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STUDY OF STRUCTURE AND PROPERTIES OF SELF-FLUXING ALLOY AFTER MODIFYING WITH NANOSTRUCTURED AND MICRON-SIZED POWDERS OF CUBIC BORON NITRIDE AND HIGH PRESSURE AND HIGH TEMPERATURE TREATMENT

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The structural features and microhardness of sintered at high pressure and high temperature with the addition of nano- and micron-sized powders of cubic BN (cBN) self-fluxing nickel alloy PG-SR4 are studied. Based on the experiments, the modes of high pressure and high temperature treatment are established. The optimal content of the nanostructured cBN additive is determined, at which the material with the highest microhardness is formed. It is shown that the microhardness of the material with the addition of micron-sized cBN is 1.5–2 times lower than that for the samples with the same content of nanostructured cBN.

Keywords: PG-SR4 self-fluxing nickel alloy, mechanical activation, nanostructured and micron-sized cBN powders, modifying.

Introduction

Self-fluxing nickel alloys of the Ni-Cr-B-Si system are widely used in the technology of wear-resistant coatings for the restoration and hardening of tribological conjugation parts at elevated contact loads and temperatures [1].The fluxing elements boron and silicon included in the alloy contribute to lowering the melting temperature and deoxidizing the metal surface of the part with the formation of strong diffusion bonds between the coating and substrate materials during melting of the sprayed coating. For example, the coatings of powders of self-fluxing alloys deposited by a gas-flame method with reflow have high physical and mechanical characteristics: strength up to 400–500 MPa, hardness within 25–64 HRC, low friction coefficient, high wear and corrosion resistance [2, 3]. The most famous and widespread method of increasing the wear resistance of self-fluxing alloys is the development of composite materials using reinforcing additives in the form of carbides, borides, and transition metal nitrides [4]. Modifying by solid refractory compounds allows to change the structure of self-fluxing alloys effectively, contributes to its dispergation and increase the physical and mechanical properties, tribological and operational characteristics of materials based on them [5].

1. Formulation of the problem

Along with the refractory nanopowders such as Al_2O_3, SiC, TiB_2, TiC, WC, and others, single and multicarbide and oxide mechano-composites are also quite efficiently used as modifying additives for self-fluxing alloys [6, 7]. In this regard, it can be assumed that the use of cubic BN (cBN), which has high physical and mechanical properties [8], will significantly increase hardness and wear resistance and, accordingly, increase the life time of products based on self-fluxing alloys instead of traditional additives of refractory compounds. In addition, BN will serve as a source of

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boron, contributing to the formation of hardening phases of chromium and nickel borides in the heat treatment process.

It is known that the application of pressure during the synthesis of the materials based on cBN prevents the reverse phase transformation of cBN into a graphite-like (hexagonal) modification of BN (hBN) and provides the best combination of structural characteristics and strength parameters of the alloy [9]. The purpose of the work is to study of the structure and microhardness of samples of self-fluxing nickel alloy of the Ni-Cr-B-Si system obtained by high pressure and high temperature (HPHT) treatment, with the addition of nano- and micron powders of cubic BN.

2. The research technique.

For the manufacture of experimental samples, the powder of self-fluxing alloy PG-SR4 (GOST 21448–75) is chosen. It contains 15-18% Cr; 3.0-4.5% Si; 2.8-3.8% B; 0.6-1.0% C; no more than 5% Fe; and the rest is Ni. Two types of cBN are used as an additive: nanostructured powder with a particle size of 100–200 nm and micron-sized powder with a grain size of the main fraction within 40–60 μm. A mixture based on the PG- SR4 alloy with the addition of nano- or micron-sized cBN for HPHT treatment is obtained by mixing and mechanical activation (MA) in the attritor of the initial powders, their compacting and preliminary sintering in a protective atmosphere, dispersing the compacts to agglomerates and sieving the agglomerates into fractions. For HPHT treatment the agglomerate fraction with the size of 100–315 μm is used.

HPHT treatment of composite powders is carried out in an “anvil with a hole” high-pressure apparatus at pressures of 1.5–2.0 GPa and temperatures of 1000–1350 °C during 20 s. First, the “cold” compression of the mixture, placed in a special container made of lithographic stone, is carried out, and then the mixture is heated under pressure by direct electric current.

The studies of cross-sections of samples are performed by optical microscopy using Micro-200 metallographic microscope (Planar OJSC, Belarus). The microhardness of the samples is measured with a PMT-3 microhardness tester by Vickers diamond indenter with a load of 50 g. X-ray analysis is performed with Bruker D8 ADVANCE diffractometer in Cu-Kα radiation in an automatic recording mode.

