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RESEARCH ARTICLE

Cold sintering of α- and γ-modifications of aluminum oxohydroxides: A low-temperature route to porous corundum ceramics

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Abstract

Objectives. To obtain porous corundum ceramics using an innovative cold sintering process starting from different phase modifications of aluminum oxohydroxide—boehmite γ-AlOOH and diaspore α-AlOOH; to study the phase and structural properties of the resulting materials; and to assess their permeability to water.

Results. Cold sintering enables the formation of single-phase corundum ceramics with an open porosity of 47.9% directly from the initial boehmite powder with the addition of 5 wt % corundum in the presence of 20 wt % water at a temperature of 450°C, mechanical pressure of 220 MPa, and isothermal exposure for 30 min. Under the same conditions of cold sintering, a mixture of diaspore and boehmite was transformed into α-AlOOH ceramics. This then turned into corundum with an open porosity of 39% when calcined in air at 600°C for 1 h. The resulting materials had permeability for pure water above 5000 L/(m^2·h·bar).

Conclusions. Cold sintering is a promising approach to producing porous corundum ceramics which can be used in filtration systems. Compared to traditional ceramic technology, the new approach reduces energy, time, and labor costs in the material manufacturing. It also eliminates the need to use auxiliary substances (binders, pore-forming agents, etc.).

Keywords

cold sintering, aluminum oxide, aluminum oxohydroxide, corundum, boehmite, diaspore, porous permeable ceramics

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НАУЧНАЯ СТАТЬЯ

Холодное спекание α- и γ-модификаций оксогидроксида алюминия: низкотемпературный способ получения пористой корундовой керамики

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Аннотация

Цели. Получить пористую корундовую керамику с помощью инновационного метода холодного спекания с использованием различных фазовых модификаций оксогидроксида алюминия — бемита γ-AlOOH и диаспора α-AlOOH, изучить фазовые и структурные свойства полученных материалов и оценить их проницаемость для воды.

Результаты. С помощью холодного спекания в присутствии 20 мас. % воды при температуре 450°C, механическом давлении 220 МПа и изотермической выдержке 30 мин из исходного порошка бемита с добавлением 5 мас. % корунда была изготовлена однофазная корундовая керамика с открытой пористостью 47.9%. При таких же условиях холодного спекания смесь диаспора и бемита превратилась в керамику α-AlOOH, которая при прокаливании на воздухе при 600°C в течение 1 ч перешла в корунд с открытой пористостью 39%. Полученные материалы обладали проницаемостью для чистой воды более 5000 л/(м^2·ч·бар).

Выводы. Холодное спекание является перспективным методом для изготовления пористой корундовой керамики, которая может быть использована в системах фильтрации. По сравнению с традиционной керамической технологией новый подход снижает энергетические, временные и трудозатраты при изготовлении материала, а также исключает необходимость в использовании вспомогательных веществ (связующих, порообразующих агентов и пр.).

Ключевые слова

холодное спекание, оксид алюминия, оксогидроксид алюминия, корунд, бемит, диаспор, пористая проницаемая керамика

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1. INTRODUCTION

Ceramics based on aluminum oxide α-Al_2O_3 are widely used in various fields of engineering due to their mechanical characteristics, chemical and temperature resistance, stable dielectric properties and compatibility with biological tissues [1–3]. Aluminum oxide exists in the form of a number of transitional phase modifications which as a result of successive transformations under heating transform into α-Al_2O_3. In practice, the following chains of transformations [4, 5] are most often realized [4, 5] (formulas 1 and 2):

$$\gamma\text{-Al(OH)}_3 \xrightarrow{300^\circ\text{C}} \chi\text{-Al}_2\text{O}_3 \xrightarrow{970^\circ\text{C}} \kappa\text{-Al}_2\text{O}_3 \xrightarrow{1100^\circ\text{C}} \alpha\text{-Al}_2\text{O}_3, \quad (1)$$

$$\gamma\text{-AlOOH} \xrightarrow{450^\circ\text{C}} \gamma\text{-Al}_2\text{O}_3 \xrightarrow{750^\circ\text{C}} \delta\text{-Al}_2\text{O}_3 \xrightarrow{1000^\circ\text{C}} \theta\text{-Al}_2\text{O}_3 \xrightarrow{1200^\circ\text{C}} \alpha\text{-Al}_2\text{O}_3. \quad (2)$$

An important problem of α-Al_2O_3 production is the high temperature of its formation (more than 1100°C), as well as the sintering temperature of corundum ceramics (more than 1500°C). Methods based on the use of aqueous reaction medium for γ-$Al(OH)_3$ or γ-AlOOH treatment are currently known. They allow for the synthesizing of single-phase α-Al_2O_3 in an autoclave at a temperature of 380–450°C [6, 7]. The decisive role in reducing the temperature of corundum formation under such conditions is played by the interaction of solid starting substances with water molecules from the reaction medium. This results in dehydroxylation of γ-$Al(OH)_3$ with the transition to γ-AlOOH and its transformation into α-Al_2O_3.

