

# Mechanical and tribological behavior of PTFE–polyoxadiazole fiber composites. Effect of filler treatment

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## ABSTRACT

Effect of RF plasma treatment of polyoxadiazole (POD) fibers in the medium of tetrafluoroethylene on structure, mechanical and tribological behavior of PTFE–POD fiber composite is studied. It is found that fiber treatment raises the composite density, heat resistance, mechanical strength and improves its tribological behavior. The tensile strength of the 10%-filled composite increases by more than 20% and elongation at break by ~50% compared to the composite filled with the original fibers. Based on the results of IR-spectroscopy, DMA and SEM it is concluded that filler treatment produces a fluoroorganic coating with good wettability by the matrix polymer which reduces the void content and raises adhesion between the matrix and the fibers. This creates conditions for the stress transfer between the components producing the macroscopic changes in the composite properties.

Changes in tribological behavior include an increase in both the load-bearing capacity and the friction coefficient compared to the original composite. Some improvement in the wear rate is observed at high specific loads.

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## 1. Introduction

Polytetrafluoroethylene (PTFE) and its composites are widely used in industry as antifrictional wear-resistant materials in a wide range of sliding velocities (up to tens of m/s) and service temperatures (from 4 to 540 K), owing to the unique combination of the service properties, in particular self-lubricating capacity, outstanding chemical, UV- and thermooxidation stability [1,2]. High creep and wear rate of the virgin PTFE restrict its use in loaded friction units and induce development of PTFE-based composites with improved dimensional stability under stress and thermal effects, high wear resistance and loading capacity.

Filling boosts the wear resistance of PTFE by 3 orders of magnitude and more, which makes antifrictional composites based on PTFE one of the main groups of materials in the field [2,3]. Experience has shown that almost any thermally stable solid able to withstand the prolonged effect of PTFE processing temperatures (360–400 °C) can be used for filling PTFE, yet the compounders mostly use a short list of substances, such as petroleum coke (carbon), graphite, bronze, molybdenum disulfide, glass and carbon fibers [2]. Relatively high hardness of the fillers with the exception of molybdenum disulfide and some grades of graphite imparts the resulting composites abrasive properties which reduce the life of precision friction units, even those with hardened

steel and chromium-plated counterparts. Abrasiveness of the composites becomes one of the key factors when selecting an antifriction composite for operation against relatively soft metals. This induced development of special grades of PTFE composites filled with powders or fibers of thermally stable polymers, such as aromatic polyamides [4], polyimides, polyphenylenesulfides, poly-*p*-benzoate [5], etc. Earlier studies showed that polyoxadiazole (POD) fiber [6], can also be successfully used as PTFE filler yielding materials with high wear resistance and low abrasiveness [7].

POD-filled PTFE composites show high wear resistance with  $K_w = (5–10) \times 10^{-7} \text{ mm}^3/(\text{N m})$  and satisfactory strength, although filling in excess of 10 mass.% reduces mechanical strength due to increased agglomeration of the fibers and weak boundary layers [7]. This necessitates the search for efficient ways of the interphase region modification to counterbalance the negative trend.

Surface treatment of fillers is widely used in polymer composite technology to improve filler–matrix bonding through enhanced wetting and/or intensified physico-chemical interaction as a prerequisite condition for higher toughness, impact strength and other service properties [8]. Filler surface treatment with silanes or other reactive compounds, e.g. acids or acid anhydrides, widely used in case of other polymer binders, found little use in case of PTFE composites because of its high chemical inertness and high processing temperatures, exceeding thermal stability of most organic modifiers. Consequently, application of a thin fluorocarbon polymer layer, resembling the matrix polymer in surface and thermal properties, onto the filler surface appears a most promising approach

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**Table 1**  
Characteristics of Arselon™ POD fibers [6].

| Property                            | Value                  |
|-------------------------------------|------------------------|
| Density                             | 1.43 g/cm <sup>3</sup> |
| Tensile strength                    | ≥400 MPa               |
| Modulus of elasticity               | ≥10.5 MPa              |
| Mass loss after 30 min at 400 °C, % | ≤5.0                   |

in this case. It can be efficiently implemented by radio frequency (RF) plasma-enhanced deposition in the media of fluoroorganic compounds, fluoroolefins or fluoroalkanes. It was employed to develop thin fluorocarbon coatings for medicine and microelectronics [9–11], and, to a smaller extent, in polymer composite technology [12]. RF plasma in the medium of octafluorocyclobutane was successfully used for the treatment of carbon fibers (CF) resulting in improved wetting of the filler, increased strength, density, thermal conductivity and wear resistance of PTFE–CF composite [12].

The aim of the study is to investigate the effect of RF plasma treatment of Arselon™ POD fibers in the medium of tetrafluoroethylene on mechanical properties, structure and tribological behavior of PTFE–Arselon™ fiber composites.

## 2. Materials and methods

### 2.1. Materials

PTFE powder produced by Kirovo-Chepetsk chemical plant (Russia) under the trade name of fluoroplast-4, grade ПН (GOST 10007–80) was used as matrix polymer. Arselon™ POD fiber produced by Svetlogorsk Production Association “Khimvolokno” (Belarus) was used as a filler.

