On question of special mechanical properties of composites such as carbographite - antimony alloys

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Abstract. The article describes the most important characteristics determining the working capacity of plain bearings, which are the compressive and bending strengths, modulus of elasticity, crack resistance, wear resistance, coefficient of friction, permissible specific load. The temperature dependence of the mechanical characteristics has a particularly important consideration. The details of those mechanical characteristics for this class of materials are not available. The investigations of the noted properties of carbon materials impregnated with antimony based alloys were carried out in the present paper.

1. Introduction
The impregnation method is widely used in metallurgy, mechanical engineering and allied industries for producing composite materials (hereinafter - CM). At that, the main goal of impregnation is to fill the pore of solid skeletons.

A carbographite frame provides the frictional properties of the impregnated carbon skeleton and the formation of a secondary metal skeleton. At the same time, it has an effect on the resulting physical and mechanical properties of the finished material. In this connection, there are requirements that are applied to matrix alloy: provision of the shrinkage to near zero, high penetrating ability with respect to the frame.

Impregnation enables one to achieve a combination of substances with different properties in one material, thereby to impart the desired characteristics to CM. Furthermore, the use of impregnation opens a good scope of work aimed at experiments in order to obtain new CM.

Anti-friction carbon-based materials are widely used in the friction unit of various machines and mechanisms. To increase the density, strength, impermeability for liquid and gaseous media, wear resistance and improving thermophysical properties, the carbon materials are impregnated by various metals and alloys. Increasing the limit temperature of exploitation can be achieved by using the antimony alloys as the impregnate. Antimony is little inclined to adhesion in friction against steel, which determines the possibility of using it under high loads and at slip velocities [1, 2, 3].

The aim of this work is to develop impregnating alloys based on antimony for impregnating carbographite skeletons, which have special properties and work in difficult conditions of exploitation at ambient temperatures up to 500°C.

2. Materials and routine of experiment
The composition of the investigated alloys is given in Table 1.

Antifriction material under investigation is manufactured using the calcined carbon skeleton mark CG-365 produced by "Elektrokarbon" plant - city of Topolchany (Slovakia) having the following...
characteristics: density - 1630 kg/m³, an open porosity - (10 - 15%), Brinell hardness - 90 kg/mm², compressive strength - 115 MPa, elasticity modulus – 140 000 MPa. Samples in the form of cylinders with a diameter of 30 mm and a height of 60 mm were impregnated on a laboratory autoclave by alloys of three compositions based on antimony in Institute of Materials and Machine Mechanics Slovak Academy of Sciences. The parameters were: temperature - 650°C, pressure -5 MPa, a pressure-transmitting medium - argon of technical purity, vacuum pressure – 1,3 Pa, the autoclave volume - 4000 cm³, high-frequency equipment - 500 kHz.

Table 1. The composition of the investigated alloys.

| № alloy | Chemical composition                  |
|---------|--------------------------------------|
| 3       | Sb+30%Sn                             |
| 3.1     | Sb+20%Sn+1%Mischmetal                |
| 3.1.1   | Sb+20%Sn+2%Ni                       |

The technological sequence of impregnation is as follows: a) consolidation of the sample in a traveling holder; b) closing of the working space of the autoclave and creating vacuum; c) melting and heating of impregnating alloy to the desired temperature; d) immersing the sample into the melt and the pressure increase in the working volume of the autoclave; d) removal and cooling the sample in inert atmosphere overpressure.

The authors studied total and open porosity, the average pore radius and microscopic examination of fracture surfaces. There are mechanical properties, the density and crystallographic lattice was determined.

Experiments to determine the open porosity, the average pore radius were determined by mercury intrusion porosimetry at the installation "Micromeritics Antopors 9200". The density (volume weight) is determined by the bottle method (in kerosene). The total porosity, %, was found through a relative density. The crystal lattice type was determined by X-ray analysis on the "Dron-2" in the copper - Ka radiation.

Mechanical tests of samples under compression with the samples size 10x10x10 mm were carried out at a temperature of 20 ± 1°C. Tests were conducted in the condition of three-point bending with a base of testing 40 mm, the size of the samples 5x5x55 mm at a temperature of 20 ± 1°C, 300 ± 1°C, ± 380 ± 1°C, 415 ± 1°C, 450± 1 °C and 500 ± 1°C. The samples were loaded on the machine "Instron - 1115" equipped with vacuum furnace Ta1C28 with a deformation rate of 8x10⁶ m/sec (1,67×10⁻¹ m/sec). Tests to determine the crack resistance and the specific work of fracture were performed on Mesniedzer type samples (size 10x10x55 mm), the damage base is 40 mm, the deformation rate is 1,67x10⁻¹ m/sec. Lateral incision was performed by a diamond tool with cutting edge thickness of 0.2 mm. According to [4], the values of crack resistance Jc (K₁c) of carbographite, measured on samples with such radius of curvature like this, are correct. The length, l, of the original incision in the samples varied in the range of l/h = 0,1-0,8 in order to assess the invariance of determined values of Jc, (h - the height of the sample). Tests of the notched specimen were performed by three-point bending with a ratio of distance L between the supports of the charging device to the height of sample h, equal to four, corresponding guidelines [5].

