Features of structure and physical and mechanical properties of metal-ceramic composite material depending on the content of boron nitride

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Abstract. In this paper, the problem of structure formation in powder compacts of PCh13M2-BN obtained by solid-phase sintering is considered. A phenomenological model of the formation of regions with increased concentration of ceramic BN particles in the volume of compacts is developed. The influence of the concentration of boron nitride in the initial charge on physical and mechanical characteristics of compacts is determined.

1. Introduction

Powder metallurgy technologies are actively used in development of self-lubricating composite materials, which are based on interaction of filler elements belonging to the category of solid lubricants, such as graphite, MoS$_2$, BaF$_2$, CaF$_2$, Sn, Ag and h-BN with metal matrix used for manufacturing of various machine parts (bearings, gears, seals and connectors) [1-3].

It is known [4-9] that introduction of solid lubricant components into the volume of materials can reduce a friction coefficient, but their efficiency depends significantly on operating conditions of a finished product. The presence of moisture has a positive effect on reduction of friction in materials containing graphite, and negatively affects materials with MoS$_2$ additives [10]. The negative effect of high humidity on performance was also established for materials with additives of BaF$_2$, CaF$_2$, which are subject to hydrolysis. In addition, use of one or another component as a filler is limited by temperature of its destructuration. Of the whole variety of non-metallic compounds related to solid lubricants, h-BN possesses good lubricating ability in conditions of high humidity and applicability in the temperature range up to 700 °C [11, 12].

There are works [15-19], in which authors evaluated the effect of concentration of hexagonal boron nitride on mechanical and tribological properties of composite materials on different bases. It was found that introduction of boron nitride into materials leads to decrease in friction at high loads and speeds [13, 15], h-BN has a low adhesive relationship with the matrix [13], its lubricity is lower than that of graphite due to higher van-der-Waals force between the layers of the crystal lattice of h-BN [15, 17]. A number of works [19] provide information on the achievement of a unique combination of mechanical and service properties of composite materials with boron nitride additives, which make them to be considered as seal materials for a flow path of steam turbines.

However, the formation of structure of composite materials with BN additives and its relationship with performance remain not thoroughly studied and are the subject of discussion among scientists.
2. Materials and procedures

For research was taken a powder of corrosion-resistant steel Fe-13% Cr-2% Mo with grain size distribution from 10 to 80 microns was chosen as base material, and was processed in accordance with GOST 13084-84. A decrease in the friction coefficient and better abrasion of the composite material were achieved by introducing a finely dispersed boron nitride powder BN according to TU.U 26.8-00222226-007-2003. The boron nitride particle diameter was from 1 to 10 μm, the particle thickness was from 0.7 to 2 μm. The effect of the concentration of BN powder in the charge on structure formation in compacts, their physical and mechanical properties, was studied on the samples of the following compositions:

1) Composition "1" - pressed specimens from pure powder PH13M2;
2) Composition "2" - powder PH13M2 + 0.5% BN;
3) Composition "3" - powder PH13M2 + 1% BN;
4) Composition "4" - powder PH13M2 + 2% BN;
5) Composition "5" - powder PH13M2 + 4% BN;
6) Composition "6" - powder PH13M2 + 4.5% BN;
7) Composition "7" - powder PH13M2 + 5% BN;
8) Composition "8" - powder PH13M2 + 6% BN.

The original powder components were thoroughly mixed. The obtained powder mixture was cold pressed into rectangular-shaped specimens of 50x10x10 mm. The compacts were sintered in electric furnaces in air atmosphere at 1200 °C. The sintering time was 2 h.

The microstructure of samples was studied using a JSM-6490LV scanning electron microscope. The density of samples was determined by hydrostatic weighing. The hardness of samples was determined by the Brinell method in accordance with GOST 9012. The static stretching was carried out on an Instron 5982 universal testing machine at an assumed operating temperature of seals (600 °C) in accordance with GOST 9651. The crosshead movement speed was 1 mm / min. The impact strength was determined on an Instron Ceast 9350 drop tower. The stored impact energy was 50 J.

3. Results and discussion

Figure 1 shows the microstructure of composite compacts with different contents of boron nitride. Pores of various shapes and sizes are observed on the polished surface of all studied samples. In the structure of a compact without boron nitride (figure 1, a), the pores have a globular shape and are predominantly located along grain boundaries, which indicates that the applied force during pressing is insufficient to form a denser structure due to high friction force between metal particles and, as a consequence, limited flow of diffusion processes during subsequent sintering.

