HIGHLY CONCENTRATED CERAMIC BINDER SUSPENSIONS BASED ON SILICON CARBIDE

V. A. Doroganov[1] **and Yu. N. Trepalina**[1]

Translated from *Novye Ogneupory*, No. 8, pp. 50 – 52, August 2010.

Original article submitted October 1, 2009.

Highly concentrated ceramic binder suspensions (HCBS) based on silicon nitride are prepared and studied. The main features of physicomechanical properties of specimens based on HCBS with heat treatment are established. The possibility is demonstrated of using HCBS as a binder in the production of refractory material for various purposes.

Keywords: highly concentrated ceramic binder suspensions, silicon carbide, refractories.

Development and introduction of highly efficient materials that operate reliably under extreme conditions is a problem of considerable importance whose solution will mainly determine acceleration of the rate of scientific and technical progress.

With combined action of high temperature, chemical and corrosive media, erosion by solid particles and an electric current, in many cases known metallic and ceramic materials from which they are manufactured cannot provide structural endurance. Among non-metallic refractory compounds, that are the main broad class of materials with special physical properties, a visible position is occupied by silicon carbide.

Obtained by Bercellius for the first time in 1824 over the next hundred years silicon carbide was used extensively in industry as the basis of certain abrasives, refractories and electrical engineering materials. Industrial preparation of silicon carbide in an electric resistance furnace due to reaction of silicon with carbon was assimilated in 1893. This method has not been changed in principle up to the present time. In nature silicon carbide is encountered extremely rarely. It was detected for the first time a crater meteorite (Arizona, USA), and it was subsequently named by Kuntz (in 1905) moissonite. In addition, silicon carbide was detected in kimberlites, olivines and volcanic breccias, and also in the form of inclusions in crystals of natural diamond.

In view of its high mechanical strength and abrasiveness over a wide temperature range SiC is used extensively in the production of silicon carbide refractories [1, 2]. Silicon carbide refractories have high physicomechanical properties and endurance, high mechanical strength, resistance to deformation at high temperature, and as a result of the absence of polymorphic transformations, it has a low linear thermal expansion coefficient (LTEC) and high thermal conductivity. Silicon carbide refractories are mainly manufactured from granular semidry or plastic mixes by compaction, ramming, or stretching. In industry there is extensive use of silicon carbide based on silica, high-alumina, argillaceous and nitride binders. Introduction of them changes the mix composition, the structure of interphase and intergranular boundaries, and correspondingly operates on finished object properties. A recent achievement in the field of silicon carbide refractories is self-bonded and recrystallized silicon carbide. The direct connection between SiC grains within them is achieved as a result of crystallization at very high temperature in a reducing atmosphere. The most effective silicon carbide materials are distinguished by high physicomechanical and corrosion property indices [3 – 9], obtained by activated or reaction sintering, and hot compaction at temperatures up to 2100ºC. A possible method for preparing silicon carbide materials is technology based on the use of silicon carbide suspensions. Studies for preparing these systems based on SiC and also a study of the properties of finished objects were the concern in the 1970s of I. S. Kainarskii and É. V. Degtyareva [10 – 12]. There is special interest in suspensions of silicon carbide prepared by special technology of highly concentrated ceramic binder suspensions (HCBS), developed by Yu. E. Pivinskii [13, 15].

Use of HCBS based on silicon carbide makes it possible to synthesize during their preparation highly reactive SiC particles, that provide formation of the optimum intergranular boundaries between grains, and sintering in this case may

[1] V. G. Shukhov Belgorod State Technological University, Belgorod, Russia.

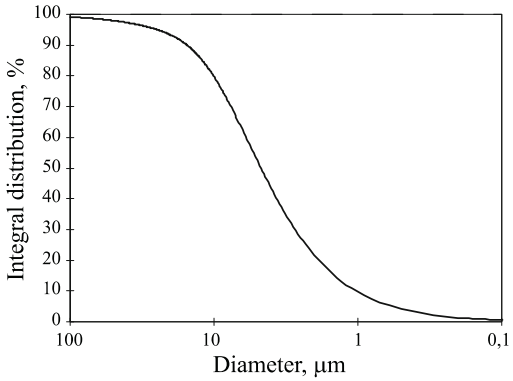

Fig. 1. Integral distribution of HCBS based on silicon nitride solid phase particle distribution.