In the field of ceramics technology, a new approach known as the cold sintering process (CSP) has been actively developed during the last decade [8]. Processes similar to those occurring with oxide and hydroxide powders in aqueous medium in a closed reactor (autoclave) are realized, in order to obtain dense or porous ceramic materials. The CSP requires a mold equipped with a heater in which feedstock in the form of powder and the "liquid phase" are placed. The most often used liquids are water, aqueous solutions of acids or alkalis, as well as hydroxides or salts, including their hydrates with a low melting point [9]. CSP is carried out under applied mechanical pressure (usually up to 500 MPa) and isothermal holding at temperatures below 500°C from several minutes to several hours. The lowering of the sintering temperature of ceramics under such conditions when compared to conventional sintering is based on the interaction of the initial powder particles with the liquid phase. The literature considers various possible mechanisms of ceramic microstructure formation during CSP. This includes partial dissolution of solid matter, ion transport through the liquid phase and deposition in energetically more favorable regions (dissolution–precipitation mechanism) [10], as well as mass transfer and coalescence of particles due to increased mobility of the crystal structure under conditions of quasi-equilibrium hydroxylation (in the case of water-based liquid phase) [11, 12]. An important role in the CSP process is attributed to surface diffusion [13]. As of the present time, more than one hundred different ceramic materials fabricated using the CSP method have been reported.

The possibility of lowering the sintering temperature by means of CSP is of particular interest in refractory compositions, including corundum. Given the demand for corundum ceramics in a range of applications, the development of CSP technology for this material will provide significant energy savings in its production. Only a few papers have appeared in literature since 2020 reporting on the successful production of α-Al_2O_3 ceramics using the CSP method. Kang *et al.* [14] succeeded in producing corundum ceramics in two stages: by CSP of a mixture of α- and γ-Al_2O_3 in glacial acetic acid at 300°C and 300 MPa for 1 h and subsequent calcination of the samples in air at temperatures above 1250°C. In [15], CSP of γ-$Al(OH)_3$ was realized in a spark plasma sintering unit, in order to obtain γ-AlOOH (boehmite) ceramics in the presence of water (450°C, 70 MPa, 20 min). The calcination of the obtained boehmite in air led to the formation of α-Al_2O_3 ceramics with a porosity of about 60%. This work also shows the transformation of boehmite powder into α-Al_2O_3 under the same conditions, but the obtained corundum sample did not have the satisfactory transport strength.

The fabrication of porous ceramics based on boehmite (porosity of about 38%) from γ-$Al(OH)_3$ was described by Yamaguchi *et al.* [16]. They performed CSP at 250°C and 270 MPa with the addition of water as a liquid phase. The authors note that the transition of aluminum hydroxide to oxohydroxide is accompanied by a significant increase in the porosity of the material. Based on the data provided in literature on the sequence of transformations of γ-$Al(OH)_3$–γ-AlOOH–α-Al_2O_3 in aqueous medium, including at CSP, the possibility of direct production of porous ceramics α-Al_2O_3 with the help of CSP may be assumed. The direct transition of another modification of aluminum oxyhydroxide, α-AlOOH diaspore, into corundum upon heating to a temperature of about 500°C is also known [17]. In the few works concerning the fabrication of alumina ceramics from natural diaspore, a high porosity of the materials obtained is noted [18]. Thus, the diaspore-corundum transition can be considered as a possible basis for the process of obtaining corundum ceramics at reduced temperature, in particular during CSP.

The aim of this work is to obtain porous corundum ceramics by means of CSP of different phase modifications of aluminum oxohydroxide—boehmite γ-AlOOH and diaspore α-AlOOH, as well as to study the phase and structural properties of the obtained materials and to evaluate their water permeability.

2. EXPERIMENTAL

2.1. Synthesis of aluminum oxohydroxide (α- and γ-AlOOH)

The synthesis of α-AlOOH (diaspore) was carried out in two stages: (1) obtaining the precursor—aluminum oxide—by precipitation from aqueous solution and (2) treatment of the precursor in water vapor. Aluminum nitrate octahydrate $Al(NO_3)_3 \cdot 9H_2O$ (high purity, *Lenreactive*, Russia) and aqueous ammonia solution NH_4OH (particularly high purity, *IREA* experimental plant, Russia) served as starting substances for precursor synthesis. Equal volumes of aqueous solutions of

Table 1. Production conditions of ceramic samples using the CSP method (BCS—boehmite cold sintering, DCS—diaspore cold sintering)

Ceramic sample	Raw material composition (powder)	CSP conditions	Calcination conditions
BCS	γ-AlOOH 95 wt %, α-Al_2O_3 5 wt %	450°C, 220 MPa, 30 min	–
DCS	α-AlOOH 75.3 wt %, γ-AlOOH 24.7 wt %		–
DCS-600	α-AlOOH 75.3 wt %, γ-AlOOH 24.7 wt %		600°C, 1 h

