

Великими можливостями для підвищення довговічності вузлів тертя машин і механізмів характеризуються полімерні композиційні матеріали зміцнені органічними волокнами. Дані композити з успіхом конкурують з кольоровими металами та їх сплавами, а в деяких випадках й перевершують полімерні та металеві аналоги за своїми властивостями. У зв'язку з цим досліджено вплив органічного волокна лола на експлуатаційні показники ароматичного поліаміду фенілон марки С-1 та можливість застосування розроблених полімерних композиційних матеріалів.

Експериментальними дослідженнями підтверджено, що армування фенілону органічним волокном лола в кількості 5–15 мас. % призводить до покращення його експлуатаційних характеристик. Це обумовлено впорядкуванням надмолекулярної структури базового полімеру внаслідок введення органічного волокна. Так, на межі поділу «фенілон-наповнювач» чітко спостерігається трансформація глобулярної структури в'язучого у фібрилярну. Це призводить до позитивного ефекту: збільшуються енергія руйнування (у 1,5 рази) та хімічна стійкість (у 1,1–1,36 при витримці у 5 % HCl та 1,27–1,6 у 10 % HCl). При цьому слід зазначити, що розроблені органоластики стійкі при температурі 673 К, в той час як вихідний полімер починає інтенсивно руйнуватися вже при 400 К. Зокрема встановлено, що при подальшому збільшенні масової частки наповнювача дані показники погіршуються, що обумовлено недостатньою адгезією між наповнювачем та в'язучим.

Використання органічного волокна лола (в кількості 5–15 мас. %) дає змогу отримати композити з покращеними експлуатаційними характеристиками: підвищеними термічними та хімічними показниками, високою стійкістю до дії ударних навантажень. Таким чином, є підстави стверджувати про перспективність застосування волокна лола як наповнювача для композитів. Органоластик із оптимальним вмістом волокна (15 мас. %) рекомендовано для виготовлення деталей трибовузлів сучасного обладнання натомість кольоровим металам та їх сплавам завдяки достатньо високим експлуатаційним властивостям

**Ключові слова:** фенілон, поліамід, органічне волокно, лола, органоластики, термостійкість, хімічна стійкість, структурування, трибовузли

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# DESIGNING THE ORGANOPLASTICS BASED ON AROMATIC POLYAMIDE, STUDY OF THEIR OPERATIONAL PROPERTIES AND APPLICABILITY

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## 1. Introduction

Tribological nodes in modern equipment in the weaving, agricultural, and automotive machinery, operated under harsh conditions, are designed for a long and reliable service life [1]. Given the above, special requirements are put forward to modern polymeric composite materials (PCM) for tribotechnical purposes. These include enhanced wear-, thermal-, and chemical resistance, high mechanical and thermophysical characteristics, the ability to form stable surfaces (transfer films), resistance against the effect of aggressive media [2]. These criteria can be implemented by selecting a polymeric matrix and a filler to construct PCM for tribological nodes.

Since a polymeric matrix is an important component that warrants most required operational characteristics of the composite [4], it should be carefully chosen. It is known

from the scientific literature [5] that the use of thermoplastic binders (polyamide, polytetrafluoroethylene, polyester, etc.) is the most promising as it makes it possible to obtain PCM that can be operated under extreme conditions. In order to obtain friction units capable of working under difficult conditions and aggressive environments, taking into consideration the cost of materials, it is advisable to use polyamides. Due to the high thermal, tribological, and mechanical properties and a relatively low cost, they attract great interest among other thermoplastic binders for creating PCM for tribotechnical purposes.

Given the above, it is a relevant task to undertake a research into the development and implementation of new PCM based on polyamide, capable of operating under the influence of high impact loads, temperatures, and aggressive environments (acids, alkalis, etc.).

## 2. Literature review and problem statement

A known method to obtain PCMs for tribotechnical purposes is the introduction to the polymeric matrix of anti-friction layered lubricants (graphite, molybdenum disulfide, shungite, etc.). However, authors of work [6] showed that the use of anti-friction fillers results in a significant reduction of strength and pore formation inside the volume of a polymer, which reduces operational characteristics of such PCMs. Although the introduction of graphite can significantly reduce the friction coefficient, as demonstrated in paper [7], the PCMs that are filled with layered lubricants cannot be operated under harsh conditions and in aggressive environments.

