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I. Ya. Guzman and A. V. Dovbysh

Translation of "Issledovaniye porovoy struktruy keramiki poluchennoy metodom shlikernogo lit'ya" Tr. Mosk. Khim. Tekhnol. Inst., 1976, Vol. 92, pp 70-72.

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16. Abstract

The porosity of the slip cast Si_3N_4 is similar to that of pressed Si_3N_4 formed at 2500 kg/cm^2. The porosity of cast Si oxynitride is equivalent to that of samples stressed at 10,000 kg/cm^2. Crucibles formed from these materials by slip casting have high thermal shock and corrosion resistance.

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STUDY OF THE PORE STRUCTURE OF CERAMICS PREPARED BY THE SLIP CASTING METHOD

I. Ya. Guzman, A. V. Dovbysh

Monophase materials made of silicon nitride and oxynitride, as /70*
well as composition materials based on silicon carbide with nitride bonds possess a complex of valuable properties, primarily high corrosion resistance in relation to melts of various salts and nonferrous metals [1-3]. The corrosion resistance of the products is significantly affected not only by the value of the porosity, but also by the structure of the products, in connection with which it is necessary to evaluate the pore structure of samples obtained by the slip casting method, which is promising for making products of a complex profile, and particularly thin-walled products.

The initial materials used for making the slips were silicon of grade KR-0 (GOST 2169-43), quartz sand from the Tashlinskiy deposit, and green silicon carbide from the Zaporozhskiy Plant. All the materials were subjected to dry vibration grinding for various times to a specific surface on the order of 5-8 m^2/g, determined by the method of low-temperature nitrogen adsorption (Table 1).

The slip casting was performed in dry gypsum molds covered with /71
a protective film of sodium alginate, in an optimal sphere of pH 8-10.

Firing is done on a muffle type device in a medium of industrial nitrogen according to the conditions recommended in works [4-5] with reduction of the holding time at final temperature to 1-2 hours for the thin-walled samples.

* Numbers in margins indicate foreign pagination.

TABLE 1. Specific surface of powders

Material	Duration of vibropulverization, hrs.	Specific surface area, m^2/g
Silicon	4	5.2
Mixture $Si-SiO_2$ (60:40)	5	6.3
Mixture SiC-Si (60:40)	$\frac{3-4^*}{0.5}$	8.2
Mixture $SiC-Si-SiO_2$ (70:18:12)	$\frac{3-4-2^*}{0.5}$	7.9

* Given in the numerator is the time for pulverization of the ingredient components, respectively; in the denominator -- time for combined pulverization of the mixture.

The properties of the castings and fired products subjected to the study of pore structure are presented in Table 2.

TABLE 2. Properties of raw and fired ceramics

Slip indicators		Raw goods indicators			Fired products	
Initial material	slip pH	apparent density, g/cm^3	apparent porosity, %	qualitative phase composition	apparent density, g/cm^3	apparent porosity, %
Si	8.1	1.63	30.1	Si_3N_4	2.38	24.1
Mixture $Si-SiO_2$ (60:40)	9.85	1.50	35.0	Si_2ON_2	2.20	19.0
Mixture SiC-Si (60:40)	9.9	1.82	33.5	$SiC-Si_3N_4$	2.19	28.0
Mixture $SiC-Si-SiO_2$ (70:18:12)	9.45	1.96	30.9	$SiC-Si_2ON_2$	2.20	27.8

The pore distribution by size was evaluated by the method of mercury porometry according to the methodology of work [6]. The results of the pore structure determination for the fired ceramics obtained by slip casting are presented in Figures 1, 2 and in Table 3.

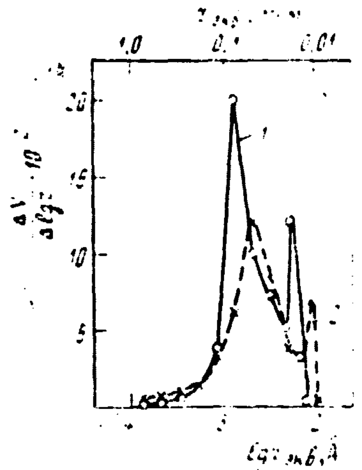

Figure 1. Differential porograms of samples: 1 - silicon nitride; 2 - silicone oxynitride

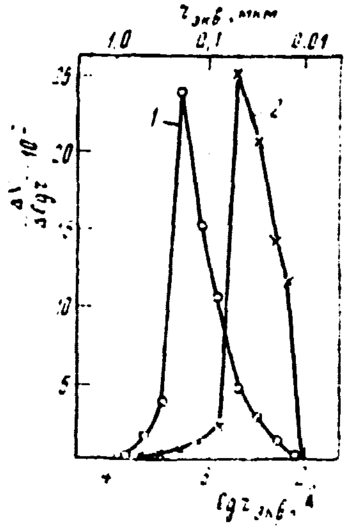

Figure 2. Differential porograms of compositional ceramics based on silicon carbide: 1 - with nitride; 2 - with oxynitride bonding.

An analysis of the differential porograms of the samples showed that in practically all the samples there is an absence of large pores (larger than one μm). The main mass of pores corresponds to a size of 0.5 - 0.01 μm. /72 The samples made of silicon nitride and oxynitride have a similar pore structure, with pores 0.1 - 0.01 μm in size comprising 83-85% (See Table 3).

TABLE 3. Distribution of pores by size in fired samples of monophasal and composition ceramics

Pore radius, m	Material			
	Si_3N_4	$SiON_2$	$SiC-Si_8N_4$	$SiC-Si_2ON_2$
Greater than 1	0.6	1.7	1.8	1.1
1-0.5	0.6	1.0	1.1	0.7
0.5-0.1	13.8	14.3	71.3	2.5
0.1-0.01	85.0	83.0	25.8	95.7
Smaller than 0.01	--	--	--	--

Despite this fact, the samples made of composition ceramic differed in their pore structure. In comparing the differential porograms of the samples made of silicon carbide with nitride and oxynitride bonding, we may conclude that the former have a coarser porosity and the main mass of the pores is within the margins of 0.5 - 0.1 μm, while in the latter it is within the margins of 0.1 - 0.01 μm (see Figure 2). The different pore structures of the samples may be explained by the character of interaction of the reaction bonding with the silicon carbide filler. Works [7.8] discuss the chemical interaction of silicon oxynitride with the grains of silicon carbide, partially surrounded by an oxide film, unlike silicon nitride, for which no such interaction is found. This chemical interaction prevents the formation of larger pores along the boundaries of the bonding and the filler.

Let us compare the pore structure of samples made of monophase ceramic obtained by slip casting with the pore structure of samples obtained by pressing at various specific pressures from powders of identical dispersity. The cast samples made of silicon nitride were similar in their pore structure to the pressed samples at a specific pressure of 2500 kG/cm^2. The oxynitride samples obtained by casting were similar to the same ones obtained by pressing at specific pressure of 10,000 kG/cm^2.

We must note that crucibles made of the indicated materials and subjected to tests for heat resistance (at 950°C in an air medium) and for corrosion resistance in relation to melted cryolite simultaneously, after 15 melts showed no visually apparent changes.

Thus, the slip casting method from silicon and its mixtures with subsequent reaction baking may be used to obtain products on the basis of nitride, and silicone oxynitride, as well as silicon carbide with nitrice bonds. These products do not yield by fineness of their pore structure to products obtained by pressing at rather high specific pressures.

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