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Optimization of the structure and properties of foam-glass ceramics

K.S. Ivanov
Earth Cryosphere Institute SB RAS, Tuymen, Russia

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Abstract. The basic properties of foam-glass ceramics — a porous inorganic material are optimized in the study. This material is used for thermal insulation of various engineering structures: foundations of buildings, roads and railways, pipelines, etc. The main raw material components of the material are sodium hydroxide and opal-cristobalite rocks: diatomite, tripoli, opoka. The mixture of components is subjected to firing and foams with the formation of a porous structure with vitreous and crystalline phases. A significant impact of two different ways of preparing the batch on the basic properties of the material was studied and analyzed. In the first method, the batch was obtained as a suspension with a high water content, which was subjected to mechanical activation in a vibratory mill. In the second method, the batch was obtained by pushing a mixture of components through calibrated holes with the help of a screw auger. Thus, the batch was an extruded tough-plastic granular mass with lower water content. As a result, the formation of a heterogeneous structure of the samples and the presence of dense non-foamed inclusions, leading to an increased average density of the material were established in the first method. The extruded batch in the second method was foamed more evenly without stratification; thereby the average density of the samples was reduced. An additional reduction in the average density of the samples by 17% due to the intensification of dissolution of silica during the autoclave treatment of extruded batch was observed. The extrusion method is recommended for the production of foam-glass ceramics in granular form, which contributes to the saving of expensive sodium hydroxide.

1. Introduction

Foam-glass ceramics is an inorganic insulating material of cellular structure. The advantages of foam-glass ceramics include the high prevalence of the raw material base — these are opal-cristobalite and zeolite-containing rocks [1, 2], low thermal conductivity, water resistance, non-flammability and also a wide scope of application. Foam-glass ceramics can be obtained both in block form (plates, shells, segments) and in the form of granules. The material can be used for thermal insulation of enclosing structures of residential and industrial buildings [3]. Another field of application for granulated foam-glass ceramics is the thermal insulation of foundations of engineering structures in areas with seasonal freezing of soils to protect against frost heaving. Such structures include: buildings with low-depth foundations, roads, railways, pipelines, etc. [4–5].

The disadvantages of the material include the need to use expensive sodium hydroxide, therefore reducing its content, taking into account the preservation of the functional properties of foam-glass ceramics, is an actual scientific and technical problem. Another limiting factor of industrial production of foam-glass ceramics is the lack of a unified technology of the material synthesis.

Opal-cristobalite rocks which include diatomite, tripoli, and flask are rich in amorphous forms of silica. The key process in the synthesis of foam-glass ceramics is the dissolution amorphous silica in the alkalis. The mixture of the rock and alkaline solution is subjected to firing and foams at a temperature of 800–900 °C. Foaming of the mixture during the physicochemical interaction in the system SiO_2-Na_2O-H_2O occurs in two stages. At the first stage, when components are mixed, the formation of hydrated alkali silicates occurs. At the second stage, during firing, the batch passes into the pyroplastic state with simultaneous dehydration of hydrated alkaline silicates and the subsequent polycondensation of silicon-oxygen anions [6–9]. As a result, a

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«silicate foam», containing both the vitreous phase and the crystalline phase, introduced with the insoluble residue of the opal-cristobalite rock, is formed.

The advantage of this method is the fact that an inorganic thermal insulation material is synthesized in one stage and the intermediate process of sintering the granular glass is excluded [10–12]. Unlike thermal insulation materials obtained by hot-foaming method [13, 14], commercial liquid glass is not used in described method. In addition, the usage of waste glass, that is necessary in classical foam-glass production [15, 16], is not required in foam-glass ceramics technology.

The methodology of mixing the opal-cristobalite rock and alkali can have a significant impact on the structure formation and functional properties of foam-glass ceramics. On the one hand, the mixing process should be accompanied by chemical interaction between the components with the formation of hydrated sodium silicates. On the other hand, opal-cristobalite rocks have a high adsorption capacity, which makes it difficult the batching process. Thus, it is obvious that the procedure of the preparing the mixture is a complex physicochemical process that has not been adequately covered in the scientific literature. For example, in studies of recent years, the methodology of mixing components is described insufficiently, but only their ratios are given [1, 6–8].

