Tribology of carbon-containing materials at high temperatures

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Abstract. The work is devoted to high-temperature laboratorv tribological tests of carbon-containing materials and their modification. The studies have shown that at a temperature of 300-600 °C and a load of 1.0 MPa, the friction coefficient of the carbon-containing composition material samples processed in the environment Se-PTFE on steel 40X13 is 0.079-0.094 that 28-79% lower than the "Angolan-2D".

1. Introduction
The search for new materials and their correct choice for friction units is one of the effective ways to improve the reliability and durability of machines. Simulation of friction units operating at high temperatures is complicated by the creation of special equipment and methodology of the experiment. The lack of standardized test methods is explained by the complexity of the processes occurring during friction, the presence of a large number of factors affecting the friction process. The problem of choosing materials for friction units is complicated by the fact that in extreme conditions of testing the operation of friction units, it is necessary to create conditions close to real. With regard to aerospace engineering, the effectiveness of the strength properties of the material in terms of mass – specific strength [1] is one of the key requirements for structural materials. Among the most promising materials are carbon-containing composition materials (CCCM) [2]. CCCM have extremely low density. Good characteristics of strength and resistance to adhesion setting, high temperature resistance determine the interest in their application in space crafts (SC), especially to ensure the performance of friction units, for example, mechanisms of rotation of antennas, in conditions of long flights of spacecraft in outer space, where materials are required, which in addition to good anti-friction properties, must have good resistance to adhesion setting. At high temperatures, for example, when flying towards the Sun, also in an aggressive environment, for example on the planet Venus, high heat resistance is required [3]. CCCM are used in SC engines and SC design at motion in the atmosphere while returning to the Earth [4]. Carbon composites with carbon nanotubes are used to manufacture non-metallic electrical conductors in current collectors in aerospace engineering, military and other industries in order to increase simultaneously the strength and conductivity [5]. The introduction of nanotubes in CCCM shows an increase in tensile strength in comparison with a composite without carbon nanotubes. The influence of nano graphite particles on the microstructure, porosity, shear strength, damping ability of CCCM [6, 7] has been explored. The influence of fiber orientation on the tribotechnical properties of CCCM [8] has been determined. The efficiency of nitrogen doping of porous CCCM with nanotubes [9] has been studied. The protection of surfaces from ablation and oxidation [10, 11, 12] and application of protective coatings
[13, 14, 15] have been considered when CCCM working at high temperatures. The influence of pyrolytic carbon oxidation in the synthesis of CCCM has been studied [16]. The mechanical properties of CCCM were being studied during compression tests with an assessment of the modulus of elasticity and strength [17], shear [18], tension [19]. It is shown in [20, 21] that indentation tests are an effective and fast method for assessing the mechanical properties of CCCM, especially in loading and unloading cycles (kinetic indentation). With regard to the problems of the aerospace industry, “fatigued-cracked” behavior of all-carbon composite materials with different fiber orientation was investigated [22]. The internal friction of the CCCM has been explored. The thermoelastic mechanism of energy loss during deformation and static hysteresis mechanism [23] has been proposed. Simulation of CCCM friction under braking conditions during landing has shown the ability of CCCM to meet the requirements for aviation brakes [24].

Tribological properties of CCCM are little surveyed. It is established that measures to improve the anti-friction properties are necessary for large-scale application of CCCM in friction units.

The objective of the work is to investigate the performance of carbon-containing materials and the possibility of modifying the friction surface to reduce the friction coefficient of 40x13 steel at a specific load of 0.5 and 1.0 MPa and a temperature range (300...600ºC).

CCCM "Angolan-2D" and "Hardcor" have been selected as objects of the tests. To improve anti-friction properties of CCCM "Angolan-2D" friction surface was being treated in the environment:
- selenium and polytetrafluoroethylene (Se-PTFE). The process of processing samples from CCCM was being carried out in a protective chamber at temperature of 8200C.
- tin selenide and polytetrafluoroethylene (SnSe-PTFE). The process of processing samples from CCCM was being carried out in a protective chamber at temperature of 880ºC.

2. Equipment

The process of saturation of samples with chalcogen was being carried out on a laboratory installation with induction heating, the scheme of which is shown in figure 1.

![Figure 1. Installation diagram: 1-Crucible, 2-HDTV-inductor with power supply, 3 - Sample, 4-the Material is chalcogen, 5-cooling, 6-Vacuum gauge, 7-a temperature data logger, 8-Vacuum gauge VIT-2, 9-Vacuum pump, 10-valve, 11 - Liner cooling.](image)

At the bottom of the crucible (1) is placed powder-shaped selenium (4), which, when heated to 685 degrees, evaporates and saturates porous metal-ceramic samples (3), suspended in a stainless grid in the middle of the crucible (1). The samples have porosity of 10 ... 30%, which includes nickel or iron. Selenium vapors react with nickel or iron to form a compound with selenium.