$Al(NO_3)_3$ prepared with a concentration of 0.15 M and NH_4OH with a concentration of 0.30 M were added to 400 mL of distilled water under continuous stirring and at a temperature of 75°C. The resulting precipitate was left to age at the same temperature for 3 h. It was then filtered and washed with a large volume of distilled water until the wash water was neutral. The filtered precipitate was air dried at 50°C for 12 h. The resulting powder was calcined in air at 1000°C for 30 min. The precursor synthesized in this way was placed in a container with a lid made of Teflon and placed inside a laboratory autoclave (*Private entrepreneur Vitsukaeva S.N.*, Russia) with a volume of 17 mL. 2 mL of distilled water was poured in advance at the bottom. The hermetically sealed autoclave was heated to a temperature of 200–300°C at a rate of 70°C/h in a SNOL-3.5,3.5,3.5/5-I1 drying cabinet (*NPF Thermiks*, Russia) and kept at the assigned temperature for up to 260 h. During the treatment, the precursor was separated by container walls from water in the liquid state, while in contact with the vapor phase. The equilibrium vapor pressure of water was 3.97 MPa. The autoclave was then cooled by placing the bottom of the autoclave in cold water outside the desiccator. The synthesized sample was removed from the autoclave and dried in air at 50°C for 5 h.

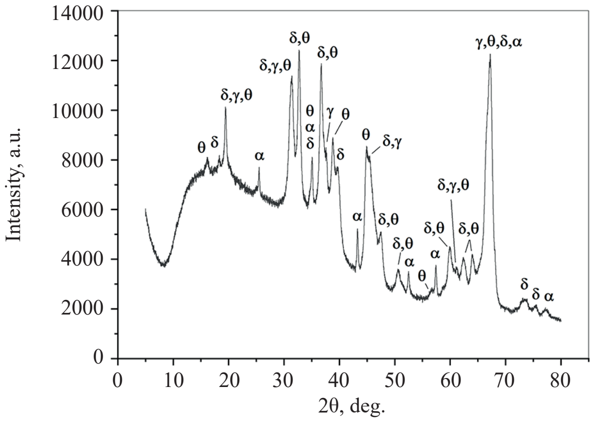
Aluminum hydroxide (hydrargillite) γ-$Al(OH)_3$ (MD brand, *Pikalevsky Alumina Plant*, Russia) served as a starting substance for the synthesis of γ-AlOOH (boehmite) in water vapor. When treating γ-$Al(OH)_3$ in water vapor, equipment and procedures similar to those described above for treating the α-AlOOH precursor were used. The temperature of isothermal soaking in water vapor was 270°C, the equilibrium pressure was 5.50 MPa, and the duration was 14 h. The product obtained was air dried at 50°C for 5 h.

2.2. Ceramics fabrication using the CSP method

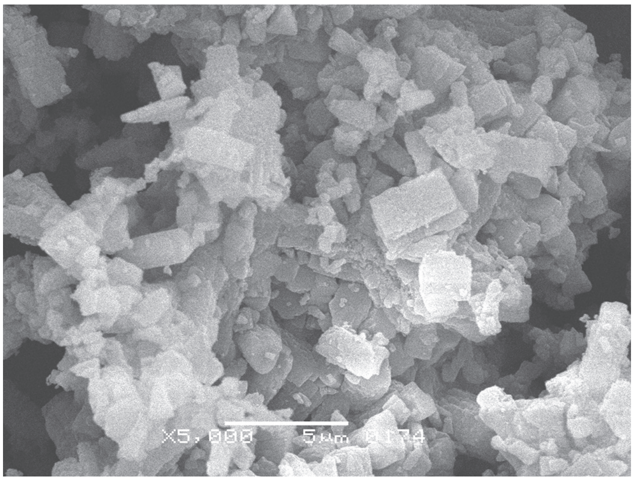
The laboratory setup for CSP in aqueous medium consisted of the following: an IP-1250 M-auto hydraulic press (*Plant of Testing Instruments and Equipment*, Russia); a custom-made steel mold with two composite punches; and a ring heater equipped with a temperature controller Thermodat-17E6 (*NPP Sistemy Kontrolya*, Russia) with two thermocouples. In order to prevent heat dissipation into the press plates and the environment, custom-made heat-insulating bars made of gabbro-diabase were placed under the punches. The unit was placed in a casing made of aluminum foil (*RUSAL – Sayana Foil*, Russia) and refractory kaolin wool MKRV-200 (*Teplopromproekt*, Russia), in order to reduce heat exchange with the environment. Sealing of the working volume of the mold was achieved by using sheets of graphite paper (*NPO Unikhimtek*, Russia) and copper sealing rings (*Metallinvest Corporation*, Russia) at the joints of the mold elements. In order to induce nucleation of the latter at CSP, 5 wt % of aluminum oxide α-Al_2O_3 (corundum) was preliminarily added into γ-AlOOH powder. 1 g of synthesized aluminum oxohydroxide was placed in the working volume of the CSP mold, 0.2 mL distilled water was poured in, and then a mechanical pressure of 220 MPa was applied to the assembled mold. Heating was carried out at a rate of 10°C/min to a temperature of 450°C, and isothermal holding was 30 min. It has previously been shown that the formation of corundum phase from boehmite powder occurs at 450°C under CSP conditions [15]. The mechanical pressure was released, the mold was cooled naturally, and the ceramic sample was removed from the mold. The ceramic sample made from α-AlOOH powder was calcined in air at 600°C for 1 h. The conditions of ceramic sample fabrication are summarized in Table 1.

