

INVESTIGATIONS ON THE CREATION OF PROMISING ANTIFRICTION POLYMERIC MATERIALS BASED ON THERMALLY RESISTANT THERMOPLASTICS

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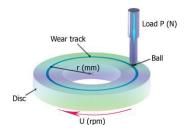
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Abstract

The present review highlights the results of tribological studies on highly thermally resistant partially crystalline (Victrex poly(ether ether ketone) and poly(phenylene sulfide)) and amorphous (poly(arylene ether ketone)s) thermoplastics, which are promising for the creation of new antifriction materials that would be able to operate reliably under extreme conditions.



Key words: highly thermally resistant thermoplastics, partially crystalline poly(ether ether ketone) and poly(phenylene sulfide), amorphous poly(arylene ether ketone)s, tribological studies, antifriction materials.

Dedicated to the memory of our teacher and mentor Prof. A. P. Krasnov

1. Introduction

The rapid development of industry warrants the extensive application of antifriction polymeric materials based on highly thermally resistant thermoplastic polymers in friction joints [1–6], which gradually displace the metallic materials [7–9]. Thermoplastics can be used in friction joints both as the components of composite materials and in the unfilled form [10].

The antifriction joints are made of tribochemically stable polymers that display stable friction coefficients and optimal wear during the long-term operation [11]. They include mainly the polymeric antifriction materials based on highly thermally resistant partially crystalline thermoplastics such as poly(ether ether ketone) (PEEK) [12, 13] and poly(phenylene sulfide) (PPS), which are produced abroad on an industrial scale. Among amorphous highly thermally resistant thermoplastics, of particular note for the creation of new antifriction materials are poly(arylene ether ketone)s (PAEKs).

The following sections will be devoted to the tribological characteristics of highly thermally resistant partially crystalline and amorphous thermoplastics that were produced for the creation of new antifriction materials for different applications, including the use under extreme conditions.

2. Tribological investigations on the materials obtained from partially crystalline thermally resistant thermoplastics

2.1. Poly(ether ether ketone)

The industrial production of partially crystalline PEEK was launched by ICI (UK) under the Victrex PEEK trademark. The 1980s were marked by the commercialization of this type of

polymers, including their composites with glass and carbon fibers

Nowadays, the most popular poly(ether ketone)s are those of Victrex production (with the crystallinity degree ranging from 35 to 48%). PEEKs are produced industrially as powders, granules, films, or fibers. Along with the high physicomechanical characteristics, this polymer features unusually high radiation resistance and extremely high stability to the action of hot water and vapor [14]. In these characteristics, PEEK surpasses all the known thermoplastics. PEEK does not undergo hydrolysis even after 800 h of exposure to hot water at 80 °C. The articles made of PEEK can withstand a short-term exposure to vapor at 300 °C. PEEK is also highly stable to different solvents (and dissolves only in concentrated sulfuric acid).

The main advantage of PEEK over other antifriction thermoplastics is a combination of the high physicomechanical properties with unique chemical resistance [15–18]. The processing temperature of this polymer composes 360–400 °C, which stems from its high melting point ($T_{\rm m}=343$ °C) [19]. The main drawback of PEEK is its relatively low glass-transition point ($T_{\rm g}=143$ °C) [20] and, consequently, relatively low deflection temperature under heavy loads (150–160 °C) [14, 21, 22].

There are many reports that describe the tribological behavior of PEEK composites filled with solid lubricating agents, short carbon fibers, and nanofillers of various chemical structures [23, 24]. However, the modern literature lacks the detailed systematic studies on the tribochemical processes that take place during PEEK friction. This is likely to be caused by the insolubility and high chemical stability of PEEK [14]. While exploring wear debris, many researchers deal with the data on melt viscosity or flow index of a polymer melt. The investigations on the effect of a molecular mass on the tribological and mechanical properties of PEEK [25] showed that an increase in the polymer molecular mass leads to a reduction in the tensile modulus and microhardness and an increase in the friction coefficient and wear resistance. Based on the micrographs of wear debris, Zhang and Schlarb [26] assumed that the sample derived from the high-molecular PEEK featuring the lower crystallinity degree [27] undergoes greater plastic strain than the samples obtained from the lowmolecular PEEK. This allows one to increase the wear resistance of the high-molecular PEEK at the low contact pressures (1 MPa, 1 m/s) since, in this case, the main wear mechanism appears to be microcutting. As the contact pressure grows (4 MPa, 1 m/s), the occurring plastic deformations exert greater impact on the formation of transferred films and, as a consequence, lead to the increased wear. The authors supposed that the high material rigidity determines the wear resistance of the polymeric material. Zhang et al. [28, 29] demonstrated that the increased rigidity of coatings based on PEEKs featuring various crystallinity degrees leads to improved tribological parameters. As the PEEK molecular mass increases, the shear modulus, impact strength, and tensile strength grow, whereas the Young modulus reduces [27, 30].