Authors of study [8] emphasize that the reinforcement of polyamides with long carbon fibers makes it possible to obtain reliable structural plastics for tribotechnical purposes. However, they arrange the carbon fibers along a single direction, which leads to the anisotropy of the materials' properties; in addition, one cannot achieve good adhesion between the components. All that makes it impossible to apply the specified PCMs at heavily-loaded friction units.

Paper [9] shows that the orientation of short carbon fibers significantly affects the mechanical and tribological properties of PCMs based on polyamides. In this case, depending on the orientation of fibers, the carbon plastics are obtained for different purposes. However, due to the high cost of carbon fibers, manufacturing friction units from carbon plastics is not cost effective from the economic point of view. Such composites are used in military and space equipment.

Currently, in order to design composites for anti-frictional purposes, other fibrous fillers are also actively used, among which the important role belongs to organic fibers (OF) [10, 11]. The expediency of using different OF for reinforcing polyamides is confirmed by the improved tribotechnical and thermo-physical characteristics [12, 13].

Friction components in machines and mechanisms are equipped with parts from organoplastics (OP), which are characterized by high environmental friendliness due to the possibility of recycling and self-lubricating ability. They demonstrate reliability (due to improved wear resistance, fatigue and damping capability) and are cost-effective (they do not require machining even when fabricating non-standard parts) [14, 15]. Such PCMs are quite capable of competing with known standard materials (bronze, babbitt, cast iron, etc.). However, despite the diversity of OP, their use is constrained by the insufficient adhesion between the polymeric matrix and a filler, as well as the OF agglomeration [16], which leads to the anisotropy of properties and pore formation.

All this allows us to argue that the research aimed at developing OP for tribotechnical purposes for heavily loaded friction units capable of operating in aggressive environments is a promising task. In this case, insufficient certainty of the influence of the ladder-type polyarylene fiber lola on the operational characteristics of aromatic polyamide necessitates further research in this direction.

## 3. The aim and objectives of the study

The aim of this work is to study influence of the organic fiber lola on the operational characteristics of the aromatic polyamide phenylone.

To accomplish the aim, the following tasks have been set:

- to consider the effect of a filler on the thermal and chemical stability of the aromatic polyamide phenylone;
- to investigate the effect of the organic fiber lola on the structure and intercomponent interaction between organoplastics.

## 4. Materials and methods to study the effect of the organic fiber lola on the thermal and chemical stability of aromatic polyamide

### 4.1. Examined materials used in the experiment

A polymeric matrix used was the aromatic polyamide phenylone, brand C-1 (TU 6-05-221-101-71), a pink dispersed powder characterized by high fatigue strength and the capability to withstand significant statistical and dynamic loads. Specifically, it is resistant to exposure to elevated temperatures (able to operate up to 523 K), radiation, and aggressive environments (acids, diluted and concentrated alkalis). It is intended for manufacturing plastic articles by a direct pressing method [17, 18].

The filler used was the fiber from the heterocyclic ladder-type polyarylene – lola (Table 1), characterized by exceptional fire-, thermal resistance and stability against the effect of concentrated acids and organic solvents. The fiber was designed at the All-Russian Scientific and Research Institute of Polymer Fibers (VNDIPV) (Mytyshi, Russia) based on the monomers anhydride and amine [19].

Table 1

Properties of the fiber lola

Indicators	Magnitude
Density, g/cm <sup>3</sup>	1.4–1.5
Modulus of elasticity, GPa	30–45
Strength, cN/tex	35–45
Elongation strength, %	3–6
Strength retention	
– at 573 K;	70–85
– after heating at 573 K (over 100 hours)	80–9
Moisture content (n. c.), %	2–3

Preparation of OP based on phenylone containing 5–20 % by weight (3 mm long) was carried out by dry mixing at a unit with a rotary electromagnetic field (0.12 Tl) using ferromagnetic particles. Next, the particles were removed from the prepared compositions by a magnetic separation method. The mixtures obtained in this manner were molded into finished products by a compression pressing method. Prior to molding, the polyamide phenylone C-1 and the compositions should be thoroughly dried. Processing undried materials into articles worsens their strength characteristics, which leads to the formation of surface defects (shells, bullae, etc.). We dried the workpieces at the thermal cabinet SPT-200 for 2–3 hours at 473–523 K. The prepreg form the thermal cabinet was immediately loaded to the mold, heated to 523 K. Next, the material was heated to 598 K and aged without loading over 10 minutes, then it was exposed to aging for 10 minutes at a load of 30 MPa at the same temperature. Next, the article was cooled at constant load to a temperature of 543 K and exposed to pressing-out [20]

