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Mechanics

Effect of Running-in on the Wear Pattern of Heavy Loaded Friction Surfaces of Steel in the Environment of Nanolubricants

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Experiments have been carried out on the four-ball friction machines, in the friction knot of which sliding and rolling frictions are implemented. Two oils, containing additives – amorphous fine carbon nanoparticles (AFC) and Fe-cluster doped carbon nanotubes (CNTs), were used as nanolubricants. The experimental technique includes tests in the selected basic load mode, which were conducted both without preliminary running-in of steel ball friction surfaces and after running-in. The working surfaces have been examined prior to works, after breaking-in and after work in the basic load mode, respectively. The surface have been studied by means of scanning electron microscopy (SEM) and Auger electron spectrometry (AES). Experimental results of operation in the basic load mode have showed that e.g. fatigue wear (pitting) ulcers appear well after on balls passing the run-in process, than on those without preliminary run-in. It is shown that in the operation process under mentioned conditions there takes place formation at friction surface of a thin secondary layer with mixture of tribosynthesis products from nanoparticles of AFC and Fe-cluster doped CNTs modifiers of allotropic modifications of carbon nanoforms: graphite, diamond-like carbon and diamond nanoparticles. This layer presumably promotes wear reduction and less formation of surface damages in the end of run-in process and at the basic load stage. *© 2023 Bull. Georg. Natl. Acad. Sci.*

wear, running-in, amorphous fine carbon, Fe-cluster doped CNTs, friction surface

It is known that during friction irreversible dissipative processes run, which originate at the friction surfaces resulting from several parallel physical-chemical transformations. Nature, morphology and physical/chemical properties of particles/nanoparticles of lubricant additives may have significant impact on the dynamics of processes of structural and phase transformation during friction under lubrication conditions and formation of secondary adaptive structures in the surface layers. The mentioned structures provide screening protection from destruction of basic metals surfaces of the friction pairs [1, 2].

Modification of tribopairs' surface directly in the running-in mode under lubricating oils environment using interaction with different particles/

nanoparticles of lubricant additives suspended in oil is an effective approach to the metal materials wear resistance increase. This approach is successfully used for improvement of ability to withstand the damages of heavy loaded tribopairs. That's why the tribonics faces a critical task of creating a new generation lubricating compositions that foresees the search for new materials and development of technologies of their entering into oil as a non-sedimented additive [3, 4].

In our previous works, we showed the opportunity of getting a new composition of highly-effective transmission oils with amorphous finely-dispersed carbon additives for reduction of friction and wear of heavy loaded steel friction surfaces [4, 5]. It should be noted that the phenomenon of graphite and diamond crystallites tribosynthesis was observed for the first time. The mentioned phenomenon has been obtained during friction of steel parts in the environment of oil containing suspended nanoparticles of amorphous carbon [5].

The recent development of nanotechnology related to production of new carbon nanoforms (CNTs, CNPs, CNWs, CNBs) doped with different atom clusters [6, 7] has come up to new frontiers through creation of new functional nanolubricants with superior performance, which are widely used in automotive, power, space and other industries.

Structural features of surface film and near-surface layers destruction were observed in the work [8] allowing us to make conclusion that formation of debris particles is initiated by fatigue damages due to significant plastic and elastic deformations developed in the thin surface and near-surface layers, respectively, at long-lasting and repeating actions, and combined action of normal and tangential forces on the friction surface.

It can also be noted that in our recent work [9] it was shown that a thin (~1 μm) amorphous (entropy "pumped") film (almost without roughness) composed of products of carbon nanoparticles conversion into graphite, diamond-like carbon and pure diamond crystallites (sp^3 state) using tribosyntesis at the running-in stage on the conjugated friction surfaces. This heterophase film makes a positive gradient of mechanical properties, which may ensure necessary conditions for normal external friction and wear without scuff, up to origination of critical fatigue stresses.