3. Results and discussion

Figure 1 shows the appearance of the initial micron-sized and nanostructured cBN powders used as a modifying additive to prepare PG-SR4 and cBN composite powders. Composition powders in form of the agglomerates based on PG-SR4 with the addition of 2.5–10 vol. % micron-sized or nanostructured cBN have preliminary been prepared as described above.

Figure 2 shows the surface structure of the compacts made of PG-SR4 powder with the addition of nanostructured (Fig. 2 a) and micron-sized cBN (Fig. 2 b) after MA and sintering. The analysis of the surface of the compacts after sintering (Fig. 2) shows that the samples based on PG-SR 4 and nanostructured cBN has low porosity and are characterized by uniform structure. As a result of MA and sintering, nanostructured cBN is evenly distributed between particles of self-fluxing alloy and is fixed on their surface, leading to the formation of a composite powder with a cladding structure (Fig. 2 a). The compacts made of the composite powder PG-SR4 and micron-sized cBN after MA and sintering (Fig. 2b) consist of agglomerated particles of self-fluxing alloy and cBN crystals and have a much more porous and inhomogeneous dendritic type structure. Then the compacts are mechanically dispersed, and the resulting agglomerates of composite powders are sieved into fractions. As a result of the HPHT-treatment of the agglomerated composite powders with the additions of nano- and micron-sized cBN, the cylindrical samples with a diameter of 10 mm and a height of 8 mm are obtained. Then they are polished at the ends with diamond paste. In the process of preparation of the cross-sections, it is defined that the material is highly brittle, and this does not allow preparing high-quality cross-sections on them to conduct further research.

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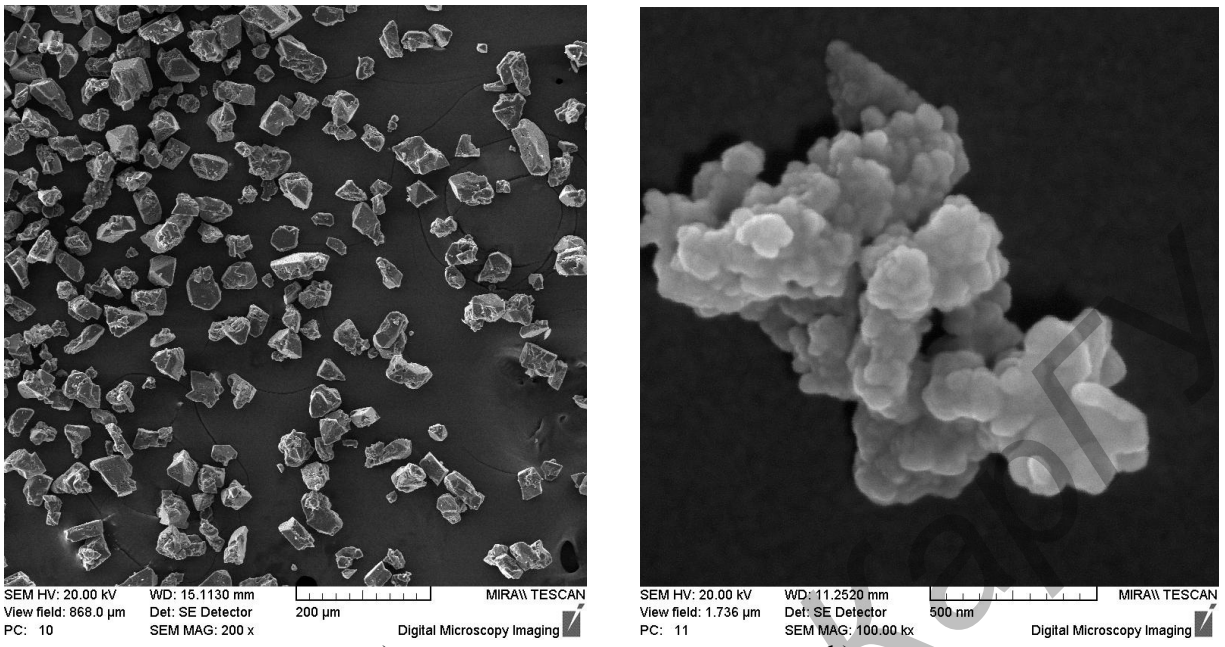
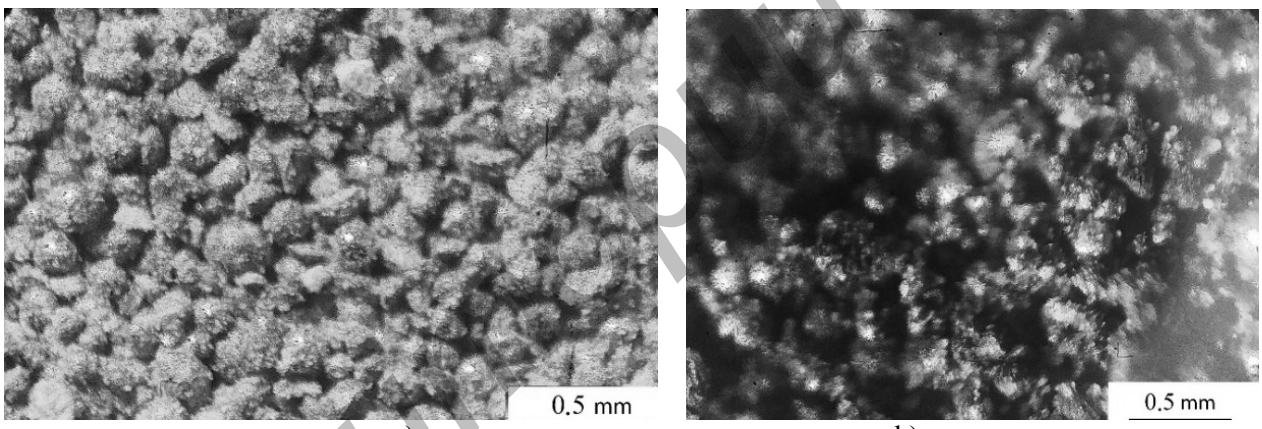