**4. 2. Procedure for determining indicators of samples' properties**

We performed differential thermal analysis (DTA) of phenylone C-1 and OP on its basis using the method of thermogravimetric analysis (TGA), employing the derivatograph Q-1500D, the system of Paulik-Paulik-Erdey made by IOM (Hungary). Temperature interval in our studies was 273–800 K, the rate of rise in temperature – 5 K/min; mass of the examined sample – 200 mg.

We estimated chemical resistance (GOST 12020-72) based on the magnitude of sample swelling (Ø50, height 3 mm) in acid HCl at different temperatures. It follows from GOST: the samples “before” and “after” the study were weighed in a glass container, with a lid closed, with an accuracy of up to 0.0002 g; we also determined their linear dimensions (not less than at 5 points). Upon completing the study, the samples were washed with water, wiped out with a lint-free cloth, weighed; we measured their linear dimensions. The temperature was maintained using a heating system equipped with special devices that make it possible to set the required temperature and maintain it up to ±2 K. In the study we used a container with a tightly closed lid to prevent acid vapors; to avoid an emergency, we wore work clothes (gas mask, bathrobe, gloves) and enabled artificial ventilation.

To analyze the morphology of change in the polymeric matrix structure when introducing an organic fiber, we used the raster universal electron microscope UEMV-100K. We analyzed the fragile fracture of samples, obtained under the influence of liquid nitrogen, by sawing, in advance, a graphite film onto the examined surface.

Microhardness at the interface “binder-fiber” was determined using the microhardness meter PMT-3M (GOST 9450-76).

Density of the samples was determined by using an additive and a hydrostatic method in accordance with GOST 15139-69.

The process of destruction of phenylone and the developed OP under impact loads was investigated employing the pendulum drop-hammer “CHEAST” (GOST 4647-80). The drop-hammer is equipped with a “Phractoscope” system, which, by using electrical converters mounted onto an impact pendulum, makes it possible, directly during impact, to measure the effort to which the examined sample is exposed (50×1×5 mm<sup>3</sup>). The acquired signal is registered as a load-time diagram by the graphical registrar.

**5. Results from studying the effect of the organic fiber lola on the operational characteristics of the aromatic polyamide phenylone**

**5. 1. Results from studying the thermal and chemical stability of phenylone and organoplastics based on it**

Fig. 1 shows the dependences of mass loss (*M*, %) on temperature (*T*, K) for phenylone C-1 and organoplastics based on it, derived from a thermogravimetric analysis. Results from the thermogravimetric analysis (Fig. 1, Table 2) indicate high thermal stability of both the starting phenylone C-1 and organoplastics based on it. The shapes of all curves “mass loss – temperature” are similar, that is the decomposition of the polyamide filled with fiber proceeds similar to phenylone. The region of operating temperatures almost does not demonstrate any qualitative changes in the thermal

behavior of the filled systems; however, the fillers have a significant impact with an increase in temperature.

Analysis of the results obtained reveals that the heat resistance of OP increases, passing through a maximum at the filler content of 15 % by weight, and then there is a decrease. It is interesting to note that phenylone, reinforced with the fiber lola in the amount of 15 % by weight, is thermally resistant up to a temperature of 673 K. At the same time, the starting polymer intensively decomposes at the same temperature.