Related to the above-mentioned, the given work set a goal of investigation of running-in effect on the wear pattern of heavy-loaded friction surfaces of steel in the environment of nano-lubricants containing amorphous fine carbon nanoparticles (AFC) and Fe-cluster doped carbon nanotubes (CNTs) under conditions of rolling friction and sliding friction.

Experimental

Experiments have been carried out on the four-ball friction machines, in the friction knot of which sliding and rolling frictions are implemented [4]. The experimental technique includes tests in the selected basic load mode, which were conducted both without preliminary running-in of steel ball friction surfaces and after running-in.

Two oils, containing additives fine carbon (FC) nanoparticles – amorphous fine carbon nanoparticles (AFC) and Fe-cluster doped carbon nanotubes (CNTs) (further oils 1J and 1G), have been used as lubricants.

According to investigations, the run-in of balls at 400 N axial load during 10 sec has been taken as the optimum running-in mode in case of sliding friction, while run-in of balls at 3000 N axial load for 20 min – in case of rolling friction.

Under conditions of sliding friction the following indicators were determined: d_w, P_{cr} and P_w, which represent ball wear scar diameter, critical load and welding load; and time to pitting beginning has been taken in case of rolling friction.

The mentioned indicators have been determined when testing oils containing carbon additives, prior to and after balls run-in. In case of sliding friction, the test duration was 10 sec, while in case of rolling

Table 1. Wear indices of friction surfaces of steel balls working in nano-dispersed carbon-containing oil medium without running-in and after it (four-ball friction machine, sliding friction, run-in time t = 10 s and axial load 400 N, test duration at the load stages t = 10 s)

Name of oils	without running-in			after running-in		
	d_w, mm	P_{cr}, N	P_w, N	d^1_w, mm	P^1_{cr}, N	P^1_w, N
Oil 1J	0,40	700	3220	0,36	790	3340
Oil 1G	0,39	720	3300	0,34	820	3480

Table 2. Time to pitting of friction surfaces of steel balls working in the medium of nano-dispersed carbon-containing oils without running-in and after it (four-ball friction machine, rolling friction, run-in time t = 20 min and axial load P = 3000 N)

Name of oils	without running-in	after running-in
	average time to pitting, min	
Oil 1J	106	119
Oil 1G	102	110

friction, the load was 4500 N, and test duration was measured by the time to ball pitting beginning.

The working surfaces were examined prior to works, after breaking-in and after work in the basic load mode, respectively. The surface was studied by means of scanning electron microscopy (SEM) and Auger electron spectrometry (AES).

Right after friction tests, the morphology, composition and phase constitution of steel ball wear surfaces have been examined using the scanning electron microscope JSM-6510LV (JEOL, Japan) and element and phase distribution at physical surface of friction and in depth from free surface of investigated specimen after SEM-EDX experiments has been exposed to the Auger electron spectrometry (AES) examination, carried out using spectrometer LAS-2000 (RIBER, France) with primary electron beam energy Ep = 5 keV.

Results and Discussion

According to experimental results, balls run-in causes substantial reduction of wear and damages (scuff, welding, pitting) of their surfaces, while after run-in – operation at relatively high (so-called “operating”) loads (Tables 1, 2).

The running-in ability criteria Kr-i has been determined upon the results of oil tests on FBM at 10 s and 7200 s test duration and at loads hazardous for balls seizing and welding – 710, 2550 and 3550 N. Based on the Table 3 data, according to Kr-i value the oils with FC at all loads have an edge over commercial oils containing chemically active additives.

Table 3. Running-in ability criteria $K_{r\text{-}i}$ for tested oils with FC and for commercial oils

Oils	Axial load P, N		
	710	2550	3550
	$K_{r\text{-}i}$		
Oil 1J	5360	9260	5800
Oil 1G	5265	9005	5716
TAP-15V*	3286	4434	-
TAD-17i*	232	4950	-

* $K_{r\text{-}i}$ for these oils have not been identified for 3550 N axial load due to balls welding in the course of work in the range of 60-300 s.