The investigation of friction processes for the PEEK samples featuring different molecular masses ($M_w = 14-56$ kDa) and those subjected to annealing at 265-290 °C allowed for establishing that the wear resistance of the samples increases with the growth of the molecular mass but in a certain range of the pv-factor [31]. Lu and Friedrich [31] presented the data on the friction of PEEK at the high sliding rate (3 m/s, 1MPa) and showed that the highest friction coefficient is observed for the most high-molecular PEEK. An increase in the load negatively affects the friction coefficient of the lowmolecular polymer due to the presence of less strong interspherulitic amorphous bonds. The authors assumed that, during friction at the elevated pressure, the larger amount of energy is absorbed by the amorphous part of PEEK which has a more developed fluctuation network in the case of the highmolecular samples, and noted that the high value of intermolecular interaction allows one to improve the wear resistance. They also concluded about the insignificant dependence of the surface energy on the molecular mass, which explains small changes in the PEEK friction coefficient with the growth of the molecular mass [31].

The main wear type of unfilled PEEK is the polymer crumbling during friction, while that for the composites on its base is the segregation of glass and carbon fibers from the polymer matrix to the surface [32, 33]. The improved wear resistance of the composites compared to that of unfilled PEEK is connected with weakening of adhesion interaction during friction [34]. The fatigue wear during rolling friction with a steel counterbody occurs due to microslippage and adhesion processes, which is characterized by a relatively high friction coefficient equal to ~0.4 [31]. The mechanism of abrasive wear is observed also in the case of PEEK filled with

polytetrafluoroethylene and potassium titanate particles that interact with the polymer surface during friction as an abrasive third-body [35, 36].

2.2. Poly(phenylene sulfide)

Partially crystalline PPS, as well as PEEK, is a prominent representative of thermally resistant construction thermoplastics. The industrial production of PPS with the following formula was realized for the first time by Phillips Petroleum [14]:

$$+$$

The main characteristics of PPS are as follows: crystallinity degree 60–65%, molecular mass 18 kDa, melt viscosity 30–50 P, $T_{\rm g}=85$ °C, and $T_{\rm m}=288$ °C. During prolonged heating (at 230–280 °C in air for 5 h), it manifests the properties of a thermoset plastic. Unfilled PPS has relatively low stress–strain characteristics and heat stability. However, a whole set of other valuable operational characteristics (high thermal, fire, chemical, and radiation resistance, low moisture absorption, and good dielectric properties) and low cost promoted the development of plastics based on PPS. The flow index of its melt strongly depends on the heating temperature and duration [14].

As well as PEEK, PPS finds extensive use in the creation of materials that are used in modern friction joints [14].

There are only limited data on the effect of PPS crystallinity on its physicomechanical and tribological properties. Ma *et al.* [37] controlled the PPS crystallinity by introducing the fibers of the same polymer into the polymer matrix. At the PPS fiber content above 25 wt %, a two-fold reduction was observed in the friction coefficient (up to the value of 0.4) relative to that of unfilled PPS in the case of a block-on-ring scheme (v = 0.42 m/s, P = 200 N). An increase in the fiber content to 75 wt % facilitated the growth of the PPS crystallinity from 26.8% to 33.4% and an increase in the breaking stress in 2.5 and 4.5 times compared to the PPS fibers and binding agent, respectively.

A distinctive feature of PPS from other thermally resistant polymers that are used in friction joints is its self-lubricating ability [38-42]. Sukumaran et al. [43] showed that during friction the PPS surface undergoes abrasive and adhesive wear, which facilitates the formation of a transferred film. Lu et al. [44] assumed that transferred PPS remains in cavities that comprise the counterbody roughness, which leads to a decrease in the friction and wear coefficients. The X-ray photoelectron spectroscopic (XPS) studies revealed the presence of iron sulfide and sulfate on the surface of a steel counterbody upon friction with PPS, which resulted from the tribochemical decomposition of both unfilled polymer and the composite materials [45–47]. Presumably, the friction of PPS leads to the decomposition of the macromolecules with the formation of pphenylene moieties [47]. Cho et al. [48] studied the friction of the PPS composites filled with PbTe and PbSe and found that the transferred films do not contain sulfur traces. Unfortunately, the authors did not discuss the reasons for the absence of tribochemical destruction of the PPS binder.

The investigation of a friction process of the modified PPS filled with molybdenum disulfide at high temperatures (100–350 °C) [48, 49] suggested that, in the case of rigid polymer chains (as the content of aromatic rings lacking flexible bridging units in the macromolecule increases), the tribological characteristics of the polymeric self-lubricating materials improve. Unfortunately, the investigations were performed with the polymers with low molecular masses ($M_{\rm w}$ up to 7.1 kDa) and did not take into account the effect of thermal destruction of MoS₂, which proceeds under rather severe conditions of polymer processing (high temperature) and tests.