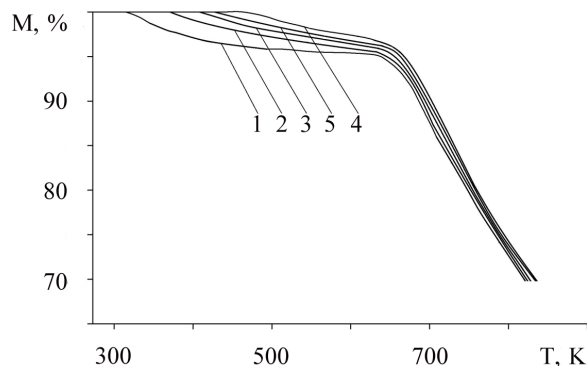


Fig. 1. Weight loss dependence (*M*, %) on temperature (*T*, K) for phenylone C-1 (1) and organoplastics based on it, containing the fiber lola: 5(2); 10(3), 15(4), 20(5) % by weight

Table 2  
Dependence of phenylone thermal stability on content of the fiber lola

Fiber content, % by weight	<i>T</i> <sub>0</sub> , K	<i>T</i> <sub>10</sub> , K	<i>T</i> <sub>20</sub> , K
0	303	688	743
5	373	691	752
10	412	700	759
15	462	704	762
20	431	695	755

Note: *T*<sub>0</sub>, *T*<sub>10</sub>, *T*<sub>20</sub> – the temperatures of losses, 0, 10, 20 % by weight, respectively, K

The dependence of phenylone swelling in hydrochloric acid over 10 days on the concentration of acid and the content of the lola fiber is given in Table 3. Table shows that an increase in the concentration of hydrochloric acid leads to a decrease in the magnitude of swelling of organoplastics by 1.1–1.36 (for 5 % HCl) and by 1.27–1.6 (for 10 % HCl) times, respectively, compared to the starting polymer.

Table 3  
Dependence of phenylone swelling in hydrochloric acid over 10 days on the concentration of acid and content of the fiber lola

Hydrochloric acid, 293 K	Swelling (%) at fiber content (% by weight)				
	0	5	10	15	20
5 %	0.79	0.73	0.68	0.58	0.59
10 %	0.55	0.43	0.38	0.34	0.41

Fig. 2 shows the results from studying the temperature dependence of phenylone swelling (1) and organoplastics on its basis (2) with a fiber content of 15 % by weight at aging over 48 hours.

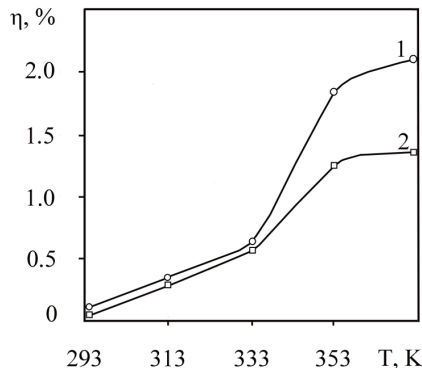


Fig. 2. Temperature dependence of phenylone swelling (1) and organoplastics on its basis (2) with a fiber content of 15 % by weight when aged for 48 hours

It should be noted that an increase in the temperature of hydrochloric acid from 293 to 373 K (Fig. 2) decreases the swelling of OP with a fiber content of 15 % by weight: it is considerably less than that of the binder.

**5. 2. Studying the effect of the organic fiber lola on the structure and intercomponent interaction between organoplastics**

Since earlier studies have revealed that the introduction of the OF lola in the amount of 5–15 % by weight leads to an increase in the density of the phenylone structure, the next task was to study the structure of the developed organoplastics. Fig. 3, 4 show the microstructures of the polymer and the organoplastics based on it, respectively.



Fig. 3. Microstructure (×10,000) of the base polymer

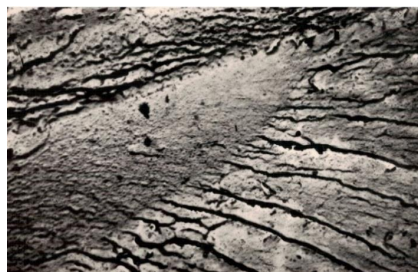


Fig. 4. Microstructure (×10,000) of the organoplastic based on phenylone with a fiber content of 15 % by weight

Fig. 5, 6 shows the averaged dependences of effort and energy as a function of time. The results obtained indicate that the energy of OP destruction is 1.5 times higher than that for phenylone.

In both cases, there is a linear increase in stress that develops to a maximum and results in the destruction of the sample. Next, there is a sharp drop in the stress and one ob-

serves a section predetermined by the fluctuations of debris. Fig. 7, 8 show the dependences of density and microhardness at the interface on content of the fiber lola.

