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Firing of Cellular Ceramics from Granulated Foam-Glass

O.A. Fomina[1,a*], A.Yu. Stolboushkin[1,b]

[1]Siberian State Industrial University, Novokuznetsk, 654007, Russia

[a]stoxan@gmail.com, [b]stanyr60@gmail.com

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Abstract. It has been presented the study results of the firing process of cellular ceramics from granulated foam glass. The chemical, mineral and granulometric compositions of the raw materials are given. It has been shown the characteristic of ceramic-technological properties of raw materials. The samples were burned from the granulated mixture in the temperature range of 850-1000 ° C. It has been established the change dependence in the physicomechanical properties of cellular ceramic samples on the temperature and firing duration. The results of the study of the macro- and microstructure of cellular ceramics are given. It has been revealed the effect of intensive formation of the pyroplastic phase and the connection between small pores at a temperature of more than 950 ° C. After the enlargement, the cells leave the three-phase ceramic system and it was the increase in the average density of cellular ceramics is 1.4-1.5 times. The influence of a solid glass-ceramic shell along the inner surface of the pores on the decrease in water absorption of cellular ceramics to 6.5-7% is established.

Introduction

There are new requirements for energy saving and energy efficiency of buildings in the modern construction sector [1]. Single-layer exterior walls of ordinary building materials do not meet the standards for thermal insulation of buildings in most parts of Russia. According to current regulations, the required thermal resistance of walling has increased more than 2.5 times [2]. The exploitation of the multilayer walls revealed negative aspects, for example the complexity of the manufacture and installation of walls, the problem of co-working layers when the temperature drops, reducing vapor permeability, and sometimes fire safety structures [3-5]. Cellular building materials have high strength and at the same time the heat engineering indices, therefore can be used in single-layer exterior walls. In recent years, active research has been conducted on the creation of highly-porous and cellular ceramics [6-16].

The authors developed a new method of making wall ceramic products to form the cellular structure of ceramics [17]. Dry ground clay raw materials are crushed, mixed with wet granulated foam glass, then using a semi-dry pressing, drying and firing of cellular ceramics. As a result, structural heat-insulating ceramic materials with an average density of 900-1200 kg/m3 were obtained [18].

As the firing temperature rises, the amount of liquid phase in the system increases and sintering and material strength are also rises. At the same time, the excessive pyroplastic state of the system leads to the destruction of the cellular structure and an increase in the average density. As a result, heat-efficient performance of the material reduced.

The purpose of the work was to study the structure and properties of cellular ceramics from granulated foam glass at different temperatures and firing times.

Research Methods

In the current research we used standard and precision analysis methods. Characterization of raw materials was carried out according to the GOST 21216-2014 “Clay raw materials. Test methods" (National State Standard) and GOST 9169-75 "Clay raw materials for the ceramic industry.

Classification". The chemical composition of the materials was determined by the method of X-ray fluorescence wave dispersive analysis (Shimadzu XRF-1800 spectrometer).

The granulometric composition was investigated by the methods of sieve analysis and diffraction of the laser radiation of suspensions (Mastersiser 2000 laser analyzer). The structure of ceramic samples was studied by optical and electron microscopy (scanning electron microscope JSM-6460LV).

Research Objects

In the current research it has been investigated the usual clay raw materials of Western Siberia, granulated foamed glass (GFG) and samples of cellular ceramics. Clay raw materials are moderately plastic low-melting loam (deposit of Novokuznetsk city, Kuzbass region). Granulated foam glass is made of silica rocks according to the original technology [19] by «Baskei Ceramic» (Chelyabinsk city). Samples of cellular ceramics were made in the laboratory using the developed method [17] at different firing temperatures.

Chemical, granulometric and mineral composition of raw materials given in Tables 1-3.

