

The study of the tribological, thermal and physical properties of phenylone C-2 based composites containing nonstoichiometric molybdenum and tungsten oxides

*Ksenija I. Karpenko**, *Evgeniy S. Novikov*, *Sergey A. Danilchenko* and *Viktor V. Avilov*

Rostov State Transport University, Theoretical Mechanics Chair, 344048 Rostov-on-Don
Rostovskogo Strelkovogo Polka Narodnogo Opolcheniya Sq. 2, Russian Federation

Abstract. The given paper lays bare the preparation and study of multifunctional tribocomposites with matrix based on Phenylone C-2 and the antifriction fillers. Besides, it contains a comparative evaluation of physical, mechanical, tribological and thermophysical properties of multifunctional tribocomposites to assess the effect of nanodimensional fillers.

1 Introduction

The latter-day engineering tends to put forward increased requirements for the development of highly effective wear-resistant materials to ensure reliable operation of friction units. The wear of rub elements leads to their premature failure. Hence, there is a need to create new antifriction composite materials that will meet the specified properties.

The given paper observes the procedures of obtaining and studying the multifunctional tribocomposites with Phenylone C-2 based matrix and the addition of antifriction fillers. Phenylone C-2 is form-stable in a wide temperature range. Besides, it is characterized by high strength properties and wear resistance, but its low tribological properties [1] demand the introduction of antifriction fillers. F4MB polytetrafluoroethylene powder (tetrafluoroethylene and hexafluoropropylene copolymer) in an amount of 10 wt.%, 3 wt.% nanodimensional particles of nonstoichiometric molybdenum and tungsten compounds and 7 wt.% high-performance C-52 cylinder oil were selected as antifriction fillers. Tungsten and molybdenum blue was obtained by treating acidified molybdate and tungstate solutions with reducing agents (zinc, hydrogen sulphide). Subsequently, blue was adsorbed from these solutions by surface-active substances.

The choice of molybdenum and tungsten compounds stemmed from their ability to possess variable degrees of oxidation. Nonstoichiometric molybdenum and tungsten compounds characterized by mixed degrees of oxidation have a layered structure; as a result they can be considered as promising antifriction additives. The highly dispersed molybdenum and tungsten oxides introduced into the polymer matrix of the composite

* Corresponding author: ksenija_karpenko@bk.ru

material ensure the absorption of mineral oil during the molding process, which gives these particles the ability to self-lubricate when they enter the friction area. Cylinder oil was selected due to its ability to maintain its tribological properties at high temperatures.

Tribological, thermal and physical properties of multifunctional tribocomposites were studied to assess the impact of nanodimensional fillers.

2 Study of composites thermophysical properties

The highly sensitive simultaneous thermal analyzer STA 449 F3 Jupiter by Netzsch was exploited to carry out thermogravimetric (TGA) and differential thermal (DTA) analysis to determine the value and sense of the thermal effects arising in the composite materials, which are connected with chemical reactions occurring at heating.

STA 449 F3 Jupiter is a top-loading system aimed at measuring the mass and thermal effects when heated at a constant speed at fixed intervals. The experimental data processing was conducted with the software provided by «Netzsch Proteus Analyses». The simultaneous thermal was carried out for both pure Phenylone C-2 and composite materials based on Phenylone C-2 matrix and polytetrafluoroethylene, with the addition of 3% wt. molybdenum and tungsten blue and 7% wt. C-52 cylinder oil.

A constant mass ratio between Phenylone C-2 and polytetrafluoroethylene remains 9: 1. All tests were carried out in platinum crucibles in the inert nitrogen atmosphere in the temperature range from 30 to 400 °C with a constant nitrogen flow equal to 20 ml / min.

The melting point and the vitrification temperature of the substances were determined by the bending of the differential thermal curves. It was proved that sharp fluctuations occur at the initial sections of the curves. They arise due to the uneven heating of platinum crucibles at the very beginning of the measurement cycle because of a relatively fast (10 °C / min) rate of temperature increase.

