Improvement of antifriction properties of tribotechnical cermets by treatment in selenium vapor

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Abstract. The issues of the high-temperature tribological tests of cermets during friction against steel 40X13 in the temperature range from +600°C to +700°C in atmospheric conditions are studied in the article. The choice of temperature and test atmosphere is due to the prospects of creating friction units for operation without lubrication in extreme operation conditions. Studies have shown that when processing cermet in selenium vapor, the friction coefficient in the temperature range of 600 ... 700°C is approximately 8-15% lower at a load of 1.0 MPa than the friction coefficient of untreated samples. It was found that the iron chalcogenide at a temperature of 500°C and above releases Se vapors, which are adsorbed at the tribological contact and create a lubricating layer.

1. Introduction
Interest in tribological research in the field of high temperatures is due to the degradation of the materials of the friction pair bringing the failure of friction units. Friction bearings for high temperatures are used in a variety of industries. For example, in roller conveyors and trolley wheels in burning kilns for ceramics and bricks, in metallurgical industries, etc. The tendency to an increase in temperatures in friction units is observed in aviation, rocket and space engineering. Tribological processes at high temperatures are complex; they proceed under conditions of a change in the structure of materials and the formation of surface oxide layers. The maximum permissible operating temperatures of friction units generally do not exceed 600 -700°C. During the friction of a tool steel die in the process of forming and quenching sheet material from high-strength boron-containing steel at elevated temperatures, structures of a composite layer are formed, including an improved hardened layer and a protective oxide layer on top coat [1]. The article [2] investigates the effect of temperature on the coefficient of friction and wear mechanisms during sliding in contact with uncoated tools under pressure in response to the drop stamping process. It has been found that at higher temperatures (>600°C) abrasive wear is found, while at lower temperatures (<600°C) adhesion wear predominates. High temperatures affect the tribological properties of braking devices [3], vibration and noise as a reaction to vibration and the behavior of the contact at the interface of friction surfaces. Temperature effects are included in the simulation model of friction in high-speed precision gears, for example, in ball screw assembly [4]. Various wear-resistant coatings are used to protect against wear of metals, for example, coatings made of composite materials with a Ti6Al4V-TiC metal matrix [5]. The tests were carried out with dry sliding in a temperature range of 25–575°C. Composite coatings showed high wear resistance at all temperatures. In [6], in order to improve the tribological properties at elevated temperatures, in particular, to reduce friction, CrCN...
coatings were prepared by the method of cathode-arc evaporation. Coatings with a thickness of 3–4 μm were applied on substrates made of hardened steel and heat-resistant alloy. The carbon content varied from 0 at.% (Fe, CrN) to 31 at.%. Coatings with a carbon content of 12–31 at.% showed almost identical tribological properties up to 700 °C. The ionic coating AlTiN destructs when heated [7] into mixed oxides Al2O3 and TiO2. The average coefficient of friction of the AlTiN coating at 700°C, 800°C and 900°C is 0.77, 0.65 and 0.57, respectively, which significantly improves the wear resistance of the coating. In [8], the effect of temperature on the tribological properties of the CrN coating on the surface of stainless steel in liquid sodium was studied. It was found that at a temperature of 250°C, the coefficient of friction is 0.2, and an increase in temperature to 550°C leads to an increase in the coefficient of friction. Under high temperature conditions, much attention is paid to self-lubricating materials. Composites containing BaCr2O4 demonstrate [9] an effective reduction in friction and wear in the temperature range 400–800°C. At 800°C, the NiCr - 20BaCr2O4 composite shows a friction coefficient of 0.27 and a wear rate of 4.5 × 10−6 mm3 / (Nm), which is approximately 2 times less than the unmodified NiCr alloy. In [10], the behavior of high-temperature wear of rapid steel with high boron content, modified by CuTi, is investigated. The experiment on wear showed that the wear resistance of rapid steel with a high boron content is much better under better oxidation and mechanical properties of the matrix. In friction pairs operating at high temperatures, cermets are widely used. Investigations aimed at improving the tribological properties of cermets are mainly aimed at the effect of the metal binding phase on the microstructure [11, 12], mechanical properties [13, 14] and the choice of the ceramic phase [15, 16].

