Modification of Graphite Greases Graphene Nanostructures


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Abstract—In the article considered the problem of the devastating impact of friction to the machine parts’ and mechanisms, and ways to solve this problem. Described the method of mechanical activation of the graphite grease in a planetary mill. Experimentally determined friction coefficients of sliding of standard and mechanically activated of the graphite grease. Established that as a result of mechanical activation friction coefficient of sliding decreases in the 2.2–2.5 times. The obtained spectra of Raman scattering allow to assume, that the reduction in the friction coefficient of sliding happens at the expense of forming in a graphite grease of graphene nanostructures.

Keywords: friction coefficient of sliding, planetary mill, mechanical activation, graphene nanostructures
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INTRODUCTION

Destructive mechanical friction is one of the main issues in the operation of machines and mechanisms. The most obvious solution to this problem is the use of technical lubricants. Most often, graphite and molybdenum disulphide are employed as such, but there are drawbacks. Graphite works as a lubricant only in humid environments, whereas molybdenum disulphide—only in dry environments. Apart from this, in order to withstand 1000 friction cycles, at least 1000 monatomic layers of each of the materials are required. In [1], it is shown that a two-dimensional single layer of carbon atoms—graphene—is capable of withstanding at least 6400 friction cycles between a steel ball and a plate, regardless of environmental conditions, while graphene laid in three or four layers can already withstand 47000 friction cycles. This is a significant increase in reliability with a general reduction in the lubricant cost. The main factor constraining the use of graphene is the lack of industrial technologies and, as a consequence, its high cost.

At present, graphite lubrication is widely used in practice in springs, torsion pendants of tracked machines, in open gears, for start-up and running-in of rubbing surfaces of internal combustion engines (pistons and cylinders), in various hydraulic installations, during rope exploitation, for greasing large size couplings operating under severe conditions, etc. The fact that colloidal graphite forms a film on friction surfaces, penetrates and is retained even in the smallest metal bumps and pores in the presence of oil as a result of adsorption is of particular interest.

It is known [2] that stationary non-equilibrium states with a high degree of organization can occur in open tribosystems. Friction units are completely covered by this definition, and thus it is possible to develop a friction unit without wear. Three mutually influencing processes take place during friction: interaction of the surfaces; changes in the surface properties as a result of the interaction and the effect of the environment; destruction of the surfaces due to the two previous processes.

The aim of the work is to develop a technology for modifying graphite lubricants with graphene nanostructures via mechanical activation in a high-speed planetary mill to increase the reliability and durability of machines and mechanisms, and to reduce energy costs for overcoming friction.

MATERIALS AND METHODS

Mechanical Activation of Graphite Lubricants

The possibility of obtaining graphene from graphite in a planetary mill has been confirmed experimentally [3–6], and thus the mechanical activation of the graphite lubricant was carried out on a laboratory planetary mill with independent rotational drives of the carrier (movable motion relative to the central axis) and grinding drums (relative motion) [7]. The diameter of the grinding drums was 120 mm, the rotation speed of the carrier ranged from 100 to 1100 rpm,
and the rotation speed of the grinding drums relative to their axes ranged from 10 to 400 rpm. The dimensions of the installation and the ranges of variation of the indicated rotation speeds made it possible to organize the motion of the grinding balls in the modes most effective for performing the mechanical activation, namely: periodic caving and circulation modes. It is under these modes that the zones, in which the grinding balls are shifted relative to each other, are maximally developed, and tangential stresses arise in the particles that hit between the balls.

The drums were loaded with grinding balls (50–100 g) and a graphite lubricant (5–25 g). Then, the drums were closed, the rotational drives were turned on, and the starting material was processed for a certain time (10–120 min). After the mechanical activation, the balls and the material were unloaded, the material was separated from the balls, and the structure and properties of the obtained product were determined.

**Determination of the Structure and Properties of the Mechanically Activated Lubricant**

In accordance with Russian State Standard (GOST) 3333-80 for mechanically activated lubrication, the following parameters were determined: dropping point temperature (based on GOST 6793–74); penetration at 25°C (based on GOST 5346–78); corrosion resistance of plates made of Grade 40 steel (based on GOST 1050–88); colloidal stability (based on GOST 7142–74); mass fraction of water (based on GOST 2477–65); shear strength (based on GOST 7143–73); and viscosity at 0°C (based on GOST 7163–84).

It was established that the mechanically activated lubricant completely complies with GOST 3333-80. The Raman spectra were then determined using a ThermoScientific DXR Raman microscope at an excitation laser wavelength of 532 nm.

Due to high molecular intensity of solidol and its large content in the lubricant (about 90%), it was impossible to determine the formation of graphene structures and detect the presence of graphite in the Raman spectra recorded for the graphite lubricant processed in the planetary mill. To exclude the influence of the solidol, the mechanically activated lubricant was dissolved in gasoline and centrifuged on an Opn-3U4.2 laboratory device for 10–15 min. The filtrate was drained from the tube, and the residue was diluted with alcohol and centrifuged. The resulting residue was diluted with distilled water and re-processed in the laboratory centrifuge. As a result of the centrifugation, the starting material was divided into three fractions. For further analyses, the lightest fraction was used, which represented a pasty substance of black color with pearlescent shine. After cleaning from the solidol, not very pronounced G, D1 and D2 peaks can be observed in the Raman spectra of the graphite lubricant. This indicates possible formation of graphene nanostructures.

To verify the formation of graphene structures during the mechanical activation of the graphite-containing compositions in the planetary mill, a second series of experiments was carried out. A mixture of GSM-2 natural crystalline graphite (ash content up to 0.5%) with a 5% triton aqueous solution was used as a starting material. The operating modes of the mill were the same as in the first series of experiments. In the Raman spectra of this mixture processed in the planetary mill for 1 h, the G, D1 and D2 peaks are more pronounced.

Since the results of the performed analyses of the mechanically activated graphite lubricant did not allow to make an unambiguous conclusion on the formation of graphene structures, it was decided to conduct studies on coefficients of sliding friction of the common graphite lubricant and the mechanically activated one.

**RESULTS AND DISCUSSION**

The studies were carried out on an UMT-01 universal friction machine (see the Fig. 1) developed on the basis of drilling machine 1 and designed for testing friction and wear of metallic and non-metallic materials under the conditions of using various lubricants, and also without oil. The test method was based on the mutual displacement of test samples 2 and 3 pressed against each other with a specified force. The rotation speed of the samples was smoothly regulated from 0 to 2500 rpm, and the clamping force of the test specimens—from 50 to 100 N. The sample size was as follows: the fixed circular plate with a diameter of 50 mm, and three rotating rollers with a diameter of 10 mm. The contact scheme was the end of the rotating roller and the planes of the fixed disk. The frictional moment and the axial load were recorded by strain gauges. As a result of the preliminary tests, it was established that the clamping force of the samples varies uncontrollably in the range of ±15%, and the error of the strain gauge, with which the circumferential force was fixed and the friction torque was calculated, was found to be ±10%. The similar results were obtained on friction machines of the other types [8]. To eliminate these drawbacks, some changes were made to the design of the machine. In particular, the device for securing fixed circular plate 3 through thrust bearing 4 was mounted on balance 5 with a measurement limit of 15000 g and an error of ±0.1 g, which allowed the clamping force of the friction couple elements to be accurately recorded.

The force $G$ generated by the frictional torque $M$ of thread 6 through block 7 was transferred to weight 8 mounted on balance 9 with a measurement accuracy