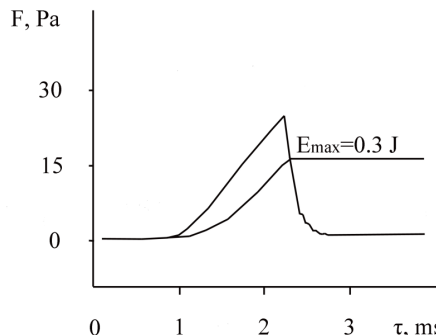


Fig. 5. Averaged dependences of the effort applied to the sample and the absorbed energy for phenylone as a function of time

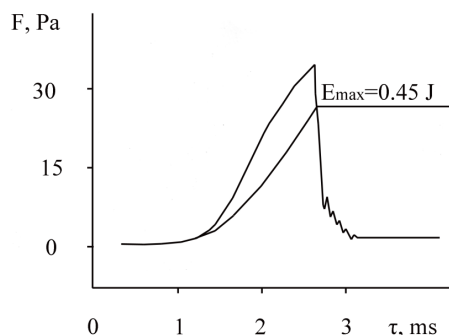


Fig. 6. Averaged dependences of the effort applied to the sample and the absorbed energy of the organoplastic with a fiber content of 15 % by weight as a time function

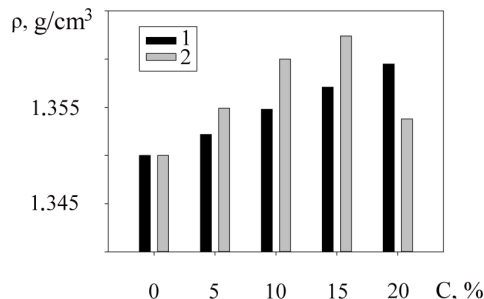


Fig. 7. Dependence of the estimated (1) and experimental (2) density ( $\rho$ , g/cm<sup>3</sup>) of phenylone on content of the fiber lola (C, %)

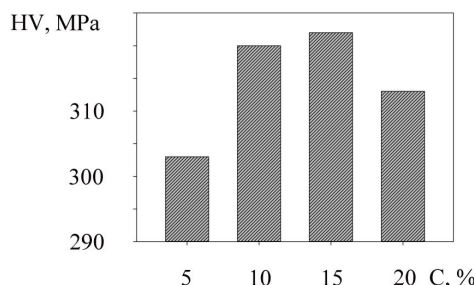


Fig.8. Dependence of microhardness (HV, MPa) of phenylone on content of the fiber lola (C, %) at the interface "binder-fiber"



The derived dependences of the effort applied to the sample and the absorbed energy are in good agreement with studies into the density and microhardness at the interface “binder-fiber”.

## 6. Discussion of results from studying the thermal and chemical stability of phenylone and the organoplastics based on it

The increase in thermal and chemical stability of phenylone when introducing the organic fiber lola can be explained in the following way. The appearance of OF in a polymeric matrix reduces the mobility of molecular chains of the polymer as a result of improving the supramolecular structure of the binder, which leads to an increase in its thermo-oxidative and chemical stability [21, 22].

These conclusions are confirmed by the results from an X-ray diffraction analysis reported in paper [12]. The introduction of a filler helps reduce the inter-planar and the shortest inter-atom distances by 6 % and 7.5 %, respectively, while increasing the average size of crystallites by 15–24 %. This indicates the intercomponent interaction between a binder and a filler.

Deterioration in the examined characteristics at the content of filler of 20 % by weight is due to increased defects in a material (formation of pores). The pores are formed due to the loosening of the structure at the interface “polymer-fiber”, which begins to prevail over the ordering of macromolecules of the binder [23]. This is evidenced by the dependence of the estimated and experimental density of phenylone on content of the fiber lola (Fig. 7), which is consistent with the results from tribological and thermophysical studies in [12, 13].

This dependence shows that at the content of a filler of 5–15 % by weight the experimental density of OP, determined by the hydrostatic method, is higher than the estimated one. It follows that the structure of the resulting organoplastics is denser than that intended. At the content of a filler of 20 % by weight, we established an opposite dependence – experimental density is considerably less than estimated, hence the material contains voids.

On the other hand, studying microhardness at the interface “phenylone-fiber” (Fig. 8) revealed that with increasing the number of fibers up to 20 % by weight one observes a decrease in contact between the binder’s macromolecules and the filler’s surface. This study also confirms the presence of defects (voids, pores) at the boundary polymer-fiber at the content of filler of 20 % by weight.