Solid lubricating coatings (TSP) are of great interest for work at high temperatures. These include graphite, boron nitride, chalcogenides - compounds of the elements of group VI of the Mendeleev table, sulfur, selenium and tellurium with metals: molybdenum, tungsten, niobium, etc. In [17], a friction mechanism for cermets based on the adsorption of chalcogen vapors on the friction surface, which react with the metal and create a lubricating layer at the points of actual contact, is proposed, transferring the dry friction mode to the friction mode with boundary lubrication. To maintain the long-term performance of the formed solid lubricating coating, it is necessary to reduce the rate of their chalcogen vapors release [18]. However, the antifriction properties of cermets during friction at high temperatures have been little studied.

The aim of the work is to investigate the efficiency of cermet processing in chalcogen (selenium) vapor at a temperature of 700 ... 750°C.

2. Materials
As materials for the samples under study a cermet based on iron and the steel 40X13 as an abradant material was chosen. For the synthesis of selenium chalcogenide, samples of 10x10x10 mm were made from sintered cermets of the following composition: C-18 ... 20%; Si-3.5 ... 5%; P-0.4 ... 1.5%; Mn-0.4 ... 0.5%; Cu-0.5 ... 1.0%; Fe-74 ... 75%, the porosity of which was 10 ... 20%.

3. Equipment and technologies
Tribological tests were carried out on a high-temperature bench VTMT-1000, which provides a friction mode for samples according to a “pin-on-disk” scheme in the temperature range from +20 to +1000°C under conditions of a range of normal loads from 35 to 300 N [19]. The design of the bench provides thermal insulation of the sample test heating unit, which allows the test samples to be heated to a temperature of +1000 °C. Tests should provide conditions that simulate the operation of a full-scale friction unit. In this regard, the most acceptable method of tribological testing of materials is the “pin-on-disk” scheme, since the results of bench tests of samples can be extended to other friction bearing designs. The temperature of the steel disk, along which the test samples were moved (heated to a given temperature), was carried out with a chromel-alumel thermocouple. The samples were tested under conditions of rotational motion with a constant angular velocity. The unit's drive is powered by an asynchronous electric motor. Setting the desired rotation speed is ensured by changing the current frequency using a frequency converter with an output frequency range of 0.1 - 400 Hz. The tests were
carried out on specimens made of cermet. The friction process was carried out in tandem with 40X13 steel. The contact area was 300 mm², the average diameter of the specimen was 66 mm, the linear velocity was 0.16 m/s, the axial load was 0.12–1.0 MPa, and the temperature was +20...+700°C. During the tests, the temperature on the friction surface and the friction moment were measured continuously. The tests were carried out in atmospheric conditions. The mechanical properties of the surface layer were determined from the indentation diagram of a Vickers diamond pyramid using a CSM kinetic microhardness tester (microindentation system based on the compact platform CSM-instruments MHT - Z - AE - 000). The method for assessing the plasticity of the material was in accordance with the International Standard “Metallic Materials - Instrumented Indentation Test for Hardness and Materials Parameters” ISO / DIS 145.

Samples were processed in selenium vapor in a vacuum chamber heated by high-frequency currents. Powdered selenium is placed on the bottom of the crucible, which, when heated above 685°C, evaporates and saturates porous cermet samples suspended in a stainless mesh in the middle of the crucible over selenium. The samples have porosity, which includes nickel or iron. Selenium vapors react with nickel or iron to form selenides. Before heating in a crucible with samples, air was evacuated to 10⁻³ mm Hg by vacuum pump, vacuum control was carried out with a vacuum gauge. After evacuation, the shut-off valve leading to pumping out was closed. The crucible was heated by an HFC inductor; the temperature in the crucible was controlled by a chromel-droplet thermocouple. Simultaneously with the increase in temperature, selenium begins to evaporate and the pressure began to rise, which was measured on a manometric vacuum gauge. Upon reaching 700°C, the heating of the crucible went into the mode of maintaining the temperature for an hour, then the heating was turned off and the cooling process took place.