The introduction of boron nitride into the initial charge leads to formation of extended grain-boundary pores in the microstructure (figure 1, b-d). The higher is the boron nitride content, the larger are the volume fraction and pore sizes. The mechanism of formation of the observed extended grain-boundary pores differs from the classical concept described in [20-23]. Due to different bulk densities of metal powder PKh13M2 and boron nitride, the volume of the mixed components in the charge is comparable. Taking this into account together with the chemical inertness and low adhesion of boron nitride, it can be assumed that the extended grain-boundary pores observed in the microstructure are areas with increased concentration of solid lubricant, which is removed along with the abrasive in preparation for metallographic analysis. An increase in the volume fraction of pores with an increase in the boron nitride content in the initial charge from 0 to 6% by weight is from 3.5% to 17%, respectively (figure 3).

The investigation of microstructure of compacts with boron nitride at higher magnification revealed the presence of dispersed nanosized 20 .. 500 nm thick boron nitride interlayers both along grain boundaries and inside grains formed during sintering (figure 2). The discovered structural features of compacts with addition of BN indicate the development of processes of diffusion mass transfer between metal particles during sintering by various mechanisms. In addition, BN particles are observed along
the edges of extended grain-boundary pores, which confirms the proposed assumption about the nature of their formation.

![Figure 1](image1.png)

**Figure 1.** Microstructure of the studied compacts with different content of boron nitride: $a$ – 0% BN; $b$ – 2% BN; $c$ – 4% BN; $d$ – 6% BN.

![Figure 2](image2.png)

**Figure 2.** View of dispersed boron nitride layers

The evaluation of hardness and density of the compacts (figure 3) showed their almost monotonic dependence on the boron nitride content. Thus, with an increase in the BN content, the density and hardness change from 6.7 g/cm$^3$ and 92 HB typical of a compact without boron nitride additives, to 6.0 g/cm$^3$ and 61 HB typical of samples with 6% BN.
Table 1 shows the results of determining the physical and mechanical properties of the studied powder compacts. Despite the monotonous gradual decrease in the hardness of compacts with an increase in the boron nitride content, the strength and impact strength are of extreme nature with the maximum values observed at 2% BN content. The maximum strength is 161 MPa, and the impact strength reaches 114 kJ / m².

The obtained data on the change in the microstructure of compacts with the boron nitride content growing allow considering the issue of its formation by first dividing the task into 2 parts: 1 - the effect of deformation during pressing, 2 - the effect of high temperature during sintering.

The boron nitride particles, due to their natural inertness, have a low adhesion ability [24] and cannot react with metal powder PH13M2. During pressing, part of BN particles uniformly distributed in the charge volume has a "lubricating effect", contributing to tighter adhesion of metal powder particles to each other. At the same time, the boron nitride particles under conditions of gradual compaction of material are partially subject to either direct rapid displacement into more free zones of reduced density, where the development of deformation processes did not begin due to insufficient applied force, or gradual slow displacement due to destruction of the relationship of layers bordering on metal particles (delamination of boron nitride particles (figure 5)). Ultimately, the directed motion of boron nitride particles during pressing leads to formation of closed grain-boundary porosity filled with BN particles (regions with increased BN concentration) in the volume of compacts. In this case, the higher is the boron nitride content in the initial charge, the greater is the volume fraction of grain boundary pores.

During sintering, the structure is formed according to the model described in [24]. At the interfaces, where there are point contacts of metal particles with each other, or the thickness of a nitride layer is so
small that due to thermal expansion of PH13M2 powder, they may come into contact with each other, the processes of diffusion mass transfer develop, sintering bridges are formed, and metal particles merge with formation of coarser grains. In this case, partial absorption of BN particles by growing metal grains is possible (Figs. 2 and 6, b). At the interfaces with a high content of ceramic particles and in areas with an increased concentration of boron nitride, the development of mass transfer and recrystallization processes is blocked. As a result, weakened boundaries are formed in the volume of compacts, which determine high abradability properties [13].

Thus, the introduction of finely dispersed boron nitride powder into the initial charge makes it possible to control the strength properties of grain boundaries formed during sintering by means of directed blocking of the development of diffusion processes during sintering, and as a consequence, to achieve the combination of strength and abradability of the composite material PH13M2-BN as required by operating conditions.

4. Conclusions
The introduction of finely dispersed BN with a graphite-like structure into the composition of powder compacts PH13M2 leads to formation of an extended closed grain-boundary porosity filled with boron nitride particles during deformation. Deformation treatment of the initial charge of powder compacts PH13M2-BN leads to formation of dispersed nanosized 20-500 nm thick boron nitride interlayers along grain boundaries due to partial delamination of boron nitride particles. During sintering, due to the development of diffusion mass transfer processes, the growing metal grains absorb dispersed BN particles.

The change in the boron nitride content in powder compacts ПХ13М2-BN allows varying their physical and mechanical properties in a wide range. The presented phenomenological models of influence of deformation and heat treatment describe the process of structure formation of composite materials on a metal base with additives of components belonging to the category of solid lubricants.

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