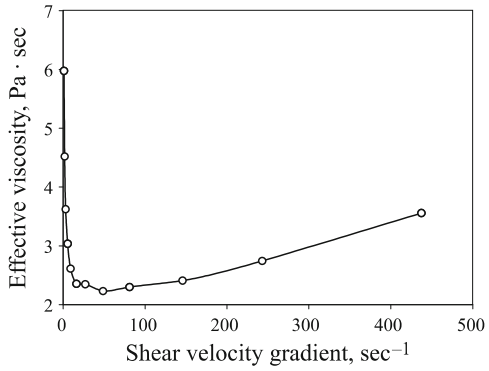

Fig. 2. Dependence of effective viscosity on shear velocity gradient of HCBS based on silicon nitride.

be carried at lower temperatures and even in an oxidizing atmosphere.

In order to establish these features of the technology starting materials used are black silicon carbide with a 98 – 99% SiC content and 0.5 – 1.0% SiO_2. Grinding of HCBS was carried out in a ball mill of periodic operation with stagewise charging of material. In view of the fact that the hardness of SiC is 9.0 – 9.5 on Mohs scale, grinding was performed in a ball mill with a volume of 80 liters with a corundum lining and grinding bodies. The diluting addition during grinding used was sodium triployphosphate and Reotan. The duration of grinding was 14 – 16 h. As a result of this a suspension was obtained with the following properties: apparent density ρ_{app} = 2.30 – 2.38 g/cm^3, volume concentration of solid phase C_v = 0.60 – 0.63, content of particles with a size of more 63 mm 1.5 – 2.5%, and relative moisture content of 15 – 17%.

A centrifuging method (separation factor 7500) was used to determine the concentration of particles with a size of less 0.1 μm in a suspension that was 0.05 – 0.08%. An integral curve is shown in Fig. 1 for the grain size distribution of the HCBS obtained. The suspension has significant polydispersion (polydispersion factor K_p about 5.5) and a quite high content of particles with a size of less than 1 mm (about 10%) with a values of median diameter of 4.7 – 4.9 μm. An HCBS based on silicon carbide has thixotropic-dilation flow with a minimum viscosity 2.2 – 2.3 Pa · sec with shear velocity gradient of 50 sec^{-1} (Fig. 2).

Slip casting in a gypsum mold was used to form cubic specimens with an edge of 3 cm. The molded specimens were dried at 100 – 110°C for 6 h, and then heat treated in an oxidizing atmosphere at 1100, 1200, and 1300°C with exposure at the maximum temperature for 3 h. the physicomechanical properties of fired specimens are presented in Figs. 3 and 4. Analysis of these dependences make it possible to conclude that the optimum firing temperature may be considered as 1100°C.

The ultimate strength in compression of specimens exceeds 80 MPa, the apparent density is 2.16 g/cm^3, the linear

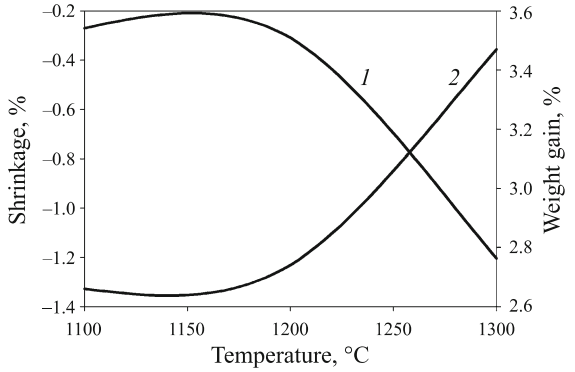

Fig. 3. Dependence of shrinkage (*1*) and weight gain (*2*) on firing temperature for specimens based on silicon carbide HCBS.

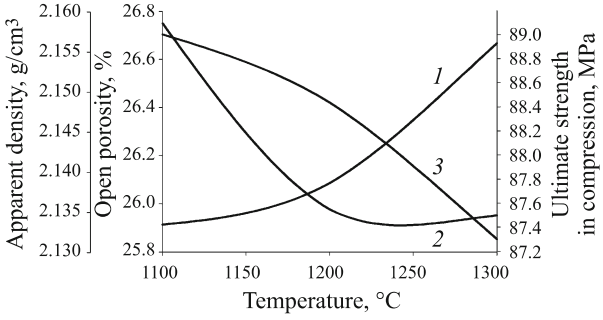

Fig. 4. Dependence of open porosity (*1*), apparent density (2) and ultimate strength in compression (*3*) on firing temperature fro specimens based on silicon carbide HCBS.

dimensions increase during firing by 0.2 – 0.3%. The latter is connected with surface oxidation of silicon carbide and formation of cristobalite. It should be noted intense volumetric oxidation of specimens commences from 1150°C is accompanied by a weight increase, growth of porosity, a reduction in density, and loss of strength.