2.3. Methods of research of synthesized powders and ceramics

The X-ray phase analysis of synthesized powders and fabricated ceramics was carried out using a PowDiX 600 diffractometer (*LINEV ADANI*, Belarus, 2022) by means of monochromatic Cu-$K\alpha_1$ radiation with λ = 1.5406 Å (0.02-mm Ni filter) on a diffracted beam (30 kV, 10 mA). The θ–2θ, continuous imaging was carried out in the



(a)



(b)

Fig. 1. X-ray diffraction pattern (a) and SEM image (b) of Al_2O_3 powder synthesized by precipitation followed by calcination at 1000°C (Greek letters α, γ, δ, θ indicate the peaks of the corresponding Al_2O_3 modifications)

interval $5° < 2\theta < 80°$ with a step of 0.01° and a speed of 3.5°/min. Qualitative phase determination was performed using the ICDD PDF powder database[1], and quantitative analysis was performed using the following formula for calculating concentrations C (formula 3):

$$C = \frac{I_{\max i}}{\Sigma_j I_{\max j}} \times 100\%, \quad (3)$$

wherein $I_{\max i}$ is the reflex intensity of i-phase with relative intensity of 100%, and $\Sigma_j I_{\max j}$ is the sum of reflex intensities with relative intensity of 100% for all phases in the studied sample. The morphology of the synthesized powders and the microstructure of the ceramic chips were investigated using a JEOL JSM 6380 scanning electron microscope (SEM) (*JEOL*, Japan). The thermal analysis of the powder sample was carried out in a gas flow (80 vol % air, 20 vol % argon) in the temperature range from 40 to 700°C at a heating rate of 10°C/min on a Netzsch STA 449C Jupiter thermal analyzer (*Netzsch*, Germany).The integral structural characteristics of ceramic samples (density and open porosity) were measured using the kerosene saturation method in accordance with GOST 2409-2014[2]. In order to determine the permeability of ceramics, the time required for 100 mL of distilled water to flow through the ceramic sample at a pressure drop of 0.09 MPa was measured. The permeability P was calculated according to the following formula (4):

$$P = \frac{V}{St\Delta p}, \quad (4)$$

wherein V is the volume of liquid passing through the membrane of area S for a certain period of time t at differential pressure Δp.

3. RESULTS AND DISCUSSION

3.1. Characteristics of synthesized aluminum oxohydroxide powders

The synthesis of the precursor for α-AlOOH powder production was accompanied by the following chemical transformations in solution (formulas (5) and (6)), as well as during calcination (formula (7)):

$$Al(NO_3)_3 + 3NH_4OH \rightarrow Al(OH)_{3\ sol} + 3NH_4NO_3, \quad (5)$$

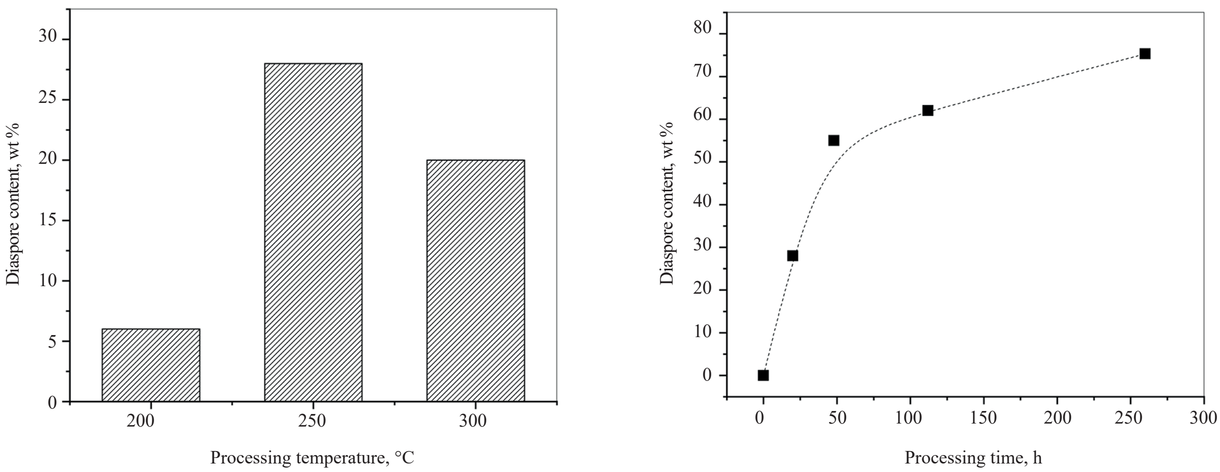
$$Al(OH)_{3\ sol} \rightarrow AlOOH_{\ sol} + H_2O, \quad (6)$$

$$2AlOOH_{\ sol} \rightarrow Al_2O_{3\ sol} + H_2O. \quad (7)$$

The slow mixing of solutions of $Al(NO_3)_3$ and ammonia produces aluminum hydroxide precipitate according to Eq. (5), which in the aging process under hot solution transforms into γ-AlOOH boehmite according to Eq. (6) [19]. The calcination of the precipitate obtained in air at 1000°C leads to the formation of aluminum oxide according to Eq. (7). In terms of phase composition, the powder obtained is a mixture of transitional modifications of aluminum oxide (γ-, δ-, and θ-Al_2O_3) and corundum (α-Al_2O_3) (Fig. 1) formed during thermal decomposition of boehmite. The heterogeneity of the phase composition is reflected in the morphology of the product. The

[1] ICDD PDF2 database (https://www.icdd.com/pdf-2/). Accessed March 29, 2024.