Table 1. Chemical composition of raw materials

Raw materials	Mass fraction of components [%]												
	SiO_2	Al_2O_3	Fe_2O_3	FeO	CaO	MgO	Na_2O	K_2O	TiO_2	S	P_2O_5	MnO	*LOI**
Loam	61.9	14.2	4.21	0.72	4.44	3.38	2.23	1.61	0.85	0.1	0.45	0.4	5.4
GFG	78.43	7.81	4.45	-	0.22	0.75	6.24	1.65	0.45	-	-	-	-

*LOI – loss on ignition

Table 2. Granulometric composition of raw materials

Raw materials	Composition of the fractions					
Loam	Fraction content [%], particle size [μm]					
	–5+0	–10+5	–20+10	–50+20	–100+50	
	30	5	5	60	–	
GFG	Fraction content [%], particle size [mm]					
	2.5	1.25	0.63	0.315	0.14	< 0.14
	0.0	11.5	72.0	14.8	1.6	0.1

Table 3. Mineral composition of raw materials

Raw materials	Prevailing Minerals	
	Clayey	Nonplastic
Loam	Montmorillonite, gidromuskovite, a little kaolinite	Quartz, plagioclase, vermiculite chlorite, K-spar, calcite, amphibole
GFG	-	Quartz, feldspar

By chemical composition (Table 1) loam is a semi-acid raw material (Al_2O_3> 14%), GFG – an acidic raw material (Al_2O_3 < 14%). Raw materials have a high content of coloring oxides (Fe_2O_3> 3%).

According to the particle size distribution (Table 2), the loam has a low content of coarse-grained inclusions (the number of particles> 0.5 mm is less than 1%). Loam is low-dispersion raw materials (the number of particles <0.001 mm – 20-30%) according to the content of fine fractions. The granulometric composition of the GFG is mainly represented by particles of 0.5-1.0 mm.

By the mineral composition (Table 3) loam is a polymineral clay material of the montmorillonite- hydromicaceous type. The non-plastic part of loam in descending order is represented by quartz, carbonates, feldspars, chlorite, and amphiboles. The GFG contains X-ray amorphous phase, quartz and feldspar (anorthite).

Results and Discussion

Ceramic samples of the cellular structure were prepared from a two-component mixture of constant composition: 75% GFG and 25% loam. The investigated compositions [17] give the lowest values of the average density and thermal conductivity of the material.

The content of GFG fractions of class -2.5 + 0.14 mm was regulated by the sieve method. Cellular ceramics with a high coefficient of structural quality from GFG of three fractions was experimentally obtained: 0.315-0.63 mm - in the amount of 12-15%; 0.63-1.25 mm - 70-75%; 1.25-2.5 mm - 10-15% (Table 2).

Cylinder specimens 40-50 mm high and 45 mm in diameter were compacted from granulated press powders. The pressing mode was two-stage with one-sided load application. Pressure - 5 MPa [18]. The firing was carried out in a stepped mode with different isothermal exposure - 30-180 minutes. It was previously established that melting and swelling of samples occur at temperatures above 1000 ° C [20]. Therefore, the maximum firing temperature varied in the range from 850 to 1000 ° C with a step of 50 ° C. The test results of ceramic samples depending on the temperature and firing time are given in Tables 4-5.

Table 4. Physical and mechanical properties of ceramic samples, depending on the maximum firing temperature

Firing temperature, [°C]	Average density, [kg/m^3]	Compressive strength, [MPa]	Water absorption, [%]	Fire shrinkage, [%]	Strength density ratio	Surface appearance
850	1027	10,6	8.8	2.8	10.3	without defects
900	1012	18.7	7.0	3.3	18.5	
950	1081	24.5	6.3	4.1	22.7	partial fusion of samples
1000	1492	28.9	4.8	7.9	19.4	

Table 5. Physico-mechanical properties of ceramic samples, depending on the exposure time at the maximum firing temperature

Exposure time, [min]	Average density, [kg/m^3]	Compressive strength, [MPa]	Water absorption, [%]	Fire shrinkage, [%]	Strength density ratio	Surface appearance
30	1120	13.6	7.5	3.0	12.1	without defects
60	1080	15.1	7.3	3.3	13.9	
90	990	18.7	7.0	3.7	18.9	
120	1150	21.9	6.5	4.1	19.1	partial fusion of samples
180	1270	24.4	5.0	4.9	19.2	