The mentioned high running-in ability of oils with FC is confirmed by the results of their tests at SMC-2 machine.

Tests have been conducted according to the following pattern: “rotating roller – self aligning shoe”. Roller material is st. 45, while shoe material is SCh-20 (grey cast-iron); surface purity is Ra =

0.63-0.50 μ (∇ 8a) for roller and Ra = 1.6-1.25 μ (∇ 6b) for shoe.

Each oil has been tested for 6 h (with 2-hour cycles), at sliding velocity 0.785 m/s ($n = 300$ min^{-1}) at the following loads: I cycle – 2.5 MPa, II – 5.0 MPa, III – 8.0 MPa. Frictional torque and temperature of oil have been recorded each 30 minutes in the course of tests.

Experiments showed that during 6 hour running-in the surface roughness of shoe (made of SCh-20) has been decreased from 1.4 to 0.3 μ in case of oil «I-50A+5%FC», while it has been dropped from 1.4 to 0.9 μ when using multipurpose transmission oil Tad-17i, and from 1.4 to 0.4 μ for the known break-in oil OM-2.

According to the efficiency of friction force decrease the oil with FC doesn't lag behind oil OM-2 at any stage of friction couple loading. At the mentioned stages a contact temperature in case of oil with FC was lower as well than for oil OM-2.

At that, according to its anticorrosion properties the oil with FC surpasses oil OM-2 (containing chemically active additives). E.g. corrosion of plates of phosphor bronze OF 6.5-0.15 at the break-in oil OM-2 equals to 25-30 g/m^{2}h (when t=150°C, t=3 h), and in case of oil with FC, corrosion is not mentioned at all.

The comparative study of friction surfaces formed on the steel balls by the end of run-in regime (determined upon reaching of constant minimal value of friction coefficient), as well as after working under conditions of stationary friction regime was conducted on the specimens passed the tribological tests in the environment of oils modified with AFC nanoparticles (a) and Fe-atom cluster doped CNTS (b) (oils 1J and 1G) when sliding under the 150 MPa load.

In order to study the mechanisms of tribo-product composition formation on the steel ball surface during sliding friction in the environment of oils having different carbon-based nanoadditives, the series of experiments have been conducted the layer-by-layer analysis of the atomic distribution and phase ratios in the investigated friction surfaces/subsurfaces using the methods of analysis of the first derivative of carbon KLL Auger signals (with plasmons).

In Fig. 1, a and b, there is shown the series of carbon KLL differential Auger-electron spectral peaks, recorded in the 200÷350 eV energy range. These peaks are obtained from friction surfaces formed on steel balls during sliding in run-in regime in the environment of oils 1J (a) and 1G (b). In both cases the mentioned series 1-4 of Auger-spectral peaks were recorded from one and the same analytical area of friction surface, after removal of layers of uniform thickness through ionic bombardment.