Calculation of the value of the critical coefficient of stress intensity (K₁c) was conducted according to the relation [6]:

$$K_{1c} = \frac{3PL}{2th^2} \sqrt{I}$$

where P - the maximum load on the diagram - the deflection of the sample, MPa;
Y - coefficient of the shape of the sample, the value is given in [7];
t - sample thickness, m.

Limit of crack resistance Jc fracture toughness was determined according to GOST 25.506-85, according to the formula:

$$J_c = \frac{6P \sqrt{I}}{th} * Y$$
where \( l \) - the crack length, m;

\[ Y \] - coefficient of the shape; the value of this function is given in GOST 25.506-85, remaining quantities in equation (1).

Besides of the critical value of the coefficient of stress intensity (\( KIC \)), the authors determined specific work of fracture (\( \gamma_F \)), equal to the ratio of the total work \( A \) of failure of the notched specimen to double area \( S \) of the weakened section of the sample, through which passed the destruction:

\[ \gamma_F = \frac{A}{2S} \] (3)

To determine (\( \gamma_F \)), the technique was used [8].

Tribological characteristics of the studied materials. Friction and wearout of antifriction materials are the main criterion for evaluating the performance of these materials. In this regard, the influence of the main factors that determine the operational behavior of the carbographite materials impregnated by antimony alloys, namely, specific loads and material of the counterface was studied.

Materials were tested on a laboratory stand of rotational motion of MI-type with a scheme "shaft-liner" in air at a sliding speed of 0.5 m/sec by counterface, which is made of steel 4X13 (hardness HRC 48) and iron SCH20 (HRC 21) with a purity of processing of surface 0.16. The material was tested in a mode of stationary wear for 18 hours, pre-burnished the samples 10x10x10 mm to form a shiny film on the surface thereof. The temperature was monitored using a thermoelectric thermometer; a junction of the thermometer is placed into the sample at a distance of 1 mm from the friction surface.

The friction coefficient is given by:

\[ f = \frac{M}{rp} \] (4)

where \( M \) - friction torque, kg/m;
\( r \) - radius of the journal, m;
\( P \) - load on the sample, kg

Mass wearout of samples is determined after every 6 hours of tests by mass loss by weighing samples before and after testing on an analytical balance VLR-200 with an accuracy of up to 0.15 mg. Linear wearout is calculated by the following formula:

\[ \Delta h = \frac{(m_0 - m)}{\rho S} \] (5)

where \( m_0, m \) - mass of the sample before and after testing, kg;
\( \rho \) - density of material, kg/m\(^3\);
\( S \) - area of the friction surface of the sample, m\(^2\)

As a criterion for evaluation of wear resistance, the authors adopted intensity of linear wear:

\[ J = \frac{\Delta h}{L} \] (6)

where \( L \) - the path of friction, m.

The commonly used materials are carbon for impregnating by metals, the impregnation pressure actually is \( P_{exm} = 5-80 \) MPa, and the filling of pores is achieved with a radius of curvature \( r_{ef} > 0.1 \) microns.

Washbourne equation is the fundamental equation of mercury porosimetry [9]:

\[ P_{exm} = \frac{2\sigma_m \cos \theta_m}{r_{ef}} \] (7)

where \( P_{exm} \) - external pressure, Pa;
\( \sigma_m \) - surface tension, N / m;
\( \theta_m \) - contact angle, deg.;
\( r_{ef} \) - effective pore radius m.

The equation gives the dependence on volume of pressed liquid metal which is non-wetting (\( \theta > 90 \) °), on the technological parameters, pressing pressure of the metal \( P_{exm} \) and temperature \( T \).

Pores with a \( r_{ef} \leq r_{ef, min} \) can act as nuclei of brittle failure of material. Therefore, as a basic raw material, coke is most suitable, which does not contain a large number of micropores and mesopores, larger pores are undesirable from the standpoint of strength, larger pores are undesirable from the standpoint of strength, because in the carbon matrix there are closed pores, which serve as nuclei for further fracture. Reducing the volume fraction of large pores can be achieved by increasing the
compression pressure. Particles with a high specific surface area increases the strength of the carbon skeleton. Soot has this property among the carbon materials.

