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SELECTION OF A PLASTICIZER FOR PRODUCING MATERIALS BASED ON SILICON NITRIDE POWDERS

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ANNOTATION:

The article presents the prospect of using materials based on silicon nitride in various branches of technology and a comparative analysis of the cost of raw materials for the production of tungsten hard alloys, tool materials based on silicon nitride, as well as a literature review of modern methods for producing silicon nitride powders and their properties. It is shown that at present the main methods of obtaining silicon nitride powders are self-propagating high-temperature synthesis (SHS).

Keywords: **Material, technology, method, synthesis, ceramics, self-propagating high-temperature synthesis of synthesis oxides, carbides, silicon nitride, tungsten carbide, method, synthesis, powder, grinding, adiabatic temperature azaite technologies, combustion wave front, carbothermal reduction, gas-phase synthesis, imide.**

INTRODUCTION:

Among non-metallic refractory compounds, silicon nitride Si_3N_4 has found the widest application. Due to its exceptionally high hardness and a number of other specific properties, Si_3N_4 is now widely used, first of all, in the tool and manufacturing industries, and is also a part of wear-resistant and chemically resistant materials. The increased interest in this class of material is primarily due to a successful set of characteristics, in particular, a successful combination of strength, hardness, thermal stability and thermal conductivity, corrosion resistance and wear resistance. Due to this, ceramics based on Si_3N_4 are often used in such industries as propulsion, rocketry, nuclear and chemical industries, metallurgy, etc.

Practically all existing methods of obtaining powder materials can be used to manufacture products from Si_3N_4. In this case, the technology includes three main operations: preparation of Si_3N_4 powder, pressing and sintering. However, due to the high hardness of Si_3N_4 powder, high melting point, low ductility and high brittleness, there are peculiarities in the implementation of this or that operation. As a rule, for the manufacture of articles from Si_3N_4 powders, powders with a particle size of less than 50 microns (often 1-10 microns) are used. The latter is due to the fact that such powders have high activity during sintering, and therefore ensure the production of dense products [1-3].

Compaction of many powder bodies under the action of external pressure occurs due to the movement of particles relative to each other, their denser packing and, ultimately, due to plastic deformation. For Si_3N_4 powder, instead of plastic deformation, the particle shape is adjusted by brittle cleavage of the particles at the sites of the greatest tangential stresses, therefore, the main characteristics of the powders in this case are

hardness, yield strength and friction force between particles during their mutual movement, which depends primarily on the shape particles. In this respect, pressing billets from Si_3N_4 powder is associated with great difficulties, since Si_3N_4 powders are almost completely devoid of plasticity and, as a consequence, there is an extremely weak adhesion interaction between powder particles in the compact. Therefore, in practice, when molding the powder, Si_3N_4 is mixed with a plasticizer.

The most common method for molding Si_3N_4 powder is pressing in closed molds. In this case, the compaction process often does not obey the laws described by various equations of the contact theory of pressing, which considers the process proceeding from the deformation of individual particles, or a theory that identifies the deformation of powder and compact bodies [4]. This approach to describing the process of pressing Si_3N_4 powder is unacceptable, since their particles do not deform at the applied pressures, and the pressing acquires the properties of a solid body only due to the presence of a plasticizer whose rheological characteristics differ from those of solids [5]. Therefore, the optimal pressing conditions are selected experimentally for each batch of Si_3N_4 powder. This is due to the fact that the compaction of solid brittle substances practically does not depend on the physical properties of the material of the particles, but is mainly determined by their dispersion and state of the surface. In this case, the main parameters that determine the optimal conditions for pressing are the given density, the uniformity of its distribution in the volume and the optimal content of the plasticizer.

This work is devoted to the study of the influence of the types of plasticizer and its content on the residual porosity of pressed in closed molds and sintered Si_3N_4 powders.

MATERIALS AND RESEARCH METHODS:

The most commonly used plasticizers were selected for the study: paraffin, paraffin solution in gasoline, rubber solution in gasoline, 2 - 5% aqueous solution of polyvinyl alcohol, starch paste. The amount of added plasticizer during pressing in closed molds was selected in the range from 5 to 15% of the weight of the Si3N4 powder. The characteristics of the plasticizers used in the research are given in table. 1.