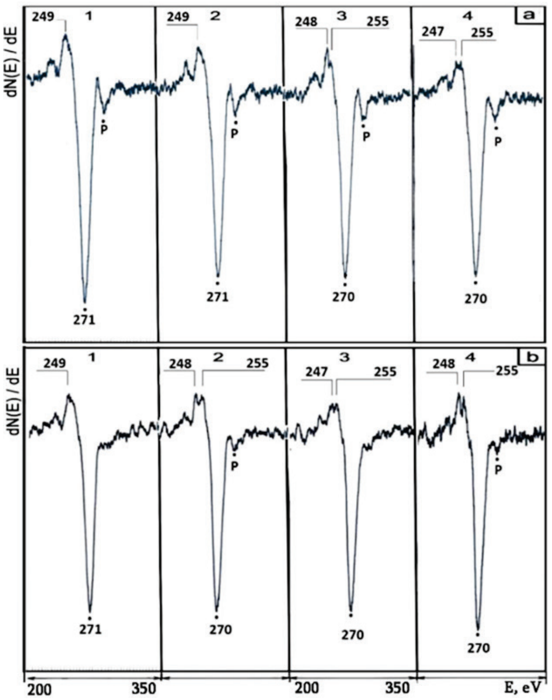

Fig. 1. Series of carbon KLL Differential Auger-electron spectral peak, recorded in the energy range of 200÷350eV, of the friction surfaces formed after sliding on the steel balls in area of oils, modified with: a – ultra dispersive amorphous carbon nanoparticles (oil 1J) and b – Fe-cluster doped CNT$_{s.}$ (oil 1G). Spectra (1-4) were recorded after sputtering of surface layer: 1 – 30 Å, 2 – 150 Å, 3 – 300 Å, 4 – 500 Å in thick by bombardment using argon ions with the 2keV energy.

Peaks #1 shown in Fig. 1a and b are recorded for each specimen exposed to running-in in oils 1J and 1G, respectively, after removal of ~30Å thick layer adsorbed from atmosphere on their surfaces, and they reflect compositional and phase state existing on the physical surfaces of the respective samples in the moment of friction cessation. It is seen that the spectra (#1) obtained from physical surfaces of both samples differ from both each other and spectrum obtained from the layer adsorbed from atmosphere. In particular, a plasmon peak P is revealed only in the spectrum obtained from the surface exposed to running-in in oil 1J. At that, main peaks of KLM Auger-transition of carbon atoms are shifted by 1÷2 eV towards smaller energies. At the same time, presence of the plasmon peak at 249 eV points at the transport of the additive – amorphous carbon nanoparticles (distinguished by a mixture of $sp^2 + sp$ states of hybridization) from the medium of oil 1J to friction surface.

Absence of plasmon peak P along with the presence of plasmon peak at 249 eV in the spectrum obtained from the surface exposed to running-in in oil 1G, points at the fact that in the given case there takes place transport of the additive – CNTS nanoparticles (distinguished by sp^2 state of hybriddization) from oil medium to the friction surface. In the respective spectra (Fig. 1a and b, curves 2, 3, 4) recorded from the depth of friction physical subsurfaces exposed to running-in in both oils, there is manifested the presence of graphite (distinguished by sp^2 state of hybridization and therefore, by intense plasmon peak at 248 eV, when a main peak is located at 271 eV) diamond and DLC-phases (which are characterized by a plasmon peak of the same low intensity at 247 eV and a plasmon peak of relatively high intensity at 255 eV, when main peak is disposed at 270 eV, that corresponds with carbon sp^3 or mixture $sp^3 + sp^2$ states of hybridization).

Thus, the comparative study of the fine structure of carbon KLL Auger-transition peaks (the shape, energy disposition of main and plasmon peaks and ratio of their intensities) showed that the thin surface layers formed on the steel balls in run-in regime during sliding in the environment of oils modified with different CNFS additives (AFC and Fe-cluster doped CNTS) have a complicated multiphase composition as a mixture of inclusions in the matrix of nanoparticles with different carbon allotropies (graphite, diamond, DLC), such as conversion of tribosynthesis products from initial CNFS additive and part of non-converted initial additive nanoparticles.

It is obvious that development of self-organization processes during running-in period above the surface layer in the form of so called intermediate “third phase” of tribological pair [10] should play a fundamental role in the establishment of friction and wear mechanisms when transferring a tribopair from run-in regime to stationary regime of sliding.

From this viewpoint, the conducting of SEM studies of friction surfaces of the same steel balls after long-term operation in the stationary regime of sliding in the environment of oils 1J and 1G was of some interest.