Taking into account the above features of mixing the components, the studies examined two different ways of the batching process. The first (wet method) consists in obtaining a suspension with a relatively high water content from milled opal-cristobalite rock, sodium hydroxide and water. It can be assumed that in the liquid phase a better quality of the reaction between sodium hydroxide and amorphous silica of opal cristobalite rock can be achieved. It should be noted that these batches in the form of liquid suspensions, are used in the preparation of various materials and binders and play a structure-forming role [17, 18]. The disadvantage of this method is the difficulty of further drying the batch before firing.

In the second embodiment, the continuous pushing of components through calibrated holes with the help of a screw auger, in one or more stages, is used (extrusion method). The process of interaction between sodium hydroxide solution and amorphous silica of the rock is intensified under the mechanical influence of the screw, resulting a tight-plastic batch with high homogeneity. Secondly, the processes of mixing components and granulation of the batch are combined in one operation. Compared to the wet method, the granulated mixture has a low water content, which simplifies its further drying, since in the wet method, the water content can reach 70 % or more, whereas in the extrusion method 16–18 %.

The purpose of the work was to study the influence of wet and extrusion methods of obtaining the batch on the structural features and basic properties of foam-glass ceramics.

2. Materials and experimental technique

2.1. Diatomite analysis

The opal-cristobalite rock was represented by diatomite with the following chemical composition, mas. %: SiO_2 — 76.71, Al_2O_3 — 7.33, Fe_2O_3 — 2.43, CaO — 0.46, MgO — 0.93, R_2O — 1.24, TiO_2 — 0.61, SO_3 — 0.72, loss of ignition — 9.57. The diatomite was dried to constant weight at 100 °C, powdered using a ball mill and sifted through a sieve with a mesh size of 0.16 mm. The structure of diatomite was studied using a Jeol JSM-6510A scanning electron microscope (Japan) and a DRON-6 diffractometer (Russia), a wavelength of 0.179 nm, Cu Kα-radiation, and a Fe-filter.

SEM image shown in Figure 1 suggests that diatomite is composed mainly of the remains of the shells of diatoms — fossilized algae, consisting of amorphous silica. The maximum size of shells is about 0.1 mm. A high content of the amorphous phase in the form of opal in the diatomite is indicated by the disperse reflex on the diffractogram of X-ray phase analysis in the range of angles of 18–26° (Figure 2a). The crystalline phases of diatomite were identified using the American Mineralogist Crystal Structure Database. The XRD pattern (Figure 2a) shows the presence of quartz (reflections at angles of 20.87°, 26.65°, 36.56°, 39.49°, 40.41° and 50.17°), montmorillonite (reflections at angles 19.60° and 34.67°), illite, having similar reflections to montmorillonite, as well as feldspar mineral albite (reflections at angles of 21.69° and 27.84°).

In order to quantify the content of soluble silica the diatomite was dissolved in NaOH solution with a concentration of 20 % in a mass ratio of solid and liquid phase equal to 1:3. To intensify the dissolution, the mixture was heated to a temperature of 90 °C and continuously stirred for 4 hours. By determining the concentration of SiO_2 in the resulting suspension using the method [19], it was established that diatomite contains 44.6 % of soluble silica in its composition. The insoluble mass of diatomite (55.4 %) falls on the clay minerals, feldspar and organic impurities.

Figure 3a shows TG, DTG and DSC curves of thermal analysis of diatomite. The endothermic effect on the DSC curve in the range of 70–200 °C corresponds to the removal of physically bound (adsorption) water from the sample. The clay minerals of diatomite are dehydrated at 490 °C, which is confirmed by the corresponding peak on the DTG curve. The exothermic effect at a temperature of 350 °C can be explained by

the burning out of organic impurities. The last endothermic effect on the DSC curve at 870 °C is not associated with a change in the sample mass and characterizes the structural changes of montmorillonite and illite, which is confirmed by the thermal analysis of these minerals [20]. Thus, the main phases of diatomite are opal, in the form of alkali-soluble shells of diatoms, quartz and clay minerals. The identified phases are in accordance with the given above chemical composition of diatomite.