4. Results and discussion
After processing cermet samples in selenium vapor, the changes in the elemental composition were checked. Figure 1 shows changes in the structure of samples

![Figure 1. Material surface spectrogram. a) before processing in selenium vapor; b) after treatment in selenium vapor.](image)

Electron microscopy showed the presence of selenium in the material on the friction surface of the sample. The control of mechanical properties was carried out by the method of kinetic microindentation, which is practically the only method for assessing the mechanical characteristics of the surface layer of a material. Of interest are the results obtained at a load of 1 N. to analyze the performance of a friction pair made of cermet. The evaluation of the microhardness of the cermets from which the samples were made was carried out according to the average value of the indentation diameter of 5 measurements. The average Hv value is 378 MPa. The plasticity of materials largely determines their crack resistance, the higher the plasticity, the higher the crack resistance coefficient (K1C). In the course of the experiment, Wplast is determined - the uncorrectable loss of power during indentation, equal to the area of the hysteresis loop on the indentation diagram of the indenter, i.e. power absorbed in the "loading -
unloading” cycle and Wр - mechanical work of indentation, equal to the sum of elastic and plastic power. In this work, the coefficient of uncorrectable power loss during indentation (Kn) was determined, which in the literature is often called the coefficient of plasticity. The term uncorrectable power loss more accurately reflects the essence of the process, because the uncorrectable power loss also includes the loss of power for cracking. This coefficient is determined by the ratio \( K = \frac{W_{\text{plast}}}{W} \). Figure 2 shows a diagram of the Vickers microindentor indentation into the cermet material.

As a result of the experiment, the characteristics of the mechanical properties of cermet samples were obtained as follows: modulus of elasticity \( E = 157.5 \text{ MPa} \); Vickers hardness \( H_V = 378 \text{ MPa} \), maximum indentation depth \( H_{\text{max}} = 3.6 \mu\text{m} \), \( W_{\text{elast}} = 0.3 \mu\text{J} \); \( W_{\text{p}} = 1.07 \mu\text{J} \).

As a result of tribological tests, the dependence of the friction coefficient at a load of up to 1.0 MPa and a speed of 0.16 m / s of the samples was established. Figure 3 shows the dependence of the friction coefficient on the load at a temperature of + 600°C, and figure 4 shows the dependence of the friction coefficient on the load at a temperature of + 700°C.

Figure 2. Diagram of Vickers microindentor indentation into the friction surface of the sample.

Figure 3. Dependence of the coefficient of steel friction 40X13 at the load at a temperature of 600°C materials: 1-cermet, 2-cermet + Se.
As can be seen from figures 3 and 4, the friction coefficient of cermet impregnated in a selenium medium over the entire load range of 0.12-1.0 MPa for test temperatures of +600°C and +700°C is lower than the friction coefficient of conventional cermet. At temperatures of +600°C and +700°C, the iron chalcogenide decomposes, forming a chalcogen, which reacts with the metal and forms a film of solid lubricant on the friction surface.

5. Conclusions
Friction of solid lubricants is a special case of boundary friction, when a lubricating layer created from adsorbed molecules separates surfaces at the points of actual contact. Cermet can be antifriction at high temperatures if it contains a filler that, when heated, emits steam with a high saturation pressure. At a load of 1.0 MPa and a temperature of +600°C, the friction coefficient of a cermet impregnated in a selenium medium is 8% lower than that of a conventional cermet, and at a temperature of +700°C, the friction coefficient of a cermet impregnated in a selenium medium is 15% lower than for conventional cermet.

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