In Fig. 2a÷d there are presented the differently magnified SEM images with of steel ball friction surface formed during sliding in the stationary regime of structural dissipative adjustment in the environment of oils, modified with nanoparticles of amorphous finely dispersed carbon (a, b) and Fe-cluster doped CNTS (c, d). Row-by-row arrangement of open micro-cracks in the surface layer formed in the run-in regime of friction is manifested on the surface of steel balls exposed to running-in during sliding friction in the environment of both oils in the mentioned regime. At that, for the surface operated in the oil 1J there is observed proliferation of micro-cracks having characteristic fractures mode to deeper subsurface layers in comparison with micro-cracks on the friction surface operated in the oil 1G (compare Fig. 2a and c).

As is clearly seen on SEM-images with high magnification (Fig. 2b and d) microtopography and

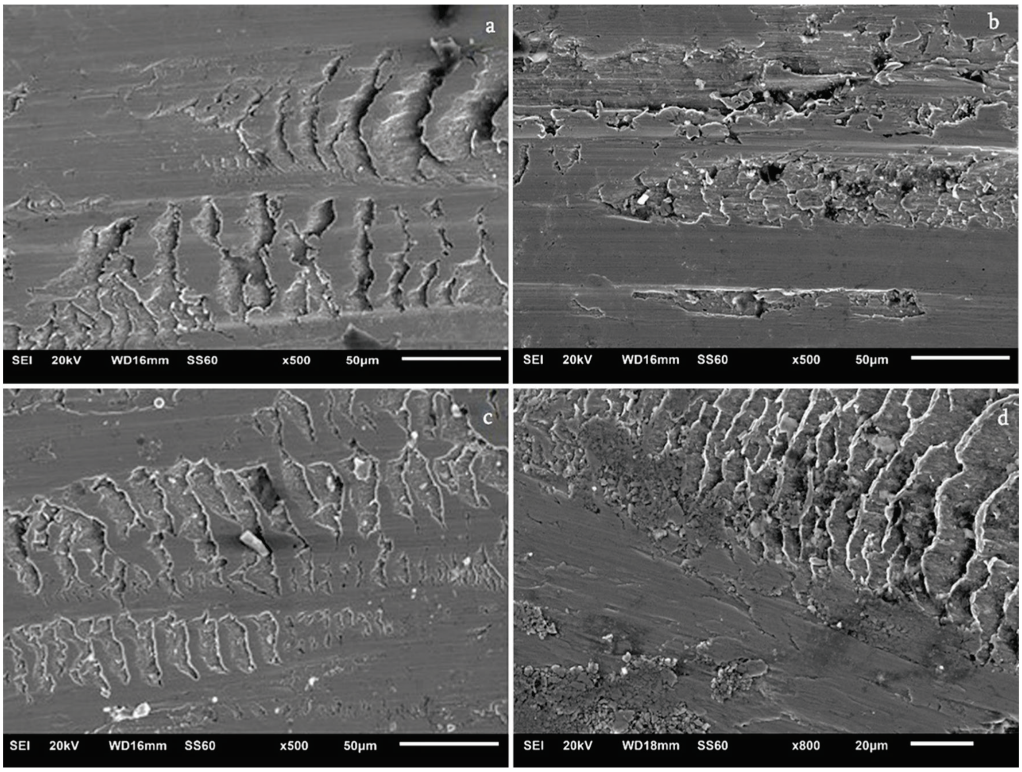

Fig. 2. SEM image of the steel ball surface, formed during friction at the stationary regime of sliding in area of oils, modified with nanoparticles of amorphous carbon – oil 1J (a, b) and Fe-cluster doped CNT_s – oil 1G (c, d).

structure of subsurface layer breakdown observed in the crack opening areas have sharply defined character of fatigue fractures with peculiar cold-hardening "waves" that are caused by cyclical pattern of elastic deformations during friction, transferred via the above-described thin surface friction layer to subsurface layers. Detected structural peculiarities of surface film and subsurface layers fractures make it possible to conclude that formation of wear products (debris) is initiated by fatigue failures due to substantial plastic and elastic deformations developed in the thin surface and subsurface layers, respectively, during long-term and repeated combined exposure of normal and tangential forces on sliding surfaces of friction pair. It is natural that gradual accumulation of fatigue effect and plasticity supply exhaustion in the microvolumes of the surface layer in the long run will lead to detachment of these microvolumes in the form of wear particles (debris).