2.2. Batch preparation process

The mass ratio between diatomite and dry NaOH in the experiments was assumed to be 9.1. Theoretically, this gives a molar ratio between the soluble silica of diatomite and alkali (in terms of Na_2O), equal to 5. In traditional liquid glass, this ratio, called the silicate module, is 3–4. A lower ratio, with the chosen methods of obtaining the material, is not economically justified. At a higher ratio the average density of foam-glass ceramics is decreased resulting in deteriorating its thermal insulation properties.

For comparison, in the studies of other authors, the mass ratio between diatomite and dry NaOH was taken to be 4.9 [6] and 4.0 [7], respectively, i.e. the mixture contained almost twice the amount of NaOH. Taking into account the chemical composition of diatomite and the accepted ratio between diatomite and NaOH, the theoretical total content of alkali oxides R_2O ($Na_2O + K_2O$) in foam-glass ceramics should be 11 %. At the same time, the foamed glass obtained from glass waste by adding NaOH as a foaming agent contained about 19 % R_2O in its chemical composition [16].

In the wet method of obtaining the batch diatomite, dry NaOH and water were taken in the weight proportion between the solid components and water equal to 1:2; the spreadability of the fluid batch according to Suttard viscometer was 80–90 mm. To intensify the interaction between diatomite and NaOH the batch was processed in a 300 ml vibratory mill, in which there were stainless steel balls with a diameter of 8 mm and a total weight of 250 g. The oscillation frequency, amplitude and duration of treatment were 1500 min^{-1}, 2 mm and 15 min, respectively.

The extrusion method of batching consisted in the primary mixing of components by hand in a steel spherical bowl, until a press powder with a moisture content of 17 % was prepared. Next, the press powder was processed in a laboratory extruder with the screw parameters: diameter and a screw step of 80 mm, rotation speed of 45 min^{-1}, maximum torque of 490 N·m. The granulating grid was made with 26 calibrated holes with a diameter of 5 mm. This parameter sets the diameter of the granules at the exit of the extruder and is the most optimal for the given parameters of the extruder. Under the influence of the screw, the mixture turned into a tight-plastic mass, which was pressed through the holes of the granulating grid as individual granules with a diameter of 5 mm, whose length was limited to 5–6 mm. To achieve the required plasticity, water was introduced into the mixture in the amount of 4–6 %.

The mixture was subjected to double extrusion, i.e. after the first passage through the granulating grid, it was returned to the extruder and extruded again. Increasing the rate of extrusion over two is impractical because of the sharp decrease in the plasticity of the batch due to the high adsorption properties of diatomite, which causes overheating and an emergency stop of the extruder. Mass homogeneity at single extrusion of the mixture was not provided. The average density of the raw grains obtained by the extrusion method was 1560–1630 kg/m^3.

2.3. Samples Preparation Technique

The effect of the metod of preparing the batch was evaluated after thermal foaming the samples according to their average density, porous structure and compressive strength. The main criterion was the average density that directly depends on NaOH consumption per unit volume of foam-glass ceramics. In addition, the value of average density affects the key characteristic of thermal insulation material – its thermal conductivity.

The mixture was subjected to drying to constant weight at 80 °C. Then the mixture was crushed to a fraction of 1–2.5 mm, poured into molds of heat-resistant steel and subjected to firing in a muffle furnace at 850 °C for 20 minutes. During firing, foaming and sintering of individual grains of the batch took place among themselves. Cooling of the molds took place together with the furnace, after which cubic samples with a rib length of 30 mm were cut out of the monolitic material. The average density of cubic samples was determined as the ratio of their mass to volume.