During processing, PPS undergoes thermooxidative destruction, resulting in the formation of -C=O, -C-O-, -S=O, and >SO₂ groups [50–52]. These bonds can be generated also under the action of shear loads that arise during friction. Luo *et al.* [46] concluded that the thermooxidative destruction leads to intensive interaction of the PPS matrix components, for example, with the fillers, the enhanced adhesion to a steel substrate in the course of coating application and during friction upon formation of a transferred film. The authors [46] assumed that the formation of a tribooxidized transferred film reduces the adhesion strength of this film on a counterbody. However, the formation of a uniform tribooxidized layer can lead to growth of an adhesion component of the friction force and facilitate an increase in the friction coefficient and wear [52].

The self-lubricating effect was also observed upon friction of the materials based on PPS filled with *para*-aramid fibers (5–30%) [41]. The tribochemically active kevlar fibers facilitate the destruction and oxidation of PPS due to significant friction heating that arises in the contact zone. This allows one to reduce wear in 2–9 times relative to unfilled PPS, depending on the filling degree. However, the friction coefficient under conditions of friction along steel for the composites explored was higher.

The comparative studies performed with the PPS nanocomposites filled with CuO and reinforced with polyaramid and carbon fibers showed that better tribological characteristics are manifested by the organoplastics based on PPS [53].

The investigation of various micro- and nanofillers based on PPS binder allowed for defining the effect of a chemical structure of the filler on the composite friction. Thus, the presence of CuO and TiO2 (up to 2 vol %) in the PPS nanocomposites affords stronger transferred films on the surface of a steel counterbody than the unfilled polymer. The introduction of nanoparticles of ZnO and SiC in any content leads to a reduction in the transferred film strength [54]. The authors explain this behavior by the presence of zero-valent copper and titanium atoms, which were detected in the transferred film by the XPS studies. During friction of the PPS + ZnO and PPS + SiC nanocomposites, the particles of reduced metal or other compounds that would facilitate the adhesion to a counterbody were not detected [38]. The same research group studied the effect of filler deformability on the tribological properties of the resulting PPS composites [55]. It was found that the fillers that undergo plastic deformations during friction, for example, silver and copper sulfides, have good adhesion to the polymer matrix and lead to the improved tribological characteristics. Tin sulfide and zinc fluoride that do not deform

under these conditions (v = 1 m/s, P = 0.65 MPa) have poor adhesion to the polymer and impair its tribological characteristics. The wear resistance of the materials based on PPS reduces upon introduction of the fillers such as Al_2O_3 [56], CaF_2 and ZnF_2 [57], as well as PbSe [58].

3. Tribological investigations on the materials based on amorphous thermally resistant thermoplastics—poly(arylene ether ketone)s

The synthesis and properties of amorphous PAEKs of various chemical structures are prolifically described in the modern literature [59–62]. Amorphous PAEKs exhibit high thermal resistance and good mechanical characteristics in combination with unique specific notched impact strength (up to 20–44 kJ/m²) and ability to operate at high loads in the wide temperature range (from cryogenic to 230–250 °C). Amorphous PAEKs surpass in the specific notched impact strength all the known thermally resistant thermoplastics. Along with this, the cardo amorphous PAEKs feature high heat stability (the softening point ranges from 230 to 270 °C) [60–62].

Unfortunately, the investigations on the tribological properties of amorphous PAEKs are very limited. The first studies devoted to dry friction of amorphous PAEKs were performed at INEOS RAS in 1994-1996 [63, 64]. Krasnov et al. [63] explored the friction processes of the amorphous PAEK based 4,4'-difluorobenzophenone and 2,2-bis(4'hydroxyphenyl)propane. During friction at room temperature, the PAEK underwent destruction, which was confirmed by the results of gel permeation chromatographic (GPC) studies. The evaluation of changes in the molecular masses of wear debris showed that friction afforded 40% reduction in the weightaverage molar mass. The wear debris contained the lower amount of a low-molecular fraction than it was observed in the initial polymer. The authors assumed that the presence of the low-molecular fraction, which consists of macrocycles, during friction can lead to the formation of thermally stable transferred films on a coupled surface and manifestation of the selflubricating effect of these polymers.

Upon thermal treatment (T = 350 °C) for 60 min, the PAEK based on 4,4'-difluorobenzophenone and 2,2-bis(4'-hydroxyphenyl)propane undergoes structuring. This effect is manifested in the loss of solubility in organic solvents as well as a reduction in the effective viscosity. The polymer processing at this temperature also facilitates the formation of cross-links, which is evidenced from the M_z/M_w ratio. During friction, the macromolecules with the same molecular masses ($M_w = 20.5-22.7$ kDa) are transferred to wear debris [65]. As the processing temperature increases, the friction coefficient and wear grow, while the polymer ability to form a transferred film reduces. The deterioration of tribological properties of the PAEKs explored was explained by an increase in the polymer branching degree.