[2] GOST 2409-2014. Interstate Standard. Refractories. Method for determination of bulk density, apparent and true porosity, water absorption. Moscow: Standartinform; 2014. https://files.stroyinf.ru/Data2/1/4293767/4293767558.pdf. Accessed March 29, 2024.



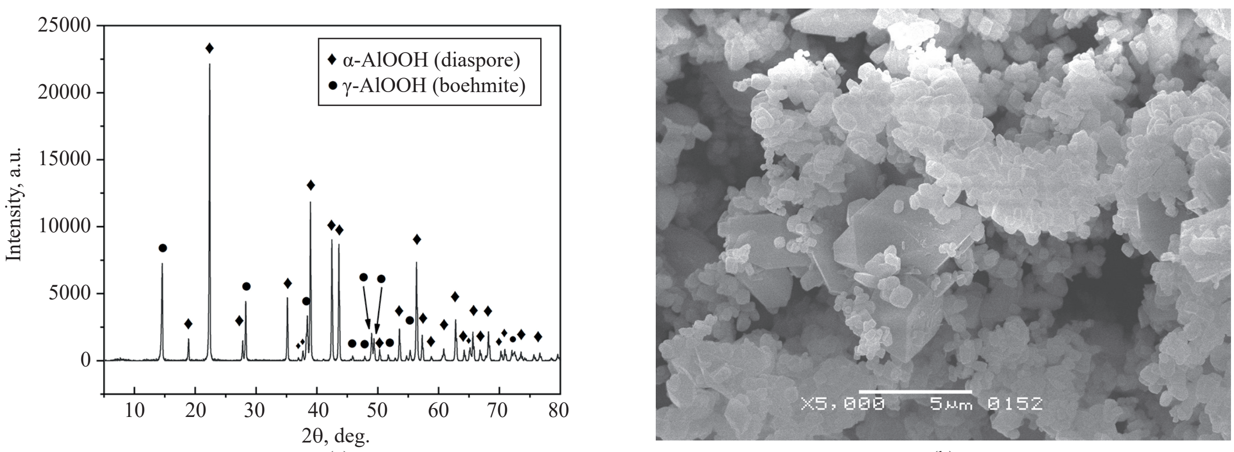
(a) (b)

Fig. 2. Diaspore contents after the treatment of alumina in a water vapor atmosphere: (a) 20 h of treatment at different temperatures; (b) different duration of a treatment at 250°C and an autogenous pressure of 3.97 MPa

SEM image (Fig. 1) shows particles of different shapes (rounded, prismatic, irregular) and sizes from 80 nm to 4.6 µm.

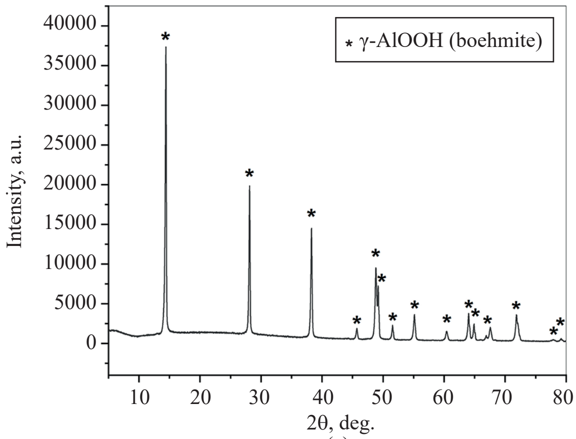
As a result of the treatment of the synthesized aluminum oxide powder in a water vapor atmosphere, the formation of a mixture of aluminum oxohydroxides: diaspore α-AlOOH and boehmite γ-AlOOH can be observed. The rate of diaspore accumulation in the system shows sensitivity to the process temperature. As a result of the incubation of aluminum oxide in water vapor at temperatures of 200, 250 and 300°C for 20 h, the formation of the largest amount of diaspore (28.1 wt %) can be observed at 250°C (Fig. 2a). The preparation of boehmite under hydrothermal conditions at temperatures from 150 to 240°C and equilibrium water vapor pressure from amorphous aluminum oxide has been described in the literature [20–22]. The formation of synthetic diaspore was observed under harsher hydrothermal conditions, at temperatures from 300 to 450°C, and pressures of 6.0 to 34.5 MPa when α-Al_2O_3 powder was treated for more than 72 h [23, 24]. At the same time, the mixture of α- and γ-modifications of Al_2O_3 passes into diaspore through the formation of the boehmite phase [23]. The formation of oxohydroxides from different modifications of aluminum oxide in water vapor most probably occurs with the preservation of the type of close-packing in crystals: hexagonal at the transition of α-Al_2O_3 into diaspore and cubic at the transformation of γ-Al_2O_3 into boehmite. The transition of boehmite into diaspore is associated with a change in the type of close-packing in the crystal. This may explain the slow accumulation of α-AlOOH in the reaction mixture during its prolonged treatment (more than 48 h, Fig. 2b).