a) b)

Fig.1. Initial cBN powders used as additives: micron-sized powder (a); nanostructured powder (b).



a) b)

Fig.2. The surface of the compacts based on PG-SR4 powder after MA and sintering in a protective atmosphere: PG- SR4 and nanostructured cBN (a);PG-SR4 and micron-sized cBN (b)

Therefore, in order to reduce the brittleness of the material, the powder of the initial PG-SR4 alloy of the same fraction has been additionally introduced into the mixture before the HPHT treatment. Figures 3a and 3b show the photographs of the surface of the samples based on self-fluxing alloy with the addition of nanostructured cBN after HPHT treatment. It has been established that the main structural components of the sintered material are the matrix self-fluxing alloy based on a solid solution of Cr in Ni, the chromium boride phase Cr2B, as well as BN particles, that are evenly distributed in the material. In the material obtained at temperatures up to 1200 °C, the aggregates of cBN particles are uniformly distributed in the matrix and can be clearly seen on the background of the matrix alloy in the form of point inclusions of submicron sizes and individual polyhedral particles of 5–10 μm in size (Fig. 3a) formed as a result of recrystallization of nanostructured cBN [9]. In the samples of the material obtained at temperatures above 1200 °C, boron nitride is present in the form of light particles with a size of 5–15 μm of lamellar form that is characteristic of the graphite-like phase of BN. Moreover, with increasing the temperature of the HPHT treatment, the growth of individual alloy particles above 300 μm can be marked (Fig. 3b).