Conclusions

The above-mentioned studies confirmed the high efficiency of steels friction surfaces running-in in the medium of oils containing nano-dispersed amorphous carbon or Fe-doped carbon nanotubes, that is based on the phenomenon of tribosynthesis of the secondary structures with mixture of tribosynthesis products from nanoparticles of AFC and Fe-cluster doped CNTs modifiers of allotropic modifications of carbon nanoforms: graphite, diamond-like carbon and diamond nanoparticles, on the friction surfaces working in the medium of lubricants of mentioned types, during sliding and rolling of balls. Such secondary surface structure is distinguished by the ability of substantial reduction of many types of wear and damages of friction surfaces. Respectively, it is possible to develop an innovative running-in nanotechnology of metal friction surfaces based on the mentioned phenomenon.

The revealed structural features of the fractures of the surface film and subsurface layers allow us to conclude that the formation of debris is initiated by fatigue damages due to significant plastic and elastic deformations developing, respectively, in the thin surface and subsurface layers under prolonged and repeated joint action of normal and tangential forces on the friction surface.

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მექანიკა

ნანო-საპოხი მასალების არეში მიმუშავების გავლენა ფოლადის ძლიერად დატვირთული ხახუნის ზედაპირების ცვეთის ხასიათზე

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** საავტომობილო ტრანსპორტისა და ლოგისტიკის დეპარტამენტი, საქართველოს ტექნიკური უნივერსიტეტი, თბილისი, საქართველო*
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ექსპერიმენტები ჩატარდა ოთხბურთულიან ხახუნის მანქანაზე, რომლის კვანძშიც რეალიზდება სრიალის და გორვის ხახუნი. შემზეთ მასალად გამოიყენებოდა ამორფული მაღალდისპერსიული ნახშირბადის ნანონაწილაკების (AFC) და Fe-კლასტერებით დოპირებული ნახშირბადის ნანომილაკების (CNTs) დანამატების შემცველი ორი ნანოზეთი. ექსპერიმენტის მეთოდიკა მოიცავს ცდებს, შერჩეულ ძირითად დატვირთვის რეჟიმზე, რომლებიც ჩატარდა როგორც ფოლადის ბურთულების ხახუნის ზედაპირების წინასწარი მიმუშავების გარეშე, ასევე მიმუშავების შემდეგ. მუშა ზედაპირები შესწავლილ იქნა, შესაბამისად, მუშაობის დაწყებამდე, მიმუშავების შემდეგ და ძირითადი დატვირთვის რეჟიმზე მუშაობის შემდეგ. ზედაპირები გამოკვლეულ იქნა მასკანირებელი ელექტრონული მიკროსკოპის (SEM) და ოჟე-ელექტრონული სპექტრომეტრიის (AES) გამოყენებით. ძირითადი დატვირთვის რეჟიმში მუშაობის ექსპერიმენტულმა შედეგებმა აჩვენა, რომ, მაგ., დაღლილობითი წყლულები (პიტინგი) გაცილებით გვიან ჩნდება იმ ბურთულებზე, რომლებმაც გაიარეს მიმუშავების პროცესი, ვიდრე ბურთულებზე წინასწარი მიმუშავების გარეშე. ნაჩვენებია, რომ ამ პირობებში მუშაობის დროს ხახუნის ზედაპირზე წარმოიქმნება თხელი მეორადი შრე ამორფული მაღალდისპერსიული ნახშირბადის ნანონაწილაკებისა (AFC) და Fe-კლასტერებით დოპირებული ნახშირბადის ნანომილაკებისაგან (CNTs) ტრიბოსინთეზის პროდუქტების ნარევით, რომლებიც შეიცავს ნახშირბადის ალოტროპული მოდიფიკაციის ნანოფორმებს: გრაფიტის, ალმასის

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