After 260 h of treatment of the synthesized aluminum oxide in water vapor at 250°C, the reaction mixture contains 75.3% diaspore and 24.7% boehmite (Fig. 3).

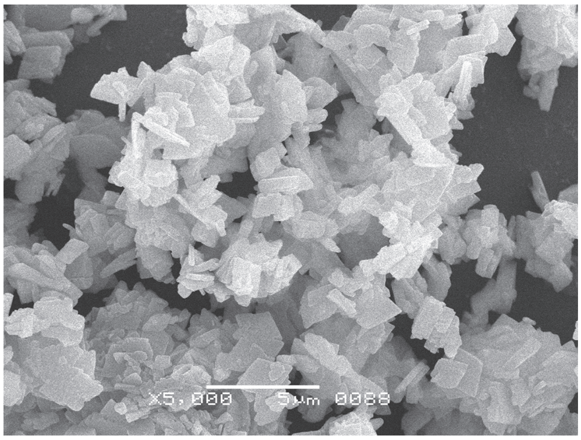


(a) (b)

Fig. 3. X-ray diffraction pattern (a) and SEM image (b) of diaspore powder synthesized in a water vapor at 250°C and an autogenous pressure of 3.27 MPa for 260 h



(a)



(b)

Fig. 4. X-ray diffraction pattern (a) and SEM image (b) of boehmite powder synthesized in a water vapor at 270°C and an autogenous pressure of 5.50 MPa for 14 h

Two types of particles can be distinguished in the obtained powder: strongly agglomerated particles of 0.3–1.4 μm in size, as well as large crystals growing among them, up to 7 μm in size, possessing smooth faces. The appearance of large crystals is a consequence of secondary recrystallization in the system.

As a result of treatment in water vapor of γ-$Al(OH)_3$ hydrargyllite powder at 270°C and equilibrium pressure of 5.50 MPa, a single-phase boehmite with predominantly lamellar particle shape was formed (Fig. 4). The particle size in the sample ranges from 0.5 to 2.9 μm. Such particle shape is also known for boehmite synthesized under classical hydrothermal conditions (in solution) [25, 26].

3.2. Structure and properties of corundum ceramics fabricated using the CSP method

CSP of synthesized powders of α- and γ-oxohydroxides of aluminum, carried out at a temperature of 450°C and applied mechanical pressure of 220 MPa for 30 min, led to the formation of transport-strength ceramic samples. The phase analysis of ceramics made from powder consisting of a mixture of 75.3% diaspore and 24.7% boehmite showed that the phase transition of boehmite to diaspore is completed during the CSP (Fig. 5a). The grains in the material obtained (Fig. 5c) are predominantly close in shape and size to the large crystals formed as a result of secondary recrystallization in the initial powder (Fig. 4). During CSP, single large crystals present in the powder served as nuclei of secondary recrystallization, leading to a significant increase in the average grain size of ceramics when compared to the original particles (from 0.94 to 2.13 μm).

Thermal analysis of the diaspore ceramics revealed an endothermic effect at around 524°C accompanied by a mass loss of 13.97% (Fig. 5b). The data obtained on the decomposition of α-AlOOH corresponds well with that previously known from the literature for diaspore powder [17]. At the same time, the total mass loss of the diaspore ceramic sample when heated to 700°C was 14.63%, which corresponds to the mass fraction of water released from aluminum oxohydroxide during the transition to Al_2O_3 (15%). Based on these data, the diaspore ceramics were calcined in air at 600°C for 1 h, leading to its complete conversion to α-Al_2O_3 corundum (Fig. 5a). The microstructure of the corundum ceramics retained the appearance of grains observed in the diaspore ceramics (Fig. 5d), while their average size increased to 2.78 μm.

CSP of boehmite powder in the presence of 5 wt % corundum resulted in the formation of single-phase α-Al_2O_3 ceramics (Fig. 6). The SEM image of the sample chip shows that the material has a developed pore space, and consists of isometric grains with sizes ranging from 0.42 to 4.23 μm with an average value of 1.42 μm. The grains have a hexagonal shape characteristic of corundum.

Corundum ceramics obtained from aluminum oxohydroxide powders of different phase composition have close values of relative density in the range of 46–48% (Table 2). At the same time, in the sample achieved in one stage CSP from boehmite powder (BCS), there is practically no closed porosity (5.9%), and open pores account for about half of the material volume (47.9%). The DCS-600 sample made from diaspore-containing powder has lower open porosity (39.0%). It should be noted that the DCS sample had only 9.0% open porosity before calcination in air. The calcination