Depending on the processing method and the type of treatment, the batch was marked in accordance with Table 1. The W0 and E0 batches were foamed according to the above regime on a ceramic substrate, without pre-drying and grinding. In this case, the determination of the average density of the samples was carried out using the hydrostatic weighing method. In order to intensify the process of dissolution of silica and reduce the consumption of NaOH, the EAD batch was subjected to autoclave treatment at a water vapor pressure of 1.5 MPa for an hour and then dried and crushed.

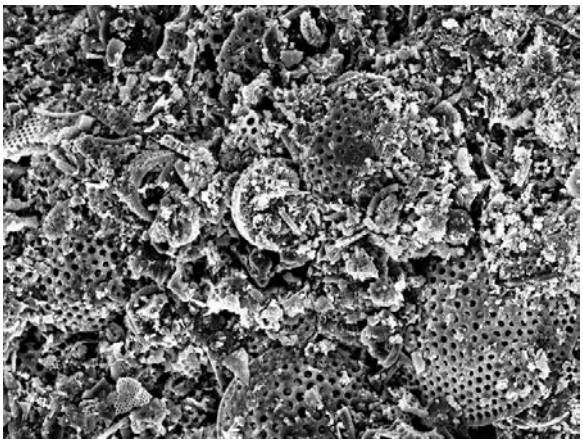


Figure 1. SEM image of diatomite, magnification x500.

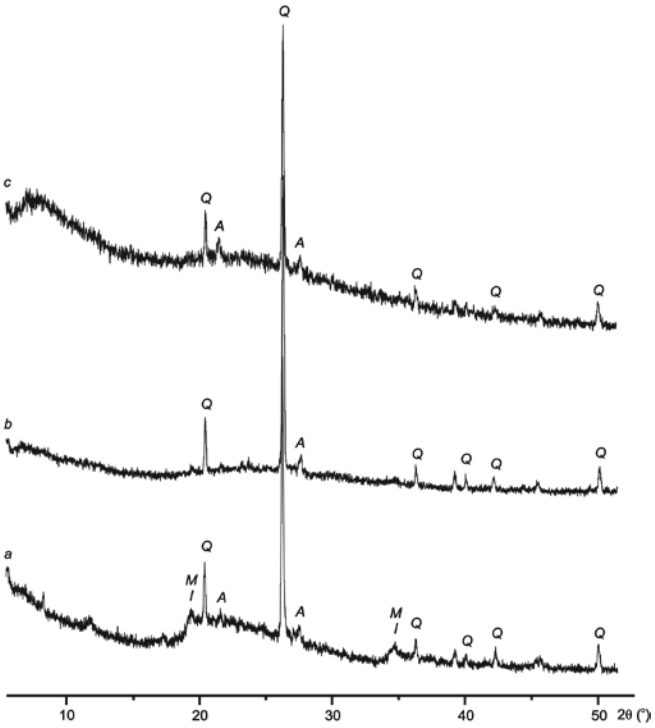


Figure 2. X-ray phase analysis: a – raw diatomite; b and c – batch WD heat treated at 100 and 800 °C respectively. A – albite, I – illite, M – ontmorillonite, Q – quartz.

3. Results and Discussion

To study the physicochemical processes during foam-glass ceramics firing thermal analysis of the WD batch (Table 1) was carried out (Figure 3b). Curves DTG and DSC shown in Figure 3b are characterized by endothermic effects in the form of a sharp peaks at 100 °C and a more extended effects (unlike the sample of raw diatomite in Figure 3a) in the range of 350–600 °C. In accordance with the TG curve, the second effect is accompanied by almost half of the total weight loss of the sample, which, apparently, is due to the dehydration of hydrated sodium silicates formed in the batch before. The last insignificant endothermic effect of the DSC curve at 760 °C in Figure 3b, is possibly associated with melting of the crystalline phase of $Na_6Si_8O_{19}$. The occurrence of this phase was diagnosed using X-ray phase and thermal analysis at heating commercial liquid glass to 750 °C, followed by decomposition of $Na_6Si_8O_{19}$ at 800 °C and the formation of a melt [21].

Table 1. Marking of batch depending on processing method and type of further treatment.

