Factors affecting the tribotechnical properties of sintered materials

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Abstract. The article considers the issues of production of powder antifriction materials based on iron by methods of pressing and rolling powder mixtures. The analysis of influence of composition of materials, technological conditions of their production and other factors on the structure, mechanical and tribotechnical properties of powder sliding bearings was carried out. During the operation of the machine, its elements, subjected to various influences, change in state, size and properties. The effectiveness of developments in the field of tribotechnical materials is determined by the properties of these materials, as well as the speed and breadth of their industrial use. The effect of porosity of samples on their tribotechnical properties and hardness after sintering and additional heat treatment is studied. The results of tribotechnical tests of iron-copper-graphite sintered sulfided compositions, not alloyed and doped with phosphorus, as well as materials including additional lubricant components: graphite, molybdenum disulfide or calcium fluoride, are presented. On the basis of tests, it was found that in conditions of liquid friction, alloying with phosphorus has a decisive effect on increasing wear resistance, and without supplying lubricant to sintered samples with impregnated oil, wear is more than an order of magnitude higher compared to the conditions of liquid friction.

1. Introduction
To obtain plain bearings, porous powder antifriction materials are widely used, capable of operating in a wide range of loads and rotational speeds both under conditions of forced lubrication and, in some cases, without it. Powder materials have a number of advantages over traditional cast materials. The advantages of porous bearings such as pores in the powder material contribute to the formation of a stable oil film on the friction surface.

The lubricant accumulated in the pores after the bearing impregnation operation allows ensuring its operation in conditions of lack or in some cases forced lubrication. Powder bearings have relatively low cost and lack of deficiency of initial components. Powder bearings have high operational reliability and simple manufacturing technology.

Many scientists and developers took part in the research of this direction. The merit of the revival of powder metallurgy and the transformation into a special technological method of pressing and sintering platinum powder belongs to Russian scientists P.G. Sobolevsky and V.V. Lyubarsky who worked in 1827. The pressing and sintering of platinum powder takes place by dissolving in tsarist vodka, depositing ammonium chloroplatinate, calcining to obtain platinum powders, their subsequent pressing, sintering or hot pressing [1].
2. Materials and methods

We conducted a study on a friction machine MI-1M. Seven sintered compositions and cast bronze material were compared. Various enterprises and workshops of the automotive industry took part in the manufacture of samples.

The following materials were used to make parts. Iron powder PZHV 3-4-160-460-34 is marked as "Zh". Graphite powder GC -2 or GC -3 is marked as "Gr". Copper powder of the grade PM -1 is marked as "D". Sulfur powder is marked as "K". Molybdenum disulfide powder is labeled "Ms". The milled ferrophosphore domain is marked as "P". Ground melting spar with calcium fluoride content of not less than 97% in the final product is marked as "Fk". Tin powder is marked as brands PO-1, PO-E, PO-2. Numbers after letters indicate the mass fraction of elements in percent. Additional treatment after sintering is paroxidation (PO).

3. Study of the tribotechnical characteristics of materials

The performance of antifriction sintered materials is positively affected by such technological operations as sulfidation and oxidation, which are widely used in workshops and in powder metallurgy. Roller samples were tested according to the established mode when friction in oil. The test results are shown in Table 1.

Table 1. Properties of typical material grades depending on the version of their manufacturing technology

| Material Tag | Properties | Additional Processing Operation | Friction in oil p = 25 kgf/cm², 0.8 m/s |
|--------------|------------|---------------------------------|----------------------------------------|
|              | Hardness (HV) | Porosity, P, % | Tear wear, μm/h | Relative wear, μm/h | Coefficient of friction | |
| ZhGr0.3D3K0.3 | 95.16 | 16.0 | - | 1.380 | 2.59 | 0.056 |
| ZhGr0.3D3K0.3P0.2 | 64.8 | 19.7 | - | 0.532 | 1.00 | 0.080 |
| ZhGr0.5D3 | 125.5 | 15.9 | - | Samples did not pass the test (teases) |
| ZhGr0.5D3 | 131.0 | 16.7 | Sulfided impregnation with sulfur, roasting T = 600 °C | 0.612 | 1.16 | 0.050 |
| ZhGr0.5D3 | 82.0 | 15.9 | Sulfided sulfur impregnation, roasting T = 550 °C | 0.800 | 1.29 | 0.050 |
| ZhGr0.3 | 123.0 | 17.0 | Steam oxidation T = 550 °C | 4.20 | 7.89 | 0.090 |
| Cast bronze Br0F1 | 66.0 | - | - | 3.2 | 6.01 | 0.020 |

In the accepted mode of friction tests, the least wear belongs to sintered material of the ZhGr0.3D3K0.3P0.2 grade, obtained using the technology of one of the automobile plants; its relative wear is taken as a unit.