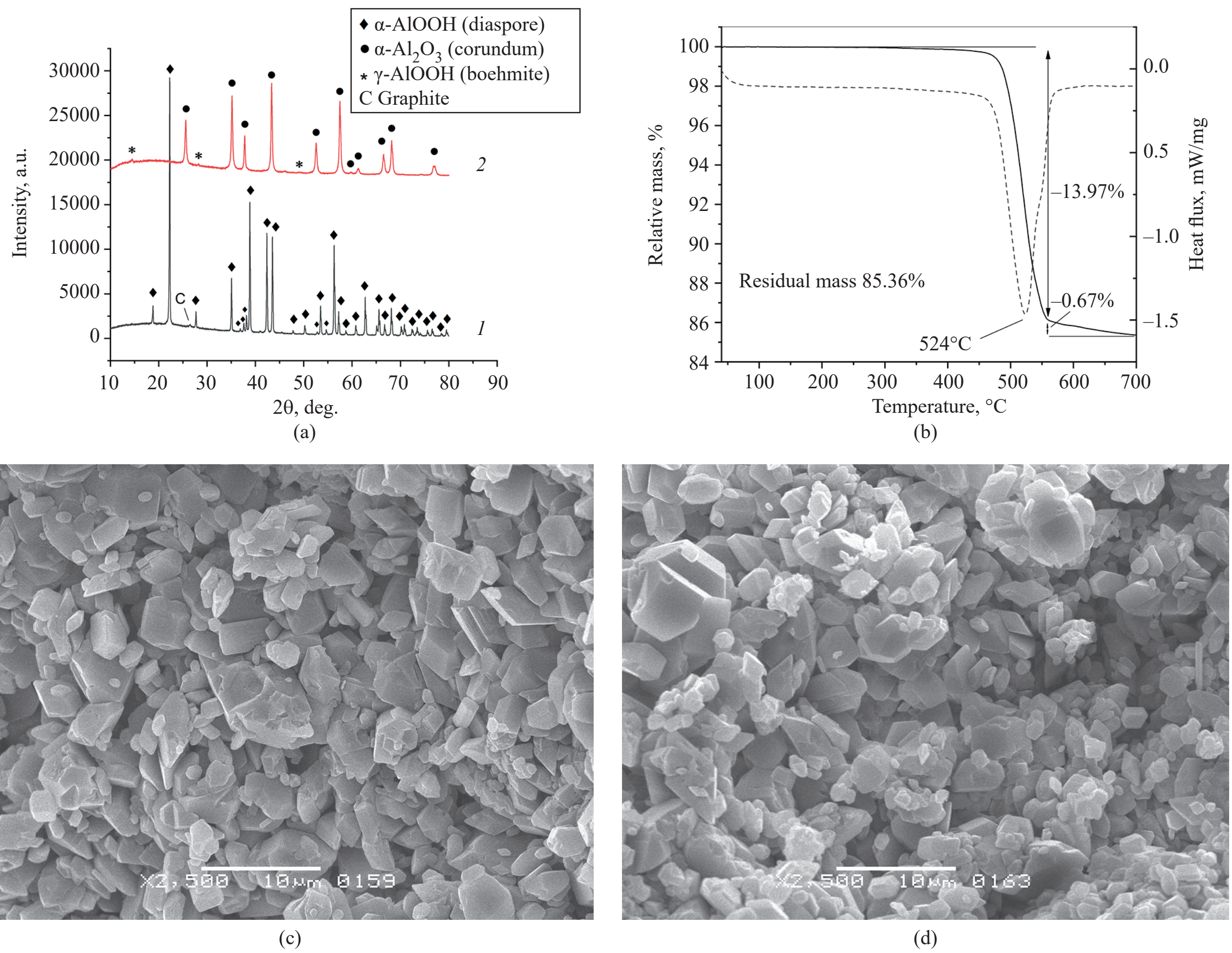


Fig. 5. Study of the ceramics obtained from diaspore powder: (a) X-ray diffraction patterns of the samples manufactured by CSP (*1*) and CSP with the following calcination at 600°C (*2*); (b) thermal analysis of the sample after the CSP; (c) SEM image of the sample after CSP; (d) SEM image of the sample after CSP with the following calcination at 600°C