No.	Method of batch processing		Type of batch treatment	
1	W0	Wet	No treatment	Firing on a ceramic substrate
2	WD		Drying and crushing	
3	E0	Extrusion	No treatment	Firing in a steel mold
4	ED		Drying and crushing	
5	EAD		Autoclave, drying and crushing	
6	EA		Autoclave and drying	Firing on a ceramic substrate

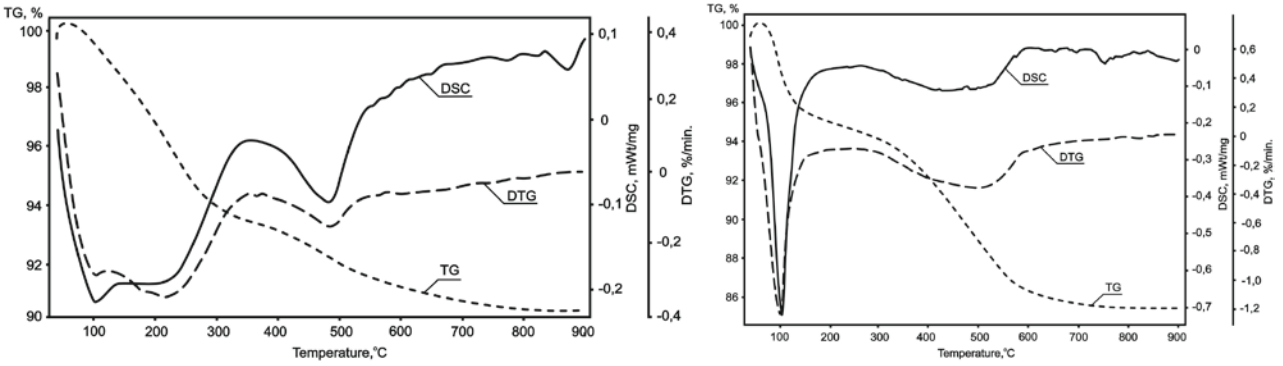


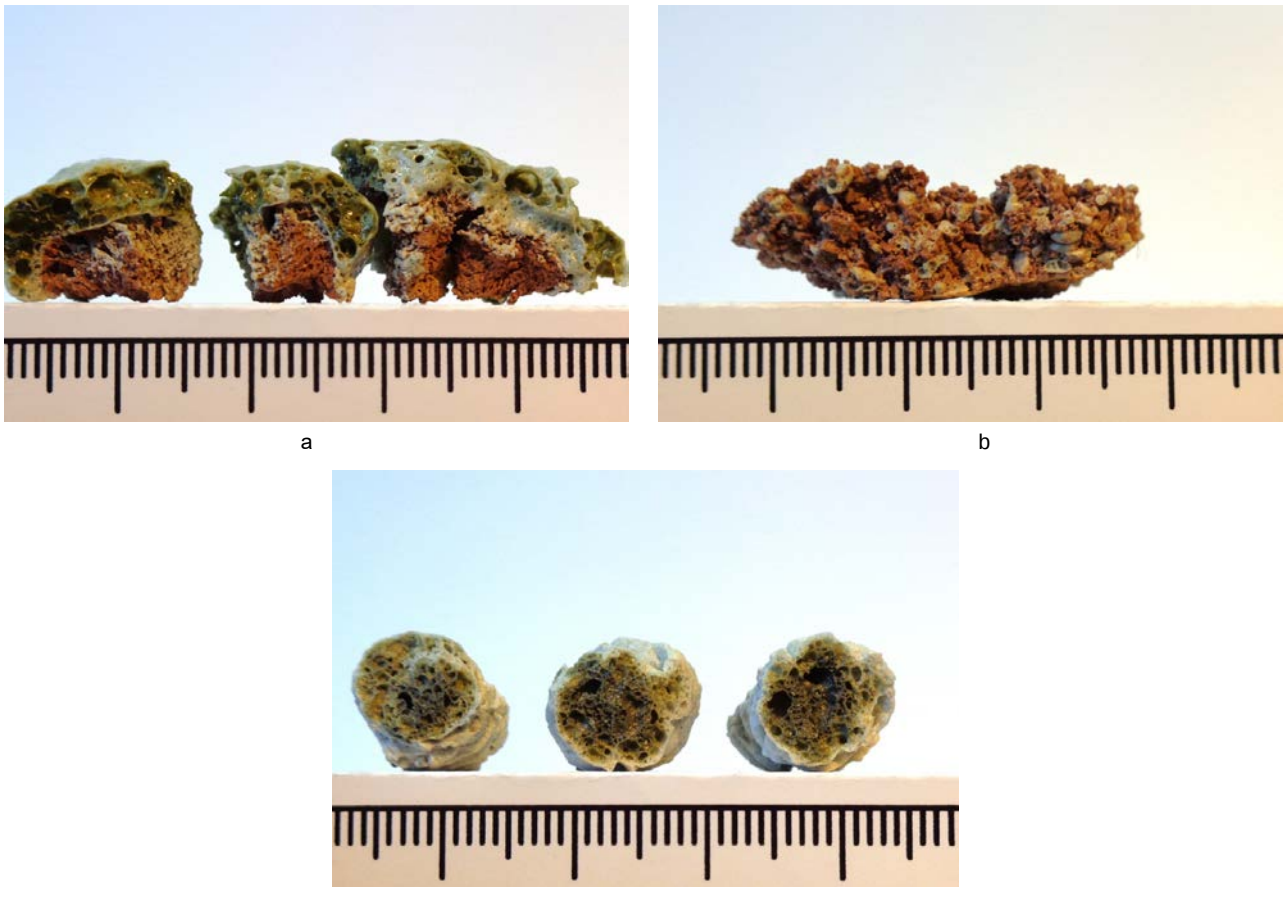
Figure 3. Results of thermal analysis: a — raw diatomite; b — batch WD.