Before heating in the crucible with samples the air was pumped out by vacuum pump (9), to 10-3 mmHg. The vacuum control was carried out by vacuum meter VIT-2. After pumping the shut-off valve (10) was closed. Water cooling (11) was supplied to the cooling jacket (5) of the crucible (1) and to the inductor with power supply (2). The heating of the crucible was carried out by a HC inductor (2), the temperature was controlled by a thermocouple of chromel-drops (7). Simultaneously with the increase in temperature, selenium began to evaporate and pressure began to rise, which was measured on a manometric vacuum gauge (6).
The tribological tests were being carried out at a high temperature stand WTMT-1000 [22], providing friction of samples by finger diagram in the temperature range 20 – 1000 °C in terms of the range of normal loads of 35 – 500 N. Taking into account thermal insulation, the heating unit allows to heat the samples to a temperature of 1000 °C.

The tests of samples of materials should be carried out in conditions simulating the work of the full-scale friction unit. Therefore, the most acceptable method of tribological testing of materials is the "disc-finger" scheme, since the results of standing tests of samples are easier to spread to other schemes of bearings sizes.

The test unit is mounted on a support platform heated by an electric spiral, with a capacity of 4 kW. Taking into account the thermal insulation, the heating unit allows the test samples to be heated to a temperature of 1000°C. The support platform is installed with the ability to turn on the thrust bearings in the form of a thrust rolling bearing, which is equipped with a cooling device running water. On the support platform, a lever is installed based on a force measuring device connected to a computer to record the results of measurements of the moment of friction of samples on steel.

The test unit of the stand consists of the upper clamping disc 2, which loads the test samples mounted on the heated lower disc 3, made of steel 40X13. The entire Assembly of the test samples is placed on the basis of 4. The stand is equipped with a device measuring the friction torque 5, signals from which are transmitted to the computer. The block diagram of the interconnection of the nodes of the high-temperature stand for testing samples according to the finger scheme is shown in figure 2.

Figure 2. The block diagram of interconnections between nodes of the high temperature test stand samples by finger pattern: a 1 – tested samples; 2 – the upper retaining plate; 3 – a lower heated disc; 4 - heated oven with a fixed thermocouple; 5 - device for measuring the friction torque; 6 – control thermocouple; 7 – thrust bearing; 8 – system of water cooling of the bearing; 9 – drive stand is combined with a mechanism of axial loading; 10 – speed control drive; 11 - the holder of the top ring; 12 – heating apparatus; 13 is a voltage regulator of the heating device.

The upper part of the test unit is attached to the drive of the stand, while it is separated from the lower heated part of the test unit. The support platform is heated to a predetermined temperature, while the thrust bearing on which it is mounted is continuously cooled by running water. When the set temperature of base 4 is reached, the drive of stand 9 with the axial loading mechanism is switched on.

The measurement system of the friction torque is switched on, the rotation drive of the upper part of the measuring unit is also switched on. It operates according to preset patterns; friction torque and the temperature in the maximum approach to the samples are being recorded.

The test stand is based on a heavy drilling machine (model 2A125), which houses the test unit and temperature control devices. The mechanism of loading the test samples by axial force and the drive to ensure a given relative rotational motion of the test samples are used from the drilling machine. Thyristor drive 10 is used for speed adjustment.

The axial load on the test samples is applied by creating a load moment on the movable vertical spindle of the machine. Calibration of the axial force on the seal is carried out according to the portable dynamometer DOSM-3-2U 5095 (GOST 9500-84), mounted on the base of the machine, against which the movable spindle rests. The sensitivity threshold of the dynamometer is not more than 0.02% of the largest measurement limit, the value of the smallest marking on the scale is not less than 0.1%. A vessel
is suspended on the lever of the loading device. The given axial load is selected by the filling of the vessel.

During the tests, the friction torque was being measured. To do this, a lever is mounted on the holder of the lower steel disk, resting against the load cell. When the upper sample holder rotates, the moment of resistance to rotation with the lever is transmitted to the load cell. Friction torque and temperature are recorded using strain gauges ZET 7111 Tensometer CAN. The data is transmitted digitally via CAN 2.0 using the Modbus protocol. The USB + - + CAN interface converter ZET 717 is designed to connect measuring networks based on smart sensors and ZETSENSOR control modules with CAN interface to a PC. The measurement accuracy is not less than 1% of the largest measurement limit. Additional calibration of the device was not carried out. The moment of rotation resistance was being recorded continuously and is stored in the device memory. Figure 3 shows the location of the test samples on the lower heated disc.