Table 1. Characteristics of plasticizers used in plasticized mixtures

Plasticizers	Solvent to soluble ratio	Ignition temperature in air, °C	Content, %	
			Ash	Coke
Paraffin	-	400	0	0
Paraffin solution in gasoline in benzene	 2:1 2:1	 260 400	 0 0	 0 0
A solution of rubber in gasoline	10:1	260	0,94	1,6
A solution of polyvinyl alcohol in water	4:1	450	0,58	1,2
Starch paste	4:1	450	2,45	6,8
Bakelite solution in alcohol	10:1	430	-	50-52

Silicon nitride powders obtained by the method of self-propagating high-temperature synthesis were used as starting materials [6]. Si_3N_4 powder was a well-crystallized fibrous silicon nitride particle with an average length of about 2 μm and a thickness of up to 200 nm. The content of α - silicon nitride is not less than 95% (Fig. 1), the specific surface of the powder is 8.2 m^2 / g. Impurity content: O_2 = 1.64 wt%, Fe = 0.023 wt%.

The granulometric composition of the silicon nitride powder is shown in Fig. 2.

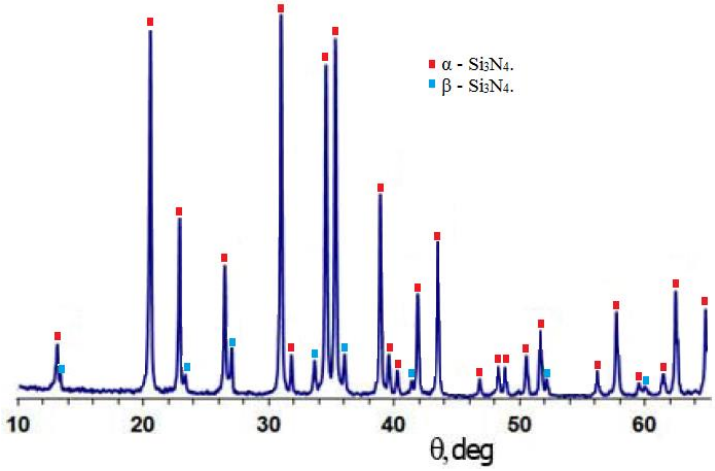

Fig.1. X-ray diffraction pattern of silicon nitride powder obtained by SHS

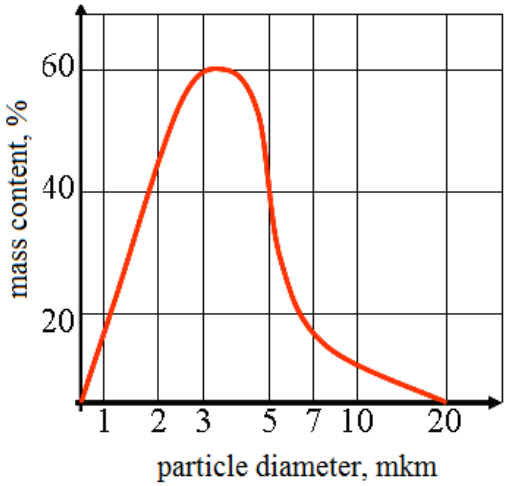

Fig.2. Granulometric composition of silicon nitride powder obtained by SHS method

Mixing of powders of silicon nitride and plasticizer was carried out in a worm-blade mixer for 3 hours. Then the resulting mixture was passed through a sieve with a mesh size of 0.16 mm. The pressed mixture was pressed in closed molds at a pressing pressure of 25 MPa. The pressed samples had a cylindrical shape with dimensions: diameter 20 mm, height 20 mm. All samples were pressed at the same pressures, but differed from each other in the content and types of plasticizer from 5 to 10%. The pressed samples were sintered in an oven with a graphite heater in an argon atmosphere. Sintering was carried out in two stages: the first stage - heating the samples to 500 °C and holding at this temperature for 30 minutes to remove the plasticizer, the second stage - heating the samples to 1800 °C and holding at this temperature for 1 hour to sinter the samples. In fig. 3. shows the samples obtained from silicon nitride.

The density of the sintered samples was determined in a dry and completely impregnated state. The dry density was determined by dividing the sample weight after extraction and drying (m^2) by its volume (V). The fully impregnated density was determined by dividing the mass of the oil-impregnated sample (m^3) by its volume. The sample volume was determined by weighing the sample in air and in a liquid of known density. The volume was calculated as the difference between the two results divided by the density of the liquid, after which the residual porosity of the samples was found.

Microstructural analysis of the samples was carried out on a NEOPHOT-21 metallographic microscope (Germany). For this, special thin sections were made from sintered silicon nitride samples.

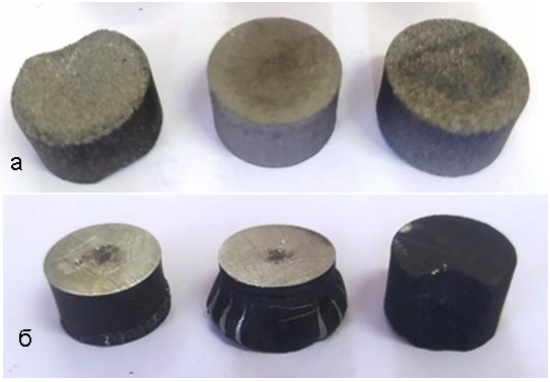

Fig. 3. Compressed samples of silicon nitride (a) before and (b) after sintering.