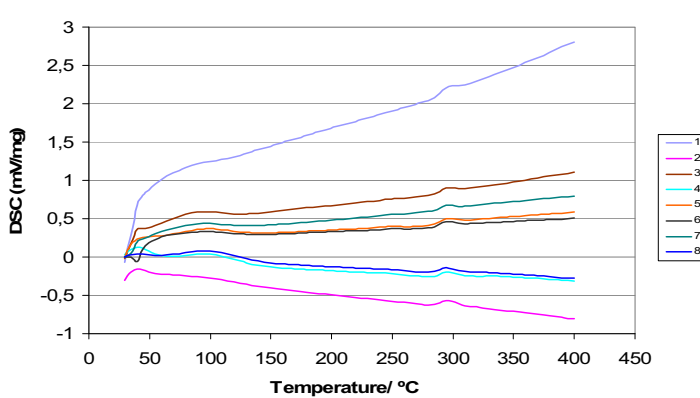


Fig. 1. DTA curves for various kinds of composites: 1) Phenylone C-2; 2) Phenylone C-2 + 10% F4MB; 3) Phenylone C-2 + 10% F4MB + 3% tungsten blue; 4) Phenylone C-2 + 10% F4MB + 3% molybdenum blue; 5) Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3% tungsten blue; 6) Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3% molybdenum blue; 7) Phenylone C-2 + 10% F4MB + 3% tungsten and molybdenum blue; 8) Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3% tungsten and molybdenum blue.

Figure 1 demonstrates an endothermic effect at about 290-300°C, which, according to the published data, corresponds to the Phenylone C-2 vitrification. A diffuse peak near 100°C refers to the vitrification temperature of F4MB polytetrafluoroethylene. There are no other thermal effects on DTA curves. It confirms the absence of chemical interaction between the

composite components. A diverse slope of the DTA curves indicates a difference in the heat capacity of the obtained materials. Heating above 400°C was not carried out because of the polymer decomposition.

3 Study of the composite tribological properties

An array of tribological tests were carried out on a friction machine of Amsler II 5018 type to assess the impact of nanodimensional fillers on the tribological properties of the studied tribocomposites. The test scheme is given in [2]. According to the results obtained (Figure 2, 3), the addition of 3% wt. tungsten or molybdenum blue to the composite leads to the reduction of the coefficient of friction by 20%. At the same time the introduction of 3% wt. tungsten and molybdenum blue decreases the coefficient of friction by 30% (Fig. 4). The addition of 7% wt. cylinder oil (C-52) to the composites containing molybdenum blue and a mixture of tungsten and molybdenum blue results in 2-fold decrease of the coefficient of friction (Fig. 2, 4) in comparison with the composite based on Phenylone C-2 and the addition of F4MB.

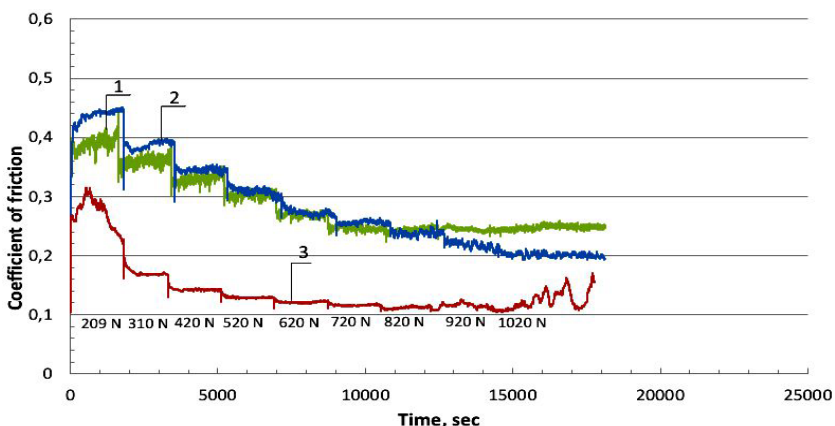


Fig. 2. Coefficient of friction-time diagram at a load range from 209 N to 1020 N and a sliding velocity equal to 0.4 m / s for Phenylone C-2 based composites: 1) Phenylone C-2 + 10% F4MB; 2) Phenylone C-2 + 10% F4MB + 3 % tungsten blue; 3) Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3 % tungsten blue.

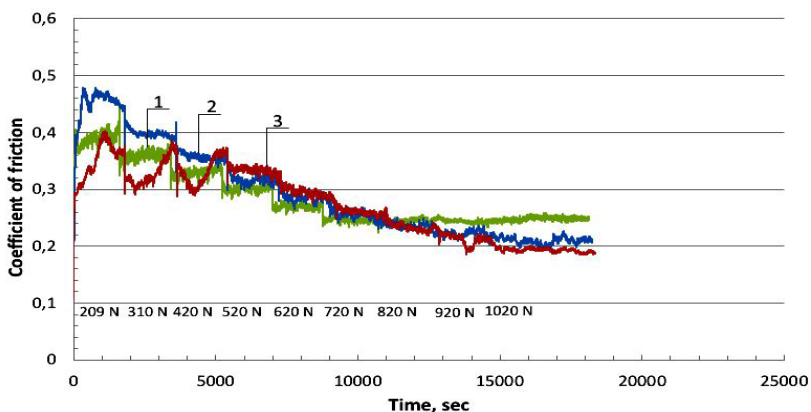


Fig. 3. Coefficient of friction-time diagram at a load range from 209 N to 1020 N and a sliding velocity equal to 0.4 m / s for Phenylone C-2 based composites: 1) Phenylone C-2 + 10% F4MB; 2) Phenylone C-2 + 10% F4MB + 3 % molybdenum blue; 3) Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3 % molybdenum blue.