Phenylone has a globular structure (Fig. 3). The surface of a chip is uniformly covered with spherical formations of the order of 30 nm, which is consistent with the scientific literature [4]. For amorphous polymers with flexible chains, the simplest type of structures is globular, which consists of a single or many macromolecules rolled in spherical particles. The possibility of macromolecules to be wrapped in a tangle is determined by their high flexibility and by the predominance of intramolecular interaction forces over intermolecular forces. Globules can be regarded as three-dimensional formations from macromolecules or their segments, but with a close order implemented in the position of axes of chain molecules.

Electron-microscopic investigations of organoplastics (Fig. 4) show significant changes in the nature of cleaved

phenylone in the presence of organic fibers. The biggest changes occur at a near-border layer and at the interface “binder-filler”: the supramolecular structure of the binder is ordered around OF, thereby forming the new structural elements – fibrils.

It is known from paper [25] that the formation of globular structures in linear polymers is accompanied by deterioration of not only mechanical properties, but also thermal and chemical resistance. The introduction of fillers can significantly improve the characteristics of composites, since the properties of polymers are largely determined by the supramolecular structure formation, which may vary in their presence.

Therefore, the increase in the chemical and thermal stability of OP compared to the starting polymer relates to the ordering of the supramolecular structure of the binder. Thus, at the interface “phenylone-fiber” one clearly observes the transformation of the globular structure of the binder (Fig. 3) into fibrillary one (Fig. 4). And this, as known from [25], leads to an increase in the thermal and chemical stability of PCM.

It should be noted that the formed fibrillary structure increases strength at the interface polymer-filler, which is evidenced by analysis of the averaged dependences of the effort applied to the sample and the absorbed energy of phenylone and the organoplastics. Thus, the resulting character of the OP curve indicates a weak capability to scatter energy. This is due to the processes of ungluing and separating the fiber from the binder at destruction. Given the high strength of the composite ( $F_{\max}=35.2$  MPa from Fig. 6, if projected onto the  $x$  axis, we shall obtain this number), this indicates a good adhesion between the base polymer and the fiber [26, 27].

Positive results from a laboratory study allowed us to proceed to industrial tests. A failure-free operation of weaving machines is inextricably linked to the operability of friction nodes (in 90 % of cases), whose repair, in the course of their faulty operation, consumes a significant economic resource. The chosen object of our study was the friction nodes at the actuator mechanism for the doffer in a weaving machine made by Tongda TD (China), which were mounted at NVO “Sinergia” (Kyiv, Ukraine) (Fig. 9).



Fig. 9. Friction unit at the actuator mechanism for the doffer in the weaving machine Tongda TD, equipped with a roller bearing made from organoplastic

The object of research was selected based on considerations that these nodes operate under conditions of friction without lubrication and at elevated vibration and pollution. In this case, standards actuator nodes for the doffer that are

made from cast iron operate only for 6,336 hours, which is approximately 1 year. Further utilization of these nodes is impossible due to catastrophic wear.

The experimental parts made from organoplastic (a fiber content of 15 % by weight), which were installed to replace standard roller bearings, worked for 8,640 hours. Industrial tests have showed no deviations in experimental nodes from the normal operation; therefore, the designed parts were left for further operation.

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## 7. Conclusions

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1. The results obtained indicate that the introduction of an organic fiber increases chemical stability of phenylone (by 1.1–1.36 at aging in 5 % HCl and by 1.27–1.6 in 10 % HCl). It should be noted that the designed organoplastics are resistant at a temperature of 673 K, while the starting polymer begins to destroy intensively at 400 K.

2. We have examined the influence of the organic fiber lola on the structure and intercomponent interaction be-

tween organoplastics. It was established that the growth of chemical and thermal stability is connected to the ordering of the supramolecular structure of the binder and to the increase in intermolecular interaction. The experimental parts made from organoplastic (a fiber content of 15 % by weight), installed in the friction unit at an actuator mechanism for the doffer in a weaving machine Tongda TD, demonstrated better efficiency than that by standard ones. The standard components fail due to a catastrophic wear in one year, while the designed parts did not demonstrate any deviations from norm after having worked for more than a year.

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