![Figure 3. The location of the samples on the lower disk after the test.](image)

The temperature of the lower steel sample (heated to a predetermined temperature) was being measured using a chromel-alumel thermocouple recorded on the device using temperature sensors ZET 7120 TermoTC-CAN. The control range when using a TXA sensor is 1200º C, a resolution is 1ºC, a limit of permissible basic measurement error of an input parameter (excluding a sensor error) is 0.5%. Duplication of the temperature readings was being carried out by the visual method according to the testimony of the single-channel universal digital device A565-001-02, working in conjunction with thermoelectric converters according to GOST 6616-86 when measuring temperatures up to 1300ºC. Accuracy class is 0.1 / 0.06, resolution is 0.1ºC.

The temperature measurement of the upper clamping disk (figure 2, item 2) is carried out by a digital temperature meter ATT-2000, 2-channel, with a K type thermocouple (NiCr-NiAl).

The temperature of the heating platform (figure 2, item 4) was being measured by a millivoltmeter for measuring temperature of type 4540. The limit of permissible basic error of the device is 1% of the normalizing value.

The samples were being tested under rotational motion with a constant angular velocity of 0.16 m/s. The installation drive is powered by an asynchronous electric motor. The setting of the given rotation speed is provided by changing the current frequency using the INNOVERT ISD mini frequency converter having an output frequency range of 0.1 - 400 Hz, and an accuracy of indicating the output frequency of 0.1 Hz.

The results of the experiment. Four types of samples have been tested: Argolon-2D, Hardkarb, and CCMC treated in Se-PTFE, SnSe-PTFE. The tests were carried out on samples of 10 × 10 × 8 mm in a pair of friction with steel 40X13 [4]. The contact area was 300 mm2, the average diameter of the sample arrangement was 66 mm, the linear velocity was 0.16 m/s, and the axial load was 0.5 and 1.0 MPa. During the tests, the temperature on the friction surface and the moment of friction were measured.
3. Results of the experiment

As a result of the tests, the dependence of the friction coefficient at a load of 0.5 MP (figure 5) and 1.0 MP (figure 6) was established in the temperature range from 300 °C to 600 °C for the tested samples of materials: "Argolon-2D", "Hardkarb" and UUKM treated in Se-PTFE, SnSe-PTFE.

The analysis of the test results was carried out relative to material "Argolon-2D". The treated friction surface of CCCM in Se-PTFE, SnSe-PTFE during friction on 40Kh13 steel at temperatures from 300°C to 600°C has a lower friction coefficient at a load of 0.5 and 1.0MPa. At a load of 1.0 MPa and a temperature of 300 ° C, the friction coefficient for the test samples is in the range of 0.079-0.122. At a temperature of 600 ° C and a load of 1.0 MP, the friction coefficient for Hardkarb samples treated in Se-PTFE, SnSe-PTFE samples is 22%, 79%, 49% lower than that of Argolon-2D, respectively.

![Figure 4](image1)

**Figure 4.** The dependence of friction coefficient on temperature at a load of 0.5 MPa materials: 1- "Angolan-2D", 2-Se-PTFE 3-SnSe-PTFE 4- "Hardcarb".

![Figure 5](image2)

**Figure 5.** The dependence of friction coefficient on temperature at a load of 1.0 MPa materials: 1- "Angolan-2D", 2-Se-PTFE 3-SnSe-PTFE 4- "Hardcarb".

At a load of 0.5 MP and a temperature of 300 ° C, the friction coefficient for the tested samples is in the range of 0.092-0.13. At a temperature of 600 ° C and a load of 0.5 MP, the friction coefficient for Hardkarb samples processed in Se-PTFE and SnSe-PTFE is 15%, 78%, 57% lower than Argolon-2D, respectively. At a temperature of 600 ° C, a load of 0.5 and 1.0 MP, the friction coefficient of samples treated in Se-PTFE is 0.11 and 0.094, respectively. In the temperature range from 300°C to 600°C, the modified friction surface of CCCM in the environment of Se-PTFE has prospects for application in spacecraft friction units.

4. Conclusions.

The studies have shown that at a temperature of 300-600 °C and a load of 1.0 MP, the friction coefficient of CCCM samples processed in Se-PTFE medium on steel 40X13 is 0.079-0.094, which is 28-79% lower than that of Argolon-2D.

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