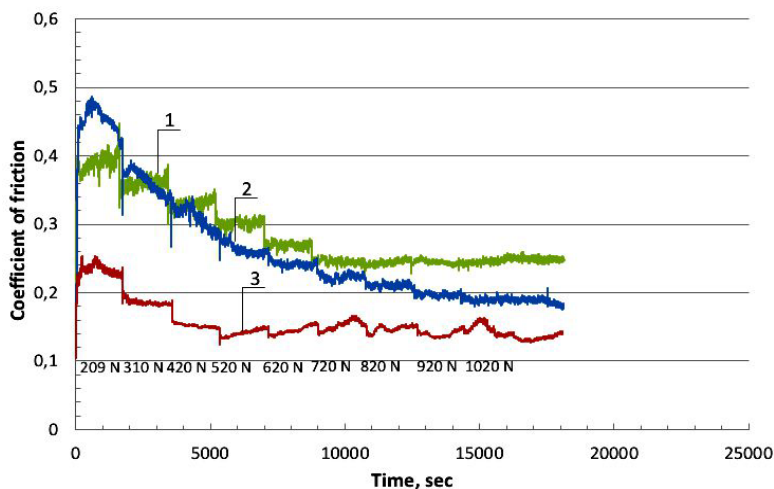


Fig. 4. Coefficient of friction-time diagram at a load range from 209 N to 1020 N and a sliding velocity equal to 0.4 m / s for Phenylone C-2 based composites: 1) Phenylone C-2 + 10% F4MB; 2) Phenylone C-2 + 10% F4MB + 3 % tungsten and molybdenum blue; 3) Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3 % tungsten and molybdenum blue.

The carried out tests provided us with the data to calculate the mass wear for all the samples studied. The results are presented in Table 1.

Table 1. Mass wear of laboratory samples after tribological tests

Material	Mass before testing, gr.	Mass after testing, gr.	Mass wear
Phenylone C-2 + 10% F4MB	11.0888	11.0556	0.0332
Phenylone C-2 + 10% F4MB + 3 % tungsten blue	10.9170	10.8317	0.0853
Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3 % tungsten blue	10.8650	10.8634	0.0016
Phenylone C-2 + 10% F4MB + 3 % molybdenum blue	11.3619	11.1828	0.1791
Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3% molybdenum blue	11.2183	11.1855	0.0328
Phenylone C-2 + 10% F4MB + 3 % tungsten and molybdenum blue	11.4114	11.3277	0.0837
Phenylone C-2 + 10% F4MB + 7% cylinder oil + 3 % tungsten and molybdenum blue	11.0402	11.0363	0.0039

4 Study of composite physical and mechanical properties

The study of physical and mechanical properties of created composites was carried out on NanoTest 600 by the method, described in [2,3]. A diamond indenter of conical shape with a cone angle of 90° and the radius of curvature 25 μm at the vertex was used during the experiment. The *P-h*-diagram, demonstrating the dependence of the load on the indenter's penetration depth was constructed and maintained for each shot. The analysis of the obtained curves was conducted by the Oliver-Pharr method [4,5]. The data obtained helped to calculate the values of the microhardness *H*, modulus of elasticity *E*, and the elastic recovery at each shot spot. The next step was the calculation of standard deviation (SD).

Moreover, there was worked out the ratio H/E и H^3/E^2 . The value H/E describes the ability of a material to change its size and shape during the deformation process. It can serve as a qualitative comparative characteristic of the material resistance to the deformation under mechanical loading. It is also used to characterize the wear ability of materials at friction. H^3/E^2 is the qualitative comparative characteristic of the plastic deformation resistance. The obtained data is manifested in Table 2.