RESEARCH RESULTS:

The study of pressed samples showed that with an increase in the content of the plasticizer in the powder mixture of silicon nitride - plasticizer from 5% to 15%, regardless of the type of plasticizer, the compressibility of

the mixtures improves and the strength of the pressing increases. The best smooth surfaces were formed in the samples obtained from mixtures containing 10 ... 12% paraffin solution 2:1 in gasoline and benzene.

The study of the porosity of the sintered samples showed that with an increase in the content of the plasticizer in the powder mixture from 5% to 10%, regardless of the type of plasticizer, the residual porosity of the samples decreases. The influence of the type and content of plasticizers on the residual porosity of sintered silicon nitride samples are shown in fig. 4.

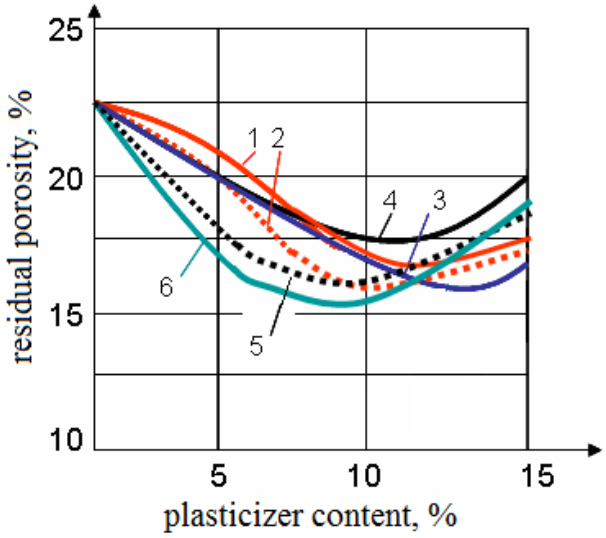

Fig. 4. The graph of the dependence of the residual porosity of the sintered samples on the content of the plasticizer: 1 - paraffin solution (2: 1) in gasoline; 2 - paraffin solution (2: 1) in benzene; 3 - solution of rubber in gasoline (10: 1); 4 - solution of polyvinyl alcohol in water (4: 1); 5 - starch paste (4: 1); 6 - bakelite solution in alcohol.

Microstructural analysis of sintered samples of silicon nitride showed that insufficient content of plasticizer (less than 7%) in the mixture will lead to the appearance in the structure of the sintered powder material of large pores with a size of 250 ... 300 microns. With an increase in the plasticizer content from 8 to 12%, the amount of large pores decreases, and the structure of the material becomes finely porous.

With an increase in the plasticizer content from 12 to 15%, small pores with a size of 20 ... 25 μm begin to enlarge to a size of 70 ... 80 μm, but in contrast to the porous structure formed due to the lack of a plasticizer content, this porous structure contains large pores with a size of 70 ... 80 μm are evenly distributed throughout the volume of the porous material. The porous structure of sintered silicon nitride samples is shown in fig. 5.

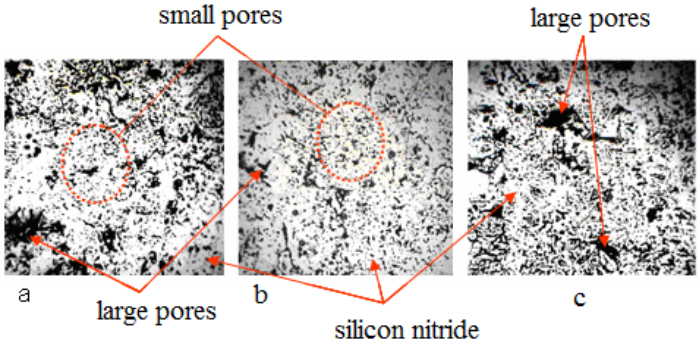

Fig. 5. Microstructure of sintered samples of silicon nitride, x300: a - plasticizer content less than 7%; b - 11%; c - 15%

CONCLUSION:

Studies have shown that the value of the residual porosity of the powder material based on silicon nitride after sintering depends little on the type of plasticizer. The value of the residual porosity of the sintered samples mainly depends on the quantitative content of the plasticizer. As the content of the plasticizer in the mixture increases to 10 ... 11%, the value of the residual porosity of the sintered materials decreases due to the improved compressibility of the silicon nitride parashka. When the content in the mixture is more than 12% of the plasticizer, the value of the residual porosity increases due to the enlargement of small pores caused by the growth of the thickness of the plasticizer layer between the silicon nitride particles.

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