3. Results of experiments and discussion

Table 2 shows the data of carried investigations of the mechanical properties of the CM, at a degree of filling of the open porosity of the original carbographite frames is not less than 50% of theoretical. A significant increase of the strength of the impregnated material under compression is 1.5-1.7 times, and under bending is 1.4-1.6 times more, observed from a comparison of the data shown in Table 2 with the properties of initial materials. This is due mainly by an increase in the bearing section in filling pores by metal, as well as a corresponding reduction of stresses on defects such as pores. The increase of mechanical characteristics of CM can be associated with an increase in the bond strength of the impregnating alloy and a skeleton of carbographite, and also the small cross section of the metal fibers, which formed in a volume of micropore carcass. The relatively low values of crack resistance are indicated by high sensitivity of the mechanical properties of carbographite materials to crack-like defects.

| Measured parameter                  | Carbographite | Impregnating antimony alloys | Composite material |
|-------------------------------------|---------------|------------------------------|--------------------|
| Limit of compressive strength (δc), MPa | 115           | 183.4                        | 182 192.3 160      |
| Compressive modulus of elasticity (Ec) | 1.1·10⁴   | 1.2·10⁴                      | 1.2·10⁴ 1.3·10⁴   |
| Limit of bending strength (δb), MPa | 40            | 60.7                         | 60.3 67.5 50      |
| Crack resistance Jc(Kic), MPa*m¹/²  | 1.23          | 0.9                          | 0.84 1.42 1.7     |
| Specific work of fracture γf, J/m²  | 132.4         | 112                          | 136 141 138      |

However, there is a significant increase of 1.5-2 times in crack resistance and the specific work of fracture, which seems to be due to the plasticity of the metallic phase of KM. Studies of the microstructure showed that the alloy fills the open pores of carbographite skeleton uniformly.

The intensity and fullness of a process of liquid-phase impregnation of carbographite largely depends on the nature of their porous structure, including pore size [10]. Figure 1 shows the filling of carbographite pores after impregnation by antimony alloys.

Analysis of the impregnability of carbographite with different porous structures by liquid melt showed that the main factor for impregnation is not "bottle" pores detectable by mercury intrusion porosimetry, but the characteristic pores, which are defined by metallographic analysis. Metallographic examination of the porous structure of the composite was carried out by direct measurement of the pore nominal diameter in microstructure photos [10].
Figure 1. The filling of carbographite pores after impregnation by antimony alloys.

Figures 2-4 show the temperature dependence of the bending strength, fracture toughness, specific work of destruction, respectively.

Figure 2. Temperature dependence of the bending strength.

Figure 3. Temperature dependence of fracture toughness.
The studies have shown that crack resistance did not decrease at a temperature to about 300 ºC, and in some cases, there was a slight increase of crack resistance and compressive fracture. This is apparently due to a change of plasticity of the metallic phase of KM.

At a temperature of 20 ºC and 300 ºC, the best mechanical properties have a material impregnated with an antimony alloy 3.1.1 [11, 12].

At the temperature of 500 ºC, this difference disappears and the properties of the CM are determined only by the properties of the carbographite frame itself. Composition of the impregnating alloy has a significant impact on the crack resistance of CM. Materials which have been impregnated by alloy 3.1.1 possess the crack resistance approximately 2 times greater than the materials impregnated by alloy 3 [13] and 3.1. [14].

To explain the results of the research, the authors carried out the investigation by differential thermal analysis (DTA) at a constant rate of heating in the air using a derivatograph C. DTA results are consistent with the data obtained.

Comparison of the strength characteristics of the developed materials (Table 2) with the appropriate characteristics of carbographite materials impregnated by antimony and its alloys, which had been produced by companies in Germany and United Kingdom, shows that the developed material is not inferior to the properties of materials: the EK-350 (Ringsdorf Werke, Germany), T-15E (Rheinische Kolebirshteyn factory Franz Wenzel, Germany), GE-45A (Schunk Hebe, Germany). And it is close to the characteristics of the materials: T-12E, T-50E (Rekota Wenzel, Germany) and Grafitor 107 (United Stites Graphite Co, USA). However, the strength characteristics of the material are inferior to those of baked carbon materials impregnated with lead-based alloys SO₅ and Babbitt B83 (Russia) [14].

4. Conclusions
Studies showed that the composite material, containing alloy 3.1.1, did not detect mass changes when heated to a temperature of 500 ºC, and the maximum permissible load in friction on steel and cast iron is 4.5-5.5 MPa. Developed alloys on the base of antimony 3.1.1 and 3.1 are versatile and well suited for other methods of impregnation, such as compression molding and autoclave CM production.
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