Development of sapphire-like glass by sol-gel technology

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Abstract
The paper describes the study of methods for converting precursors into optically transparent aluminum oxide and identifies the most promising methods for the synthesis of sapphire glasses.

Introduction
The development of technological processes of manufacturing transparent and strong polycrystalline materials for optical engineering was made possible only as a result of systematic fundamental research of the kinetics and mechanism of sintering processes. Studying the effect of temperature, pressure, the surrounding atmosphere, impurities, and thermal prehistory of the initial materials on the compaction process of powders during sintering led to the development of two methods for obtaining polycrystalline materials with a density close to theoretical, i.e., the density of a monocrystalline substance is both transparent and durable.
Currently, methods for the synthesis of alumina ceramics, which include elements of sol-gel synthesis, are widely used. Alkoxy technology is a promising method for producing finely dispersed aluminum oxide.

Results and discussion
Xerogels of aluminum oxide were obtained by the alkoxide sol-gel method from aluminum-tri-sec-butoxide. Isopropyl or butyl alcohol was used as a solvent for liquid-phase hydrolysis; the molar ratio of aluminum alkoxide to alcohol was 1:8. To carry out the hydrolysis process and subsequent gelation, the following reagents were added to the alcohol mixture based on aluminum alkoxide: nitric acid, aluminum acetylacetate solution, alcoholic acrylamide solution, acrylic acid, citric acid, boron ethoxide, and ethanolamine. The maturation of the gels took place within 24 hours. The resulting xerogels dried at 40 – 50 °C for 3 – 5 days, depending on the geometry of gels. As a result of the rapid solvent evaporation rate, a xerogel had strain related to the capillary effect, resulting in cracking. Most xerogels formed during drying disintegrate into dense or unconsolidated agglomerates, depending on the type of the initial components. Suitable further drying, the gels were obtained using nitric acid, acrylamide, and boron ethoxide. Isopropyl or butyl alcohol was used as a solvent to reduce the viscosity of the system. Distilled water was used as a hydrolyzing agent. Acid hydrolysis was carried out in the

variant with an alkoxide sol-gel method. The formation of dense agglomerates was observed, which after drying, milled to nanopowder in a planetary mill. Depending on the type of initial components, the resulting xerogels desintegrate during drying into dense or loose agglomerates. The gel structure stabilizes the smallest particle size, preventing their rapid agglomeration. From these powders, blanks were formed by dry pressing in a steel mold on a hydraulic press at a pressure of 100 MPa. The pressed blanks were annealed for 1 hour at a temperature of 1000 °C. After that, the samples were sintered under the pressure of 5GPa (50 000 atmospheres) at the temperature of 500-600 °C.
Figures 1 shows the transmission spectra of typical samples of transparent ceramics.

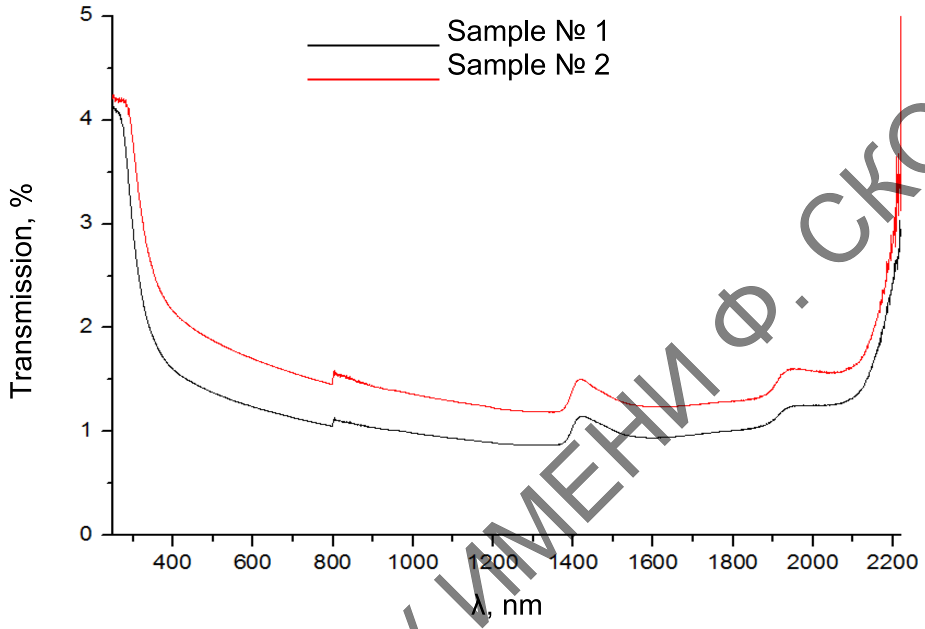

Fig. 1. The transmission spectra of samples №1 (h=1,5 mm) and №2 (h=1,6 mm) of transparent aluminum oxide ceramic samples prepared in GSU

Using the obtained powder to create products for chemical technologies can significantly reduce the total cost of equipment, production, and energy consumption compared to getting a single crystal counterpart. Table 1 shows the characteristics of transparent ceramic samples №1 and №2 synthesized by the sol-gel method and compares the features of the sol-gel silica glass and standard synthetic sapphire.

Table 1. Characteristic of transparent materials

Sample	Density, g/cm^3	Microhardness, MPa	Light transmission in the visible range,%
Quartz glass obtained by the sol-gel method	2.19	5128	90
Synthetic sapphire	3.98	20 000	95
Ceramic aluminum oxide (№1) GSU	4.15	9775	10 – 15
Ceramic aluminum oxide (№2) GSU	4.05	9272	15 – 20

Conclusion
The properties of the materials obtained by the sol-gel method are determined mainly by the composition and conditions of preparation of the gel, as well as its subsequent heat treatment regimes. An important task at this stage is to find the minimum time and temperature regime for the annealing of the crystalline phase of alumina and increase the strength of the preforms.

Samples of transparent ceramics were obtained from the aluminum nanopowders synthesized by the sol-gel process. The microhardness of the samples was about 104 MPa, the density of the sample was about 4.0 g/cm^3. The light transmission was about 15 – 20 % in the visible range. It is essential to find “condensing” additives and optimal synthesis parameters that will make it possible to obtain sapphire glass with the high light transmission in the visible range with the minimum time and temperature conditions for annealing the samples.