X-ray phase analysis of the mixture depending on the heating temperature is presented in Figures 2b and 2c. The significant changes in crystalline component of ditomite under heat treatment ot the batch WD are not seen in comparison with the diffraction pattern of raw diatomite: Figures 2b and 2a respectively. The disappearance of the amorphous gallo in the range of angles 18–26° after drying at 100 °C, which is typical for the dissolution of silica phases of diatomite under the influence of NaOH, is seen in Figure 2b. The diffractogram in Figure 2b is characterized by the disappearance of the peaks of montmorillonite and illite, the structure of which is subjected to a significant restructuring under the influence of alkalis [22]. The presence of a glass phase at 800 °C is confirmed by the formation of gallo in XRD pattern shown in Figure 2c at angles of 8–13°. The last is consistent with the data other authors who performed x-ray phase analysis of liquid glass at 800 °C [21]. Thus, at the batch firing, physically bound water is removed at 100 °C, then the process of dehydration of sodium silicates starts at 350 °C and continues until the melt is formed at 750 °C resulting in foaming the composition.

The structure of the samples fragment obtained from the batches W0 and WD is shown in Figures 4a and 4b respectively. The stratification in height of the sample W0 foamed in a liquid form on a ceramic substrate is seen in Figure 4a. The top of the sample is represented by a foamed mass with a characteristic glass luster, and in the lower part a dense mass of red color prevails, in appearance resembling ceramics, which is unacceptable in the technology of thermal insulation material.

In order to eliminate negative stratification during firing, the batch W0 was subjected to preliminary drying. Unlike the sample W0 in Figure 4a, an obvious delamination is not observed in a fragment of the foamed sample WD presented in Figure 4b. However, among the foamed particles up to 3 mm in size, there are dense ceramic inclusions of red color. The latter indicates that the liquid batch had already been separated during its drying and the grains of insoluble residue were distributed in the batch after its grinding that is seen in Figure 4b.

The average density of the samples obtained from the batches W0 and WD was 640 and 720 kg/m^3 respectively, which is a relatively high value for a thermal insulation material. Obtaining a more viscous batch with lower water content is impractical because of the difficulty of mixing, and therefore, further research was focused on the mechanized method of obtaining the batch using extrusion.



a b c

Figure 4. Porous structure of foam-glass ceramics samples (scale is given in mm): a — batch W0; b — WD; c — E0.