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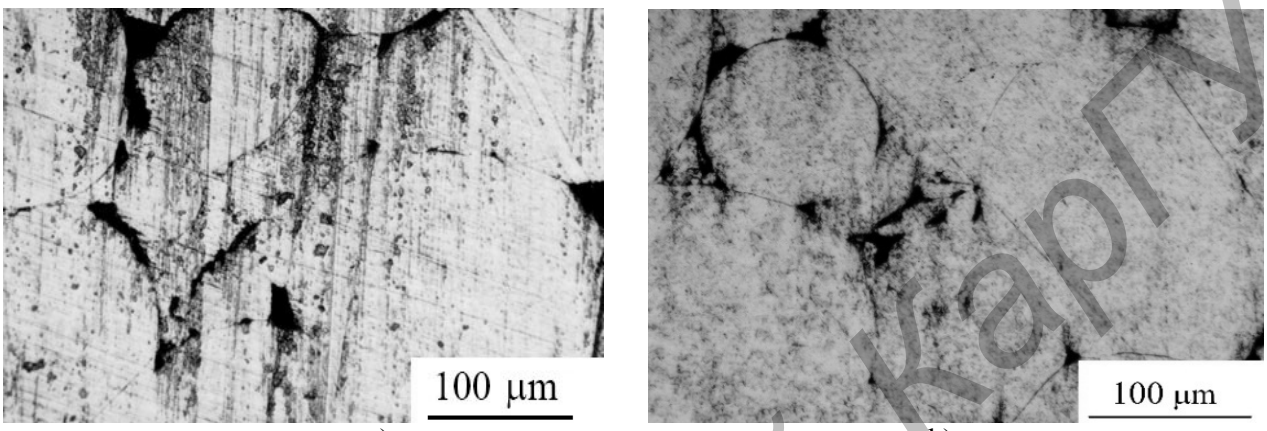
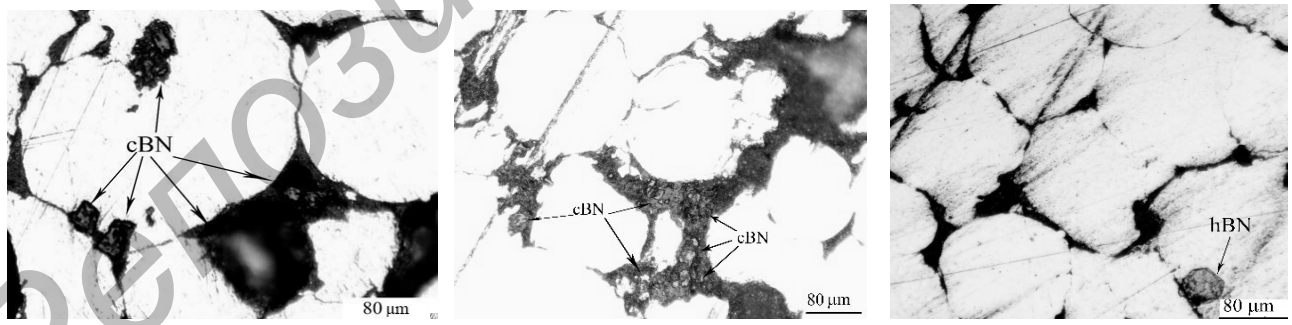
Microhardness measurements of the samples show that for the material containing nanostructured cBN in the range of 2.5–3.5 vol. %, microhardness values are 11.8–13.7 GPa, and this is 18–32% higher than that for the samples containing no additives. An increase in concentration of nanostructured cBN promotes embrittlement of the material, and an increase in temperature leads to a decrease in microhardness due to the developing reverse phase transformation of cBN into a graphite-like modification of BN.



a) b)

Fig.3. The surface structure of the samples based on self-fluxing alloy PG-SR4 and nanostructured cBN after the HPHT treatment: The temperature of the HPHT treatment is 1100 ^{o}C (a); 1350 ^{o}C(b)

Figure 4 shows the surface of the samples based on self-fluxing alloy with the addition of micron-sized cBN after the HPHT treatment. The analysis of the material structure shows that cBN crystals are mainly between the alloy particles (Fig. 4a). At the same time, together with the initial cBN, the material contains small fragment-type cBN crystals formed as a result of crushing of larger particles under pressure (Fig. 4b). In addition to particles of the indicated types, cBN crystals with a size of 5–8 μm of a pyramidal habit are also observed. They could be formed as a result of dissolution and crystallization of cBN from the melt under pressure (Fig. 4b). Moreover, as in the case of the use of nanostructured cBN additives, a graphite-like BN is formed in the material containing micron-sized cBN with an increase in temperature (Fig. 4c).



a) b) c)

Fig.4. The surface structure of the samples based on self-fluxing alloy PG-SR4 and micron-sized cBN after the HPHT treatment: large cBN crystals along the boundaries of the alloy particles (a); small cBN crystals formed as a result of crushing and recrystallization of primary cBN crystals (b); formation of graphite-like BN(c)

The maximum microhardness of PG-SR4 samples with the addition of micron-sized powder cBN is in the range of 6.4–7.1 GPa, and this is 1.5–2 times lower than that for the material with the same content of nanostructured cBN sintered at the same parameters of the HPHT treatment.

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Conclusion

The results show the promise of using of cBN nanostructured powder as a modifying additive for chromium-nickel self-fluxing alloys. It is shown that nanostructured cBN is distributed in a matrix based on self-fluxing alloy in the form of inclusions of submicron sizes and individual polyhedral particles up to 10 μm in size, while cBN micron-sized powder crystals are located between self-flux alloy particles.

In the process of HPHT treatment of the PG-SR4 alloy with the addition of cBN, the latter undergoes a number of structural and phase transformations: recrystallization of cBN, crushing of large cBN crystals, formation of secondary cBN crystals as a result of dissolution and recrystallization of the initial cBN, and the formation of a graphite-like BN.

Microhardness of the material with the addition of 2.5–3.5 vol. % of nanostructured cBN is 11.8–13.7 GPa, and this is 18–32% higher than that for the samples containing no additives of cBN. The samples with the addition of the micron-sized cBN powder are characterized by microhardness values in the range of 6.4–7.1 GPa, and this is 1.5–2 times lower than that for the material with the same content of nanostructured cBN.

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