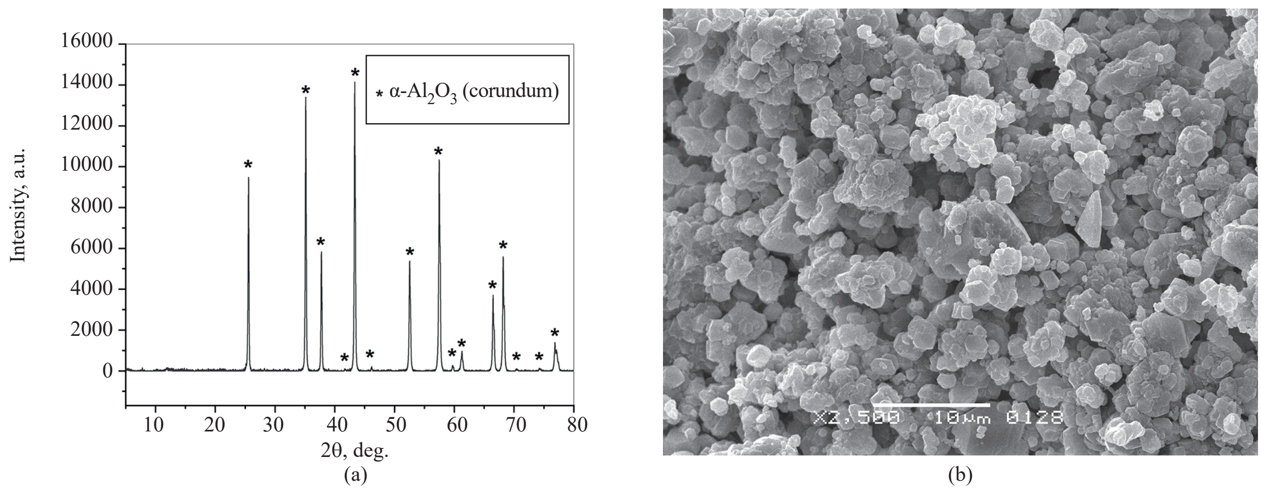


Fig. 6. X-ray diffraction pattern (a) and SEM image (b) of ceramics obtained by a cold sintering processing of a boehmite powder

Table 2. Integral structural characteristics and permeability of corundum ceramics fabricated using the CSP method

Ceramic sample name	Relative density, %	Open porosity, %	Permeability, L/(m^2·h·bar)
BCS	46.2	47.9	5370
DCS-600	47.2	39.0	5020

had a negligible effect on the closed porosity, an increase from 12.3 to 13.8% was observed. Taking into account the different theoretical density of boehmite and diaspore (3.08 and 3.38 g/cm^3, respectively), we can conclude that the decisive role in the formation of open porosity is played by the phase transition of oxohydroxides into α-Al_2O_3, which has a higher theoretical density (3.96 g/cm^3).

The values of open porosity observed in the fabricated corundum ceramics are comparable to those reported in the literature for ceramic substrates made of α-Al_2O_3 and intended for the fabrication of filter membranes (30–40% of open porosity) [27, 28]. The permeability of porous materials obtained in the present work for pure water is more than 5000 L/(m^2·h·bar) (Table 2). It is consistent with the value of open porosity. Corundum ceramics made of boehmite powder have a higher level of open porosity and permeability than those of diaspore ceramics. Similar permeability values have been reported by a number of authors for α-Al_2O_3 ceramic filter membranes. For example, in [29] corundum substrates with a permeability of 4000 L/(m^2·h·bar) were immersed in a suspension of α-Al_2O_3 powder followed by drying and sintering, in order to obtain single-layer membranes with a permeability of about 1000 L/(m^2·h·bar). Similarly, Naseer *et al.* [30], using less permeable substrates (614 L/(m^2·h·bar)), produced single-layer membranes with a permeability of 388 L/(m^2·h·bar) and bilayer membranes with a permeability of 311 L/(m^2·h·bar). By filtering a suspension through a substrate with a permeability of 4700 L/(m^2·h·bar), a membrane with a permeability of 550 L/(m^2·h·bar) was obtained in [31]. In other studies, microfiltration membranes composed entirely of corundum possessed permeabilities for pure water ranging from 2150 to 8000 L/(m^2·h·bar) [27, 28, 32]. In comparison with literature data, the permeability of porous ceramic materials obtained in this work allows us to consider the possibility of their use as components of filtration systems. Depending on the purpose of the system (from macro- to nanofiltration), such materials can be promising both for independent application, and as substrates for membranes of finer water purification. A significant advantage of such porous corundum ceramics over the currently known analogues is a high level of energy efficiency, expressiveness and the low labor intensity of the method of its manufacture: CSP of aluminum oxohydroxides, including widely available boehmite powder. The further development of this technology of porous corundum ceramics involves the study of the structure of its pore space, mechanical properties and the possibility of manufacturing multilayer materials with variable porosity.

4. CONCLUSIONS

The present work shows the possibility of using the CSP method for the fabrication of porous corundum ceramics based on aluminum oxohydroxides and water. The CSP of boehmite γ-AlOOH powder with the addition of α-Al_2O_3 corundum in the presence of 20 wt % water at a temperature of 450°C and an applied mechanical pressure of 220 MPa for 30 min allows single-phase α-Al_2O_3 ceramics with an open porosity of 47.9% to be directly obtained. At CSP of a mixture of α-AlOOH diaspore powders (75.3%) and boehmite (24.3%), single-phase diaspore ceramics are formed. These completely transform into corundum ceramics with an open porosity of 39.0% as a result of calcination in air at 600°C for 1 h. Ceramics made of aluminum oxohydroxides have a permeability level for pure water of more than 5000 L/(m^2·h·bar), allowing us to consider them as a material promising for use in filtration systems. The use of CSP provides an increase in the efficiency of porous corundum ceramics production, when compared with the traditional technology due to energy saving and the absence of the need to use auxiliary substances (binders, pore-forming agents, etc.).

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Authors' contributions

A.A. Kholodkova—general management, scanning electron microscopy, permeability measurements, and writing the text of the article.

A.V. Smirnov—scientific editing, conceptualization.

M.V. Kornyushin, L.A. Arbanas—experiments on the cold sintering.

V.E. Bazarova—density and porosity measurements.

L.A. Arbanas, A.N. Khrustalev—X-ray diffraction analysis.

A.V. Shumyantsev, S.Yu. Kupreenko—thermal analysis.

Yu.D. Ivakin—conceptualization, synthesis of aluminum oxohydroxides.

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