Table 2. Average values of the physical and mechanical properties of the studied composites

Material	Microhardness H , GPa	Modulus of elasticity E , GPa	Elastic recovery	H/E	H^3/E^2
Phenylone C-2	0.395	5.950	0.226	0.066	$1.7 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB	0.349 SD=0/009	5.116 SD=0.068	0.225 SD=0.008	0.068 SD=0.001	$1.624 \cdot 10^{-3}$ $SD=0.096 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB + 3 % tungsten blue	0.336 SD=0.072	5.542 SD=0.687	0.257 SD=0.061	0.06 SD=0.008	$1.294 \cdot 10^{-3}$ $SD=0.57 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB + 3 % tungsten blue + 7% cylinder oil (C-52)	0.226 SD=0.04	3.694 SD=0.578	0.202 SD=0.041	0.062 SD=0.009	$0.887 \cdot 10^{-3}$ $SD=0.322 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB + 3 % molybdenum blue	0.283 SD=0.05	5.316 SD=0.623	0.202 SD=0.048	0.053 SD=0.007	$0.847 \cdot 10^{-3}$ $SD=0.384 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB + 7% cylinder oil (C-52) + 3% molybdenum blue	0.251 SD=0.059	4.512 SD=0.741	0.203 SD=0.049	0.055 SD=0.008	$0.82 \cdot 10^{-3}$ $SD=0.363 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB + 3 % tungsten and molybdenum blue	0.382 SD=0.015	5.610 SD=0.196	0.311 SD=0.015	0.068 SD=0.002	$1.777 \cdot 10^{-3}$ $SD=0.16 \cdot 10^{-3}$
Phenylone C-2 + 10% F4MB + 7% cylinder oil (C-52) + 3% tungsten and molybdenum blue	0.289 SD=0.03	4.408 SD=0.281	0.256 SD=0.031	0.066 SD=0.005	$1.264 \cdot 10^{-3}$ $SD=0.262 \cdot 10^{-3}$

Based on the results of the research, it was found the composite with Phenylone C-2 based matrix with the addition of 10% wt. F4MB and 3 % wt. tungsten blue possesses the highest values of microhardness and modulus of elasticity. Besides, it is characterized by high values of the coefficient of elastic recovery, resistance to deformation under mechanical loading and resistance to plastic deformation.

The standard deviation values for each of the above mentioned properties turned out to be the lowest for the sample «Phenylone C-2 + 10% F4MB + 3 % tungsten and molybdenum blue». It indicates that the given composite has more homogeneous structure.

It was established that the physical and mechanical properties of «Phenylone C-2 + 10% F4MB + 3 % tungsten and molybdenum blue» sample were close to those of the initial Phenylone C-2. The difference in the values of microhardness and modulus of elasticity does not exceed 6%.

Therefore, we were able to obtain a composite material possessing nearly the same physical and mechanical properties as Phenylone C-2, but better tribological ones.

Conclusions

The given paper reveals a comparative evaluation of physical, mechanical, tribological and thermal properties of multifunctional tribocomposites to assess the effect of nanodimensional fillers. The results of thermal and physical researches demonstrate that the initial structure of the materials studied remains unchanged. This fact is evidenced by the peak absence on DTA curves.

Based on the data obtained during the tribological tests, it was concluded that the introduction of nanodimensional powders of tungsten and molybdenum non-stoichiometric compounds into the tribocomposite leads to an increase in mass wear as compared to the initial Phenylone C-2 + 10% F4MB matrix, despite the decrease in the coefficient of friction. At the same time, the addition of cylinder oil (C-52) to these composites results in an even greater reduction in the coefficient of friction and a decrease in mass wear.

The results of the conducted study of physical and mechanical properties prove that except for Phenylone C-2 + 10% F4MB + 3 % tungsten and molybdenum blue, the values of microhardness and modulus of elasticity for the other samples were lower in comparison with the initial matrix. The samples with the added cylinder oil (C-52) show the most significant reduction that reached 33%.

The received data can be recommended for specifying the material production.

This study was supported by the Russian Science Foundation (grant number 14-29-00116).

References

1. Yu.K. Mashkov, Z.N. Ovchar, M.Yu. Baybaratskaya, *Polymer Composite Materials in Tribotechnics* (in Russian) (Nedra, Moscow, 2004)
2. P.G. Ivanochkin, S.A. Danilchenko, E.S. Novikov, *Procedia Engineering*, **150** (2016)
3. P.G. Ivanochkin, S.A. Danilchenko, E.S. Novikov, D.S. Manturov, *Springer Proceedings in Physics*, **207** (2018)
4. Yu.I. Golovin, *Nanoindentation and Its Possibilities* (in Russian) (Mechanical Engineering, Moscow, 2009).
5. W.C. Oliwer, G.M. Pharr, *J. Mater. Res.*, **7**, 6 (1992)