The formation of a rigid-chain system owing to the introduction of bulky cardo groups into the macromolecule leads to an increase in the value of $T_{\rm g}$. In the case of PAEKs bearing the cardo groups, the values of $T_{\rm g}$ are higher by 60–100 °C than those for the polymers lacking the cardo functionalities. The cardo PAEKs were obtained for the first time and characterized

by Prof. S. N. Salazkin [66] a long time ago, and the priority of INEOS RAS in this field is maintained to date.

The synthetic approach to PAEKs based on 4,4'dihalobenzophenone and phenolphthalein was patented by the Chinese researchers [67]. At present, the PAEK based on 4,4'difluorobenzophenone and phenolphthalein (PEK-C) is produced in China on an industrial scale [68]. The results of investigations on friction processes for the cardo PAEKs are now available only for one representative of this class of polymers, namely, PEK-C [67, 68]. Tian et al. [69] studied the effect of radiation processing on the PEK-C polymer and its tribological properties. It was found that an increase in the radiation dose facilitates an increase in the polymer wear resistance as well as a reduction in the friction coefficient and size of the wear debris particles. The authors assumed that these changes are connected with the formation of a cross-linked structure under radiation impact and segregation of a lowmolecular fraction of the polymer to the surface, which enables the formation of a transferred film during friction.

The investigations on the effect of temperature on tribological properties of PEK-C showed that, in the temperature range of 20–170 °C, the polymer features the stable friction coefficient equal to ~0.25. Then it drastically grows, and at200 °C reduces to 0.28 [70]. The polymer wear increases linearly in the range of 50–200 °C. At 25–50 °C, the wear degree does not change. The analysis of SEM images testifies that the surface of the PEK-C samples at the friction rate of 0.39 m/s and contact pressure of 39.2 N in a finger-disc scheme did not undergo adhesive wear by the resulting third-body at 25 and 210 °C.

The elucidation of a dependence of the tribological properties of PAEKs on their chemical structures and molar masses is an urgent challenge of materials science. It is successfully solved by the researchers from the scientific school of the full member of the Academy of Sciences of the USSR V. V. Korshak, Prof. I. A. Gribova, and Prof. A. P. Krasnov [62, 71–75]. The results of investigations on the effect of chemical structures of a large variety of amorphous random co-PAEKs on their tribological properties [72] evidence that the best characteristics (low wear at the pressure of 25 MPa) are displayed by the co-PAEKs containing 2-phenyl-3,3-bis(4'hydroxyphenyl)phthalimidine and hydroxyphenyl)propane moieties. A valuable property of all the co-PAEKs explored was their high tribooxidative stability. While studying the effect of the compositions of copolymers obtained by the interaction of 4,4'-difluorobenzophenone with a mixture of bisphenols 2,2-bis(4'-hydroxyphenyl)propane and 2phenyl-3,3-bis(4'-hydroxyphenyl)phthalimidine at different ratios on the tribological properties of PAEKs [73], it was found that the lowest friction coefficient (0.24) and minimum wear (0.2 mg over the testing time) are characteristic of the co-PAEK bearing 10 mol % of the cardo bisphenol moieties relative to the homo-PAEK based on 4,4'-difluorobenzophenone and 2,2bis(4'-hydroxyphenyl)propane (friction coefficient 0.6, wear 1.0 mg over the testing time).

4. Conclusions

The analysis of the extensive data on the tribological properties of highly thermally resistant partially crystalline

thermoplastics (PEEK and PPS) evidence the extremely high demand in the antifriction materials based on these polymers for the production of friction joint details for different mechanisms, including those operating under extreme conditions: under heavy loads and at high sliding rate [24, 45, 47]. The results of investigations on the tribological properties of highly thermally resistant amorphous PAEKs, performed predominantly by the Russian researchers, testify great potential for controlling the properties of antifriction materials on their base owing to the possibility of variation of chemical structures and molecular masses of the initial polymers in a wide range.

The antifriction materials based on thermally resistant thermoplastics (PEEK, PPS, and PAEKs) hold great promise for the application in aerospace, electrotechnical, machine-building, chemical, gas, oil, nuclear, and other fields of industry [14, 76, 77]. This is possible owing to the unique complex of functional properties of these materials (a combination of high heat, thermal, chemical, and radiation resistance with good mechanical and tribological characteristics), which provide an opportunity of their exploitation under extreme conditions (high physicomechanical loads, enhanced humidity, and in chemically aggressive media in a wide temperature range: from cryogenic to over 250 °C).

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