The porous structure of the foamed samples in the form of granules obtained from the batch E0 is shown in Figure 4c. In comparison with the batches W0 and WD (Figures 4a and 4b, respectively) pores are distributed throughout the volume and delamination is not seen (Figure 4c). Consequently, obtaining the batch

as a tight-plastic mass with a low water content contributes to the uniform foaming of foam glass-ceramic samples, while the insoluble residue of diatomite is distributed in the mixture without further separation during firing process. The average density of samples from the batch E0 is 530 kg/m^3, which is 1.2 and 1.7 times lower in comparison with the batches W0 and WD, respectively. The volume density of the granules, taking into account their average size of 10 mm, is 290 kg/m^3. The volume water absorption and compressive strength of granules in the cylinder determined according to Russian State Standard GOST 9758-2012 “Non-organic porous aggregates for construction work. Test methods” were 6 % and 1.8 MPa, respectively. Accordingly to these properties the granules can be used as insulating layers in various engineering structures: for example, to protect the soils of the roads foundations from seasonal freezing and frost heaving [4].

From the batches marked as ED and EAD, cubic samples of foam-glass ceramic with an average density of 540 and 450 kg/m^3 and compressive strength of 7.4 and 3.4 MPa respectively, were obtained. Thus, the average density of the autoclave treated samples ED is decreased by about 17 %. The most likely explanation of the autoclave treatment effect is the formation of an additional amount of hydrated sodium silicates in the batch caused by intensification of the dissolving the silica phases. As a result, this can contribute to an increase in the amount of the liquid phase in the melt and give an additional amount of a pore-forming agent during the dehydration of sodium silicates.

In addition, due to the existence of known methods of hydrothermal synthesis of commercial zeolites from clays and alkalis [22], it is likely that they can be formed under the influence of pressure and steam. During firing, dehydration of artificial zeolites is likely, as a result they can participate in the process of foaming the material, reducing the average density. However, this is a theoretical assumption, since zeolites were not traced in XRD pattern of the batch WD (Figure 2b).

High glass phase content in the samples E0 is proved by a characteristic glass luster that observed in Figure 4c. Along with this, during firing, greenish color sometimes turning into black is seen in the samples W0 and E0 presented in Figures 4a and 4b respectively. The reason for this may be the formation of ferrous oxide as a result of the reduction of Fe_2O_3. The melting of the outer surface of the sample can prevent the penetration of oxygen and contribute to the creation of a reducing environment due to the organic compounds contained in diatomite [23].

The EA batch was foamed on a ceramic substrate and glass-ceramic samples in the form of granules were obtained. In accordance with Russian State Standard GOST 9758-2012, the samples had the following properties: an average grain size of 10 mm, a bulk density of 250 kg/m^3, a volume water absorption of 7 %, and a compressive strength in the cylinder of 1.5 MPa. Bulk density of EA samples was 14 % lower compared to the samples E0 due to the autoclave treatment. The thermal conductivity of 10 cm thick layer of granules, determined in accordance with the methodology of Russian State Standard GOST 7076-99 “Building materials and products. Method of determination of steady-state thermal conductivity and thermal resistance”, was 0.71 W/(m·K).

Due to the presence of intergranular voids, granulated foam-glass ceramics from the batches E0 and EA have a significantly lower bulk density (290 and 250 kg/m^3, respectively) compared to foam-glass ceramics, obtained in mold from the batches ED and EAD (540 and 450 kg/m^3, respectively). Consequently, a larger volume of foam-glass ceramics in granular form is released that is economically justified, given the wide scope of this material [4]. In addition, due to intergranular voids and lower density of granulated foam-glass ceramics, there is a proportional decrease in the specific consumption of expensive NaOH per unit volume of material, compared to cubic samples synthesized in mold.