Compared to a material that does not contain phosphorus, their resistance is more than double. Some authors also point to an increase in wear resistance of iron-based sintered materials containing phosphorus.

Sintered materials that do not contain antitussive components (sulfur, phosphorus) show unsatisfactory tribotechnical properties.

Oxidation (for example, superheated steam treatment) markedly restores the antifriction properties of the sintered materials, but not as significantly as sulfidation carried out by impregnation with sulfur followed by annealing or by addition of sulfur to the original powder mixture.

Cast bronze OF-1 has the lowest coefficient of friction, and in terms of wear resistance, it even exceeds, for example, a sintered material such as oxidized ZhGr0.3D3-PO [2].
Serviceability at the given load of the bearing is strongly dependent on external friction conditions in the sense of supply of lubricant to the friction zone into the friction, liquid, boundary and dry. The degree of this constraint is different for each material tag. Table 2 shows examples from the results of the tribotechnical tests of iron-copper-graphite sintered sulfided compositions not alloyed and doped with phosphorus. There are materials including additional lubricant components: graphite, molybdenum disulfide or calcium fluoride.

From the table it follows that:
- in liquid friction conditions, phosphorus doping has a decisive effect on increasing wear resistance. The presence of solid lubricants in the composition of the material in this friction mode has no significant effect on improving operability;
- under conditions of boundary friction, i.e. without supply of lubricant to sintered samples, they were only impregnated with oil. Wear compared to the conditions of liquid friction is more than an order of magnitude, but samples containing solid calcium fluoride in their composition have less wear. It is sufficient to have only 1% to surpass an alternative material containing 1.5% graphite and 4% molybdenum disulfide in the charge;

Table 2. Tribotechnical characteristics of sintered samples depending on lubrication conditions

| Tag designation | Wear, W μm/\(\text{hour}\) | Friction coefficient |
|-----------------|--------------------------|---------------------|
|                 | In an oil bath           |                     |
| ZhGr0.2D3K0.3   | 1.4                      | 0.08                |
| ZhGr0.2D3K0.3R0.2 | 0.46                | 0.08                |
| ZhGr0.2D3K0.3R0.2Ms3 | 0.61            | 0.07                |
| ZhGr0.2D3K0.3R0.2Fk4 | 0.44          | 0.07                |
|                 | Self-lubricating mode of oil impregnated sample (boundary friction) |                     |
| ZhGr1.5D3K0.3Ms4 | 10.31-13.75           | 0.10                |
| ZhGr0.2D3K0.3R0.2Fk1 | 8.7-9.1           | 0.05-0.06           |
|                 | Dry friction of samples (without oil impregnation) |                     |
| ZhGr0.2D3K0.3R0.2Fk1 | 51                 | 0.33                |
| ZhGr0.2D3K0.3R0.2 | 76                  | 0.25                |
| ZhGr0.3DK0.3R0.2Ms3 | 74                  | 0.30                |
| ZhGr0.3DK0.3R0.2Fk4 | 41                  | 0.24                |
|                 | After nitrocementation and hardening wear, W = 34 μm/\(\text{hour}\) |                     |

- in the absolutely dry friction mode, wear increases even more (by one or even two orders of magnitude) compared to boundary friction. Materials that do not contain dry lubricant have the greatest wear. The best values for materials that contained calcium fluoride in an amount of 1–4%. Molybdenum disulfide is less effective [3].

Thus, calcium fluoride in dry friction conditions provides high performance of bearings. Noting the role of calcium fluoride as an effective lubricant, certain limitations on its content by other evaluation factors should be taken into account. Although increasing its content to 4% still allows slightly increasing the wear resistance of the bearing under dry friction conditions, the mechanical strength of the sintered composition is noticeably reduced due to weakening of the metal frame. Table 3 shows this [4].

Thus, it can be seen from Table 3 that the optimum content of calcium fluoride in the material should not exceed 1%.

Based on the studies conducted, the sintered material of the brand ZhGr0.3D3K0.3R0.2Fk1 is widely used in the manufacture of sintered parts operating under dry friction. One of these parts was the packing ring of the rear axle of the truck [5].

Among the factors, affecting the performance of the sintered bearing article, there is the nature of the microstructure.
It is well known that the medium of microstructures of iron-based materials not subjected to thermal hardening is preferred in terms of wear resistance to pearlite microstructure. However, there are certain technological difficulties in machining materials with a lamellar pearlite or pearlite structure with excessive cementite.

In order to improve workability, annealing on the microstructure of the granular pearlite is usually used, which allows a sharp improvement in cutting conditions.

The thermal annealing of sintered articles has become traditional. But as studies have shown, annealing on the microstructure of granular perlite reduces mechanical properties. At the same time, the doping of low carbon compositions with phosphorus provides a wear resistance superior to that of materials with a granular perlite structure [1, 6].