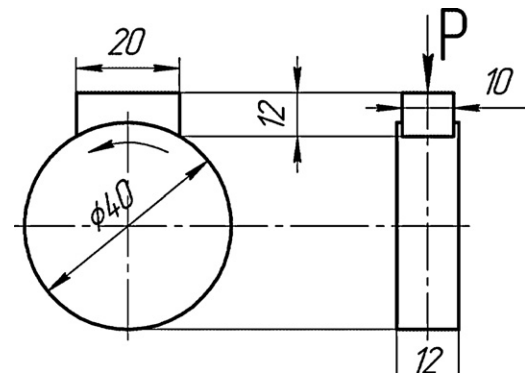
Some characteristics of the fibers as determined by the producer are listed in Table 1.

The conditions of RF-plasma treatment of the POD fiber yarns were similar to the conditions used earlier for modifying CF yarns for PTFE–CF composites [12]. Briefly, 5.28 MHz RF-plasma was excited at 0.5 kV in the medium of tetrafluoroethylene (TFE) at a pressure of ~0.1 kPa. Prior to modification the yarns intended both for modification and for fabrication of original fiber composites were thermally treated in air to remove the sizing. After modification the yarns were cut into ~10 mm long fibers.

Compositions of PTFE with the cut fibers were prepared by mixing dry components in a high speed mixer. The filler content was kept constant at 10 mass.%. The compositions were pressed into billets at 50 MPa at ambient temperature and sintered at  $375 \pm 5$  °C with heating–cooling rates of 40 °C/h. The composites based on the plasma-modified fibers are further referred to as modified composites and those based on the original fibers as original composites. Commercial PTFE composite F4K20 (USSR technical specification 6-05-1412-76) containing 20% of coke flour was used in tribological tests for comparison purposes.

### 2.2. Mechanical tests

Mechanical tests were conducted on Instron 5567 machine. The dumb-bell shaped specimens for the tensile tests with 10 mm × 2 mm × 1 mm dimensions of the working part were machined from the billets. Tensile tests were conducted at an ambient temperature at 20 mm/min crosshead rate. Compression tests were conducted with cylinder-shaped specimens measuring 15 mm in diameter and 22 mm high at 1 mm/min crosshead rate. The elasticity modulus was estimated from the initial portion of the stress–strain curves limited by 3% relative strain.



**Fig. 1.** Scheme of the conformable block-on-ring arrangement of the friction pair. The dimensions are in mm.

Dynamic mechanical analysis (DMA) of PTFE and its composites was conducted on DMA 8000 analyzer (Perkin Elmer) operated at 1 Hz loading frequency and 2 °C/min heating rate in bending mode using single cantilever configuration with an oscillation amplitude of 0.05 mm. The specimen had a rectangular shape measuring 12 mm × 30 mm × 1 mm.

### 2.3. Tribological tests

Tribological tests were conducted on 2070 CMT friction machine with conformable block-on-ring arrangement of the friction pair without external lubrication at 1 m/s sliding velocity. The scheme of the friction pair arrangement and the parts dimensions are shown in Fig. 1. The counterface in the shape of a roller was machined from 40× chromium steel hardened to HRC 46–48 and lapped to the roughness of  $R_a \leq 0.16$  μm. The composite specimens were sintered with dimensions specified in Fig. 1 and were used for the tests without additional machining. The tests were run to determine the wear rate and the load bearing capacity of the composites. Prior to the tests the specimens were rubbed-in at 1 MPa to ensure complete alignment with the counterface. Then the specimen was subjected to two 7 h friction tests at each load. Mass wear was measured after each 7 h test by weighing on an analytical balance with an accuracy of  $10^{-5}$  g. The mass losses  $\Delta m$  for the second 7 h-long test period at a given pressure were averaged for four parallel tests and the specific wear rate  $K_w$  was calculated according to the formula  $K_w = \Delta m / (\rho \cdot L \cdot P)$  where  $\rho$  is the composite density,  $L$  is the load and  $P$  is the friction path. Load was increased in steps of 100 N to produce the pressure increment of 0.5 MPa.

### 2.4. Structural and morphological analysis

IR spectra were recorded on a Nicolet™ 5700 FTIR-spectrometer in transmittance mode.

Differential scanning calorimetry (DSC) was conducted in Diamond DSC (Perkin Elmer) at 15 °C/min scanning rate in nitrogen flow. The sample thickness was ~0.5 mm, the sample weight was ~17 mg.

SEM observations were conducted in Vega II LSH/TESCAN/scanning electronic microscope; the sample surfaces were sputtered with gold to reduce the charging effects.

Vicat heat resistance temperature was determined at 5 kg load and 50 °C/h heating rate in air according to GOST 15088–83 with two specimens for each composite. The difference between the results of two parallel measurements lied within 3 °C.

Density measurements were conducted by hydrostatic weighing in distilled water on XS-204 balance/Mettler Toledo/.

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