Thus, it has been established that of HCBS technology in preparing objects based on silicon carbide makes it possible

to obtain silicon carbide materials by different molding methods (semidry compaction, vibration compaction, casting, etc.) without additional introduction of a different form of binder. Here the required physicomechanical properties are achieved at a temperature up to 100°C even with firing in an oxidizing atmosphere. However, high open porosity of an object promotes intense volumetric oxidation of silicon carbide and rapid loss of operating properties, particularly at elevated temperature. Therefore there is a requirement for using composite compositions or creation of additional protective coating at the surface of SiC objects. Protective coatings may be synthesized both with the use of vacuum plasma deposition [16], and with use of special pastes applied to an object during firing [17] by a traditional method.

The research was carried out within the framework of FCP "Scientific and scientific-teaching personnel of innovative Russia," for 2009 – 2013 in the area "Creation and treatment of composite ceramic materials," GK No. P398.

REFERENCES

1. I. S. Kainarskii, and É. V. Degtyareva, *Carborundum Refractories* [in Russian], Metallurgiya, Moscow (1963).
2. G. G. Gnesin, *Silicon Carbide Materials* [in Russian], Metallurgiya, Moscow (1977).
3. l. N. D'yachkova, E. V. Zvonarev, V. M. Shelekhina, et al., "Preparation of silicon carbide materials by reaction sintering," *Inzh.-Fiz. Zh.*, No. 2, 260 – 263 (1997).
4. K. Osami, "New ceramic materials," *Nippon. Kikai Gakkaisi. J. Jap. Soc. Mech. Eng*., **85**. No. 769, 1398 – 1405 (1982).
5. N. Koesake and O. Asua, "High quality ceramic of silicon carbide," *Kagaku Koge. Chem. Hid*., **33,** No. 11, 977 – 982 (1982).
6. I. Kim, M. Mitomo, and H. Hiroshuru, "Grain growth and crack resistance of fine-grained silicon carbide ceramic," *Taikabutsu. Refractories*, **78**, No**.** 11, 3145 – 3148 (1995).
7. S. Yutaka, N. Takayuki, and W. Fumihiro, "Fabrication of nanograined silicon carbide by ultra-high pressure hot static pressing," *J. Amer. Ceram. Soc.*, No. 3, 771 – 773 (1999).
8. Yu. N. Vil'k and A. P. Garshin, "Some properties of materials based on self-bonded silicon carbide and its application potential," *Ogneupory Tekhn. Keram.,* No. 7, 11 – 14 (1996).
9. L. I. Fedotova, A. G. Rozenblat, and A. I. Rekov, "Change in the microstructure of self-bonded silicon carbide on heating in a vacuum," *Ogneupory*, No. 4, 57 – 59 (1972).
10. I. S. Kainarskii, É. E. Degtyareva, and I. P. Safronova, "Rheological and technological properties of silicon carbide suspensions," *Ogneupory*, No. 12, 27 – 32 (1974).
11. I. P. Safronova, É. E. Degtyareva, and I. S. Kainarskii, "rheological and technological properties of silicon carbide suspensions and castings based on them with addition silica and clay," *Ogneupory*, No. 1, 45 – 51 (1975).
12. I. P. Safronova, É. E. Degtyareva, and I. S. Kainarskii, "Study of the effect of additions on the strength of silicon carbide castings," *Ogneupory*, No. 3, 41 – 44 (1975).
13. Yu. E. Pivinskii, *Ceramic and Refractory Materials. Selected Work, in 2 Vol. Vol. 2* [in Russian], Stroizdat, St Petersburg (2003).
14. Yu. E. Pivinskii, F. S. Kaplan, and P. B. Dyakin, "Regulation of the physicomechanical properties of HCBS based on silicon carbide," Physicochemical treatment of lean natural raw material and industrial wastes in preparing heat-resistant materials: Proc. All-Union Conf, Syktivkar (1989).
15. Yu. E. Pivinskii and P. B. Dyakin, "Rheological and physicochemical properties of HCBS and ceramic concretes in the system Al_2O_3–SiO_2–SiC," Fundamental studies and new technology in building materials science: Proc. All-Union Conf., Belgorod (1989).
16. S. V. Zaitsev, I. Yu. Moreva, and E. I. Yevtushenko, "Features of forming nitride and oxide modified films of titanium on a ceramic surface," XXI All-Russian Meeting on Temperature Resistant Functional Coatings, St. Petersburg, 26 – 28 April, 2010, Admiral, St. Petersburg (2010).
17. V. A. Doroganov, E. A. Doroganov, N. S. Bel'maz, et al., "Development and study of composite refractory materials based on modified dispersed systems," *Novye Ogneupory*, No. 11, 35 – 41 (2009).