This is evidenced by the results (Table 4) of the comparison of materials of the grades ZHGr1.7DZK0, 3P0.2 (annealing at 710-740 °C within 6 hours) and ZHGr0.3DK0.3P0.2 (not subjected to annealing). It is interesting to note that both brands contain 0.2% phosphorus. The effectiveness of phosphorus as an alloying element is found only in low-carbon material due to the poor solubility of phosphorus at a high carbon content in the iron.

### Table 3. Effect of calcium fluoride additives on the mechanical characteristics of the sintered material ZhGr0.3D3K0.3R0.2

| Type of tests | Units of measure | Content of calcium fluoride in % (wt.) |
|---------------|-----------------|--------------------------------------|
| Tensile strength | kgf/mm² | 25.9 | 25.3 | 23.2 | 22.7 | 20.9 | 18.6 |
| Specific elongation | % | 2.93 | 4.1 | 3.10 | 3.10 | 2.70 | 2.60 |
| Hardness (HV) | kgf/mm² | 90.4 | 94.4 | 89.4 | 87.6 | 89.9 | 92.4 |

### Table 4. Comparative values of low-carbon and high-carbon (annealed) antifriction materials

| Grade, type of microstructure, type of heat treatment | Carbon, C % | Physical and mechanical parameters | Tribotechnical properties |
|------------------------------------------------------|-------------|-----------------------------------|--------------------------|
|                                                      | Porosity P, % | (HV), hardness | Ultimate strength kgf/mm² | Specific elongation n, % | Wear W, μm/hour | Friction coefficient |
| ZhGr1.7D3K0.3R0.2 annealing on granular pearlite structure within 6 hours | 0.9 | 22.8 | 65.9 | 19.0 | 3.2 | 0.494 | 0.05 |
| ZhGr1.3D3K0.3R0.2 | 0.10 | 21.6 | 63.6 | 22.5 | 3.5 | 0.410 | 0.08 |

Thus, the use of phosphorus-doped sintered material instead of the use of pearlite annealing material requiring additional annealing after sintering, from the point of view of production technology, is economically advantageous [8]. In addition, the wear resistance of the phosphorus-doped ferrite sintered material is noticeably higher not only according to the results of tests on a standard friction machine, but also according to the mileage tests of real products - thrust washers of car pivots, as shown in Table 5.

The operational wear resistance of the second low-carbon material is 1.3-1.4 times is higher than that of the first granular pearlite annealed material of the microstructure, which is used for sintered automotive parts.

The use of the low-carbon material with phosphorus for parts such as guide bushings of engine valves is most promising [9].

In fact, the maximum operating mode on the surface of the valve sleeves is full of the exhaust, the temperature reaches 500-550°C. The ordinary motor lubrication loses its necessary properties. In
addition to sulphides, calcium fluoride is introduced into the material of the sintered bushings as an effective solid lubricant [10]. Standard bushings with a microstructure of granular pearlite did not contain such lubricant. The results of tests of sleeves of two materials ZhGr1.5D3K0.3 (standard with annealing) and ZhGr0.3D3K0.3R0.2Fk1 (1% with fluoric calcium) are given in table 6.

Table 5. Test results of "washer" with different microstructures

| Sintered grade | material | Description of microstructure | Porosity of part, % | Car mileage in thousands of km | Mean values | Confidence limit of 90% |
|---------------|----------|--------------------------------|---------------------|-------------------------------|-------------|------------------------|
| ZhGr1.7D3K0.3R0.2 | After additional annealing: grainy pearlite sulphide and phosphide eutectic grains along grain boundaries | 15 | 186 | 105 |
| ZhGr1.3D3K0.3R0.2 | Solid solution of copper and phosphorus in iron sulfides and rare grains of cementite | 15 | 239 | 145 |

Table 6. Comparative wear of guide bushings of the motor engine exhaust valve (bench tests)

| Materials | Test mode | Description of microstructure | Average maximum wear of bushings for 100 hours. |
|-----------|-----------|--------------------------------|-----------------------------------------------|
| Standard for Part ZhGr1.5D3K0.3 | 60 hours run-in, 100 hours run-in - part manufacturing quality test | Granular pearlite, sulfides | 19.85 |
| Advanced for Part ZhGr0.3D3K0.3R0.2Fk1 | 40 hours of wear test (total 200 hours with a maximum power of 2800 rpm) | Solid solution of copper and phosphorus in iron, individual grains of sulfides, cementite and calcium fluoride | 16.95 |

4. Conclusion
The parts that were tested on two engines of medium-duty trucks were explored.

According to the average data, the wear of standard exhaust valve bushings is 17% more than that with a ferrite microstructure.

It has been confirmed that the prospect of using the material ZhGr0.3D3K0.3R0.2 with a calcium fluoride additive is more economical and it is wear resistant.

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