The samples properties can be compared with the results of other authors. Foam-glass ceramics was synthesized at a lower mass ratio between diatomite and NaOH equal to 4; cubic samples with an average density of 200–220 kg/m^3 and compressive strength 2.0–2.3 MPa were obtained [7]. In this study the higher average density and compressive strength of cubic samples of 450 kg/m^3 and 3.4 MPa, respectively, was caused by the higher mass ratio between diatomite and NaOH equal to 9.1. Thus, the extrusion method is preferable in obtaining granular foam-glass ceramics, since its bulk density is almost half the average density of cubic samples: 250 and 450 kg/m^3, respectively, i.e. a larger volume of material is released.

4. Conclusion

1. The separation of the samples along with the formation of a porous structure on top and dense ceramics from below due to the high water content in the liquid batch of the wet method was observed. The samples with increased average density and non-uniform structure containing both foamed particles and more dense ceramic particles were obtained after pre-drying the liquid batch.

2. The batch with lower water content, obtained by the extrusion method, was foamed evenly, without stratification, thereby structure of foam-glass-ceramics was optimized and its average density reduced. An additional decrease in the average density of samples from 540 to 450 kg/m^3 due to the activation of dissolving the diatomite silica phases during the autoclave treatment of the extruded batch was defined. The additional saving NaOH in proportion to the decrease in the average density of the material was achieved.

3. Combining the processes of mixing and granulation of the batch in a single operation is the advantage of the extrusion method in foam-glass ceramics technology. The maximum economic and technological efficiency of the extrusion method is achieved during obtaining granular foam-glass ceramics as a result of reducing the consumption of NaOH and bulk density due to the presence of intergranular voids. Using the extrusion method and autoclave treatment the samples of granulated foam-glass ceramics with a bulk density of 250 kg/m^3, thermal conductivity of the granule layer of 0.71 W/(m·K) and compressive strength in the cylinder of 1.5 MPa were obtained.

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Contacts:

Konstantin Ivanov, +7(922)0424352; sillicium@bk.ru

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Оптимизация структуры и свойств пеностеклокерамики

К.С. Иванов
Институт криосферы Земли ТюмНЦ СО РАН,
625000 г.Тюмень, ул.Малыгина, 85, а/я 1230

Ключевые слова: строительный материал, теплоизоляция, пеностеклокерамика, силикаты

Аннотация. В исследованиях оптимизированы основные свойства пеностеклокерамики — пористого неорганического теплоизоляционного материала. Данный материал применяется для теплоизоляции различных инженерных сооружений: фундаментов, оснований автомобильных и железных дорог, трубопроводов и т.д. Основными сырьевыми составляющими материала являются гидроксид натрия и опал-кристобалитовые горные породы: диатомит, трепел, опока. Смесь компонентов подвергается обжигу и вспенивается с формированием пористой структуры, которая включает как стекловидную, так и кристаллическую фазы. В исследованиях проанализировано и установлено существенное влияние двух различных способов приготовления смеси компонентов на основные свойства материала. В первом способе шихта представляла собой суспензию с высоким содержанием воды, которая подвергалась механической активации в вибрационной мельнице. Во втором способе, шихта была получена экструзией, т.е. путём продавливания смеси компонентов через калиброванные отверстия с помощью шнека. Вследствие этого, шихта представляла собой гранулированную туго-пластичную массу с низким содержанием воды. В результате было установлено, что более высокое содержание воды в первом способе синтеза приводит к формированию неоднородной структуры образцов, их расслоению, наличию плотных не вспененных включений и повышенной средней плотности материала. Шихта, полученная экструзионным методом, вспенивается более равномерно, без расслоения, благодаря чему снижается средняя плотность пеностеклокерамики. Дополнительному снижению средней плотности образцов на 17 % способствует автоклавная обработка шихты, полученной экструзионным методом, за счёт активизации процесса растворения кремнезёма диатомита. Экструзионный метод рекомендован для получения пеностеклокерамики в гранулированном виде, что способствует экономии дорогостоящего гидроксида натрия.

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Контактные данные:

Константин Сергеевич Иванов, +7(922)0424352; эл. почта: sillicium@bk.ru