal testing machine (RetroLine, Zwick GmbH & Co. KG, Germany) at room temperature following the international standard DIN EN ISO 527. The crosshead speed for determining the Young's modulus was chosen as 1 mm/min, while it was kept at 50 mm/min for calculating the tensile strength. Five samples were examined for each PEEK composite, in order to calculate the mean values.

2.3 | Tribological tests

Tribological investigations were carried out on a block-on-ring (BoR) testing device at room temperature under dry sliding conditions, as it is described in an early study. This test configuration is a commonly used approach for characterizing tribological behavior of polymer/steel tribopair. The dimension of the specimen is 4 mm × 4 mm × 10 mm, which was prepared from the injection-molded sheet. The testing area was the area of 4 × 4 mm². Steel ring made of 100Cr6 (INA IR60X50X25, Schaeffler Technologies AG & Co. KG, Germany) was...
used as counterpart. Its surface roughness (Ra-value) is approximately 0.1 to 0.2 μm. After tribological characterizations, the steady-state friction coefficient and specific wear rate were evaluated. The specific wear rate (ws) was determined according to formula (1): \[ ws = \Delta m / (\rho \cdot F_N \cdot v \cdot t) \text{ [mm}^3/\text{Nm]} \] (1)
in which, \( \Delta m \) is the mass loss of the composite material, \( \rho \) presents its density, and \( F_N \) denotes the normal load. \( v \) and \( t \) are the sliding speed and time, respectively. For each pv-combination, at least three tests were conducted for determining the average friction coefficient and specific wear rate.

2.4 Analysis of the worn surfaces transfer films

Worn surfaces of polymer samples and transfer films formed on the steel ring were characterized by using a Keyence laser scanning microscope VK-X1050, Japan, and a JEOL scanning electron microscope (SEM, JSM-6460 LV SEM, Japan) with energy-dispersive X-ray (EDX) spectroscopy. In addition, the thickness of the transfer films was determined by applying a focused ion beam (FIB) system (FEI Altura 875 Dualbeam, USA). In order to avoid the damage of the transfer films during the FIB cutting process, the counterbody surface was sputtered with a thin layer of platinum prior to the cutting process.

3 RESULTS AND DISCUSSION

3.1 Mechanical properties

Mechanical performance of polymer materials is of vital importance for their applications, which is also very important for tribological applications of such materials. Generally, high mechanical properties can significantly enhance their load-bearing capacity and broaden the pv-limit of polymer-based tribomaterials. Figure 1 shows the tensile properties of PEEK composites. As is seen in Figure 3A,B, addition of high amounts of carbon fibers and nanosilica into PEEK matrix leads to enhanced stiffness and strength compared to that of PEEK-Tr. However, high filler loading impairs the ductility of the PEEK/CF/nanosilica composites (Figure 3C). The elongation at break of PEEK/CF/nanosilica composite with 10 wt.% nanosilica content is about 3%.

3.2 Friction and wear properties

The steady-state friction coefficient and specific wear rate of PEEK-Tr are shown in Figure 4. As can be seen, the friction coefficient exhibits less dependence on the pv-conditions except at 8 MPa and 4 m/s, which is between 0.3 and 0.45 in the studied range of pv-conditions (Figure 4A). More surprisingly, PEEK-Tr presents the lowest friction coefficient of 0.26, once the pv-product was raised to 32 MPa m/s. Considering the specific wear rate of PEEK-Tr, it is observed that, unlike the friction coefficient, the wear resistance of PEEK-Tr is much susceptible to the pv-level. Under pv-conditions higher than 6 MPa m/s, an increase in the pressure at same velocity leads to an decrease of the specific wear rate up to 51%, as is shown in Figure 2B.

With respect to the friction performance of PEEK/CF/nanosilica composites, it can be clearly seen from Figure 5A that the friction coefficient of the composites decreases with increasing pv-product, independent on the nanosilica content. The lowest friction coefficient can be found under the highest load condition, that is, 8 MPa and 4 m/s, which is less than 0.1. More importantly, the nanoparticle content exerts pronounced effect on the friction property under low- and moderate-load conditions up to 6 MPa-m/s (Figure 5A). At 1 and 2 MPa-m/s, high amount of nanosilica leads to much better friction performance. However, moderate amount of nanofillers results in low friction coefficient when the pv-condition is raised to 1:4 and 3:2 MPa:m/s. Under high-load conditions, the nanofiller content has almost no impact on the friction behavior of the PEEK/CF/nanosilica composites. All the composites present similar friction coefficient. Figure 5B shows the dependence of specific wear rate on the pv-conditions and nanosilica concentrations. Unlike the dependence of the friction coefficient on the load conditions and nanofiller contents,
the specific wear rate exhibits distinct trend. In the studied pv-range, PEEK/CF-N10 shows generally the highest specific wear rate (Figure 5B), especially at 1 MPa and 1 m/s. Its specific wear rate is twofold higher than that of other PEEK/CF/nanosilica composites at 1 MPa m/s. In contrast, the PEEK/CF/nanosilica composites filled with low loadings of nanosilica, that is, 1 and 2 wt.%, present much better wear performance in the studied range of pv-conditions. Overall, it can be concluded from Figure 5 that PEEK/CF-N2 displays simultaneously lowest friction coefficient and high wear resistance in a wide range of load conditions among the PEEK/CF/nanosilica composites studied.

Compared the tribological properties of PEEK/CF/nanosilica composites with those of the traditional PEEK composite (PEEK-Tr), it can be observed that addition of nanoparticle into the PEEK/CF composite significantly reduces the friction coefficient and enhances the wear resistance, especially under moderate- and high-load conditions (Figures 4 and 5). For instance, the friction coefficient of PEEK/CF/nanosilica composites at 8 MPa and 4 m/s shows a reduction of more than 100% compared to that of PEEK-Tr. Similar phenomenon can be also observed for the wear resistance of the materials. The specific wear rate of the PEEK/CF/nanosilica composites at 4 MPa and 4 m/s is around $8.8 \times 10^{-6}$ mm$^3/(Nm)$, which is $1.5 \times 10^{-6}$ mm$^3/(Nm)$ for PEEK-Tr (Figure 4). As mentioned above, PEEK/CF-N2 exhibits the best tribological performance in the studied range. Its friction coefficient and specific wear rate are similar to those of a high-performance PEEK-tribocomposite under extreme pv-conditions reported in the literature,\textsuperscript{33} which is filled with 10 vol.% carbon fiber, 8 vol.% solid lubricant, and 2 vol.% nanosilica. However, this tribocomposite presents much higher mechanical properties due to the high amount of carbon fibers (Figure 3A). In order to evaluate the potential for the practical applications of PEEK/CF-N2 composite, we compare its friction and wear performance with that of a commercially available high-performance PEEK-based tribocomposite (Victrex WG101, Victrex plc, UK). The results demonstrate that both the friction coefficient and specific wear rate of PEEK/CF-N2 are lower than those of WG101 under similar pv-conditions measured from a BoR tribometer which were reported in Reference 34, which indicates that PEEK/CF-N2 can be an excellent candidate for producing polymer-based tribological components in service.

### 3.3 Tribological mechanisms

In order to elucidate the friction and wear mechanisms of the studied PEEK composites, worn surfaces of the polymer specimens after the sliding wear tests were inspected by using a laser scanning microscope. The micrographs of the worn surface of PEEK/CF/nanosilica composites at 1 MPa and 1 m/s are shown in Figure 6. The composition of the materials exerts obvious influences on the surface morphologies of the polymer sample. In comparison to the relatively...
rough worn surface of PEEK/CF-N10 with deep plowing furrows in some contact regions (Figure 6D), PEEK/CF-N1 exhibits smoother worn surface, as is shown in Figure 6B. As considering the worn surfaces of PEEK-Tr and PEEK/CF-2, both composites present similar micrograph of the worn surface, whereby shallow plowing furrows on the contact region can be clearly observed (Figure 6A,C). It is commonly accepted that the adhesion component dominates the friction performance under lower load conditions within polymer/steel tribosystems.31,35 This smooth worn surface of PEEK/CN-N1 enlarges the real contact area between the steel and polymer sample. As a result, friction coefficient of PEEK/CF-N1 shows the highest value among all the materials studied due to enhancing adhesion between the sliding pair (Figure 5A). On the contrary, deep plowing furrows induced by fractured carbon fibers or some nanosilica agglomerates at high filler content lead to high material loss of the sample during the sliding wear test, which significantly impairs the wear resistance of PEEK/CF-N10 at 1 MPa and 1 m/s, as is shown in Figure 5B.

When the pv-combination was increased to 8 MPa and 4 m/s, the difference of the worn surface morphology between different PEEK composites becomes insignificant (Figure 7), which is associated with the cross-talk contribution of the frictional deformation component. Under extreme load conditions, the carbon fibers were forced to bear the high pressure applied on the sample due to its high stiffness compared to that of PEEK matrix. As a result, the adhesion component of friction is reduced. In this case, the friction and wear properties of PEEK/CF/nanosilica composites with distinct compositions do not differ from each other owing to the same carbon fiber concentration of these nanocomposites (Figure 5, pv-conditions above 16 MPa m/s). More importantly, addition of nano-sized ceramic particles clearly changes the tribological mechanisms of the PEEK composites (Figure 7B-D). The worn surface of PEEK/CF/nanosilica composites is much rougher than that of PEEK-Tr (Figure 7A), which indicates that the real contact between the sliding pair is diminished compared to that at low pressure. This decreasing real contact area leads to less adhesion between the polymer sample and steel ring interface. Consequently, PEEK/CF/nanosilica composites exhibit lower friction coefficient (Figures 4 and 5).