Samples have a maximum strength at a firing temperature of 1000 ° C, and a minimum average density at 900 ° C (Table 4). The maximum value of the design quality coefficient have the samples at the firing temperature of 900 ° C, while simultaneously providing strength and heat-shielding indicators of cellular material. Therefore, the optimum firing temperature range is 900-930 ° C. With an increase in the exposure time from 30 to 180 minutes at maximum temperature, the strength of the samples increases from 13.6 to 24.4 MPa (Table 5). Water absorption is reduced from 7.5 to 5%. The minimum values of the average density (990 kg / m3) have samples at a shutter speed of 90 minutes with virtually no decrease in the coefficient of structural quality. Granules of the GFG are melting during this time, and in their place in the material were developed an airs cells. As a result, it has been formed the cellular ceramics with a glass-ceramic frame. Exposure of more than 90 minutes leads to further melt formation and compaction of the material. Therefore, the optimal isothermal holding time at maximum temperature is 80-90 minutes.

The study of the macro- and microstructure of the samples as a function of the firing temperature was carried out using optical and electron microscopy (refer with: Fig. 1-3).

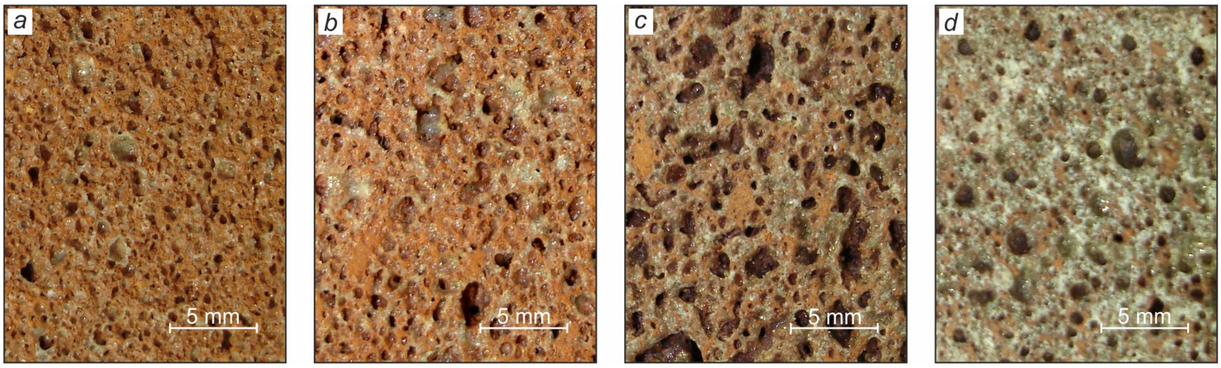

Fig. 1. Macrostructure of cellular ceramic samples from granular mixture based on GFG and loam, polished, reflected light, firing temperature, ° C: a – 850; b – 900; c – 950; d – 1000

In reflected light, all ceramic samples have a cellular structure with a predominance of closed macropores-cells of predominantly spherical shape (Fig. 1).

Polished sections show a change in the color of the material with increasing firing temperature. The solid phase of cellular ceramics is formed in the form of interporous partitions made of clay deposited on the surface of GFG granules. During firing, the Fe_2O_3 contained in loam provides the red color of the interporous partitions. The glass phase interacts with interporous partitions and triggers the mechanisms of liquid-phase sintering of ceramics during the melting of granules from GFG. During reduction to the ferrous form, FeO reduces the red color even at 950 ° C. This is clearly seen at 1000 ° C (Fig. 1, d).

The firing temperature affects on the shape and size of the pores. It is prevailed the closed pores with a size of 0.5-1.0 mm at the temperature of 850 ° C (Fig. 1, a). The amount of the pyroplastic phase increases as the temperature rises. The viscosity of the three-phase ceramic system is reduced. As a result, there is a partial merging of small pores with each other and their enlargement (Fig. 1, b). At the temperature of 950 ° C, it has been appeared the elongated (length — 3-5 mm) closed and semi-closed shape pores-cells of capillary type (Fig. 1, c); the thickness of interpore partitions increases. At a temperature of ≥ 1000 ° C, the amount of the melt increases and destruction of the cellular structure occurs with the release of a significant amount of the gas phase from the ceramic system. There is an increase in fire shrinkage (> 2.5 times, referred with: Table 4) and compaction of a ceramic sherd (≈ 1.5 times, referred with: Table 4).