Analysis of the worn surface of PEEK/CF-N2 by means of FIB under high resolutions provides additional information on the tribological mechanisms of PEEK/CF/nanosilica composites. As is seen from Figure 8A, PEEK/CF-N2 exhibits quite rough surface with some fractured carbon fibers as indicated by the black arrows. Close
inspection of the worn surface demonstrates that PEEK/CF-N2 presents quite poor carbon fiber/matrix-interphase (Figure 8B). More interestingly, carbon fibers protrude out of the area of polymer material, which means that they bear the load applied on the composite under high-load conditions during the sliding process. In addition, particulate wear debris accumulates adjacent to the carbon fibers, as can be clearly seen in Figure 8B. Similar morphologies can be also observed on the worn surfaces of other PEEK/CF/nanosilica composites studied in this work (Figures S1 and S2). These phenomena lead to diminishing real contact areas during the sliding wear test. As a result, the adhesion between the sliding pair is also reduced. Thus, the PEEK/CF/nanosilica composites exhibit much lower friction coefficient than that of PEEK-Tr under the same pv-condition, which shows smoother worn surface (Figure 9). Moreover, the rolling of tiny rigid
nanoparticles between the sliding pair under extreme load conditions can also contribute to the friction reduction, as reported in our early studies. The accumulation of the wear debris and inorganic particles adjacent to the carbon fibers serves as third bodies between the sliding pair, which might prevent the severe wear of the polymer matrix.

It has been generally accepted that the formation of transfer films on the steel counterpart, that is, structure and properties, plays a vital role on determining the tribological performance of polymer/steel tribosystems. Analysis of the transfer films formed on the steel counterface reveals that the wear particles of PEEK/CF/nanosilica composites are entrapped into the deep grooves (dark areas in SEM images in Figure 10) on the steel surface in the earlier sliding process that reduces the surface roughness and thus decreases the abrasion of the counterpart asperities. More interestingly, it is also manifested that PEEK/CF/nanosilica composites present similar morphology of transfer films independent on the load conditions and nanofiller content (Figure 10).

Inspection of the transfer film structure in its thickness direction by using FIB cut of the steel counterpart clearly reveals that the transferred materials are entrapped into the deep grooves of the steel counterbody (Figure 11) and smooth the counterface. In addition, transfer films can be also found on the smooth areas of the counterbody surface, as is shown in Figure 11. The thickness of the transfer films can attain hundreds of nanometers. As is seen, there is almost no difference of the transfer films between PEEK/CF/nanosilica composites with different particle contents under the same pv-condition. It can be therefore concluded that the distinction of the friction and wear properties of the PEEK/CF/nanosilica composites...
can be mainly contributed to the adhesion and abrasion nature between the sliding pair. Quantitative SEM/EDX-analysis of the transfer films formed on the steel ring surface in a broad area (Figures S3 and S4) reveals that, independent on the load conditions and compositions of the tribocomposites, the transfer film presents similar elemental compositions, for example, C-, O-, Si-, Cr-, and Fe-element, as is shown in Figure 12. Considering the atom content of the transfer films, as-received ring without transfer film exhibits the highest content of Fe-element. When transfer film was generated, the content of Fe-element drops due to the partially covering of the composite on the steel surface (Figure 10). In addition, the tribofilm of PEEK/CF-N10 generated under a load condition of 8 MPa and 4 m/s shows the lowest Fe-element content and highest content of Si- and O-element, which means that more transfer materials are presented in the inspected areas. Nevertheless, based on the micrographs and EDX-analysis, it can be concluded that there is no significant distinction of the transfer film generation between different load conditions and material compositions. In this study, different friction and wear properties are mainly caused by the distinct patterns of the polymer worn surfaces, as mentioned above.

4 | CONCLUSIONS

In the present study, tribological properties of PEEK/CF/nanosilica composites with distinct nanosilica loadings were systematically investigated under dry sliding conditions on a BoR apparatus. The following conclusions can be drawn:

1. It is revealed that PEEK composites filled with carbon fibers and silica nanoparticles exhibit much better friction and wear performance compared to those of PEEK-Tr. More importantly, their tribological performance is strongly dependent on the nanofiller content.
2. PEEK/CF/nanosilica composite filled with 2 wt.% nanosilica exhibits excellent tribological properties in the whole pv-range studied, which can meet the requirements of the industrial applications.
3. It is of great interest to reveal that the transfer film shows similar morphology and composition independent on the nanosilica contents and pv-conditions. The dominant tribological mechanisms, which determine the friction and wear behavior of the PEEK/CF/nanosilica composites, are the abrasive or adhesive wear.

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CONFLICT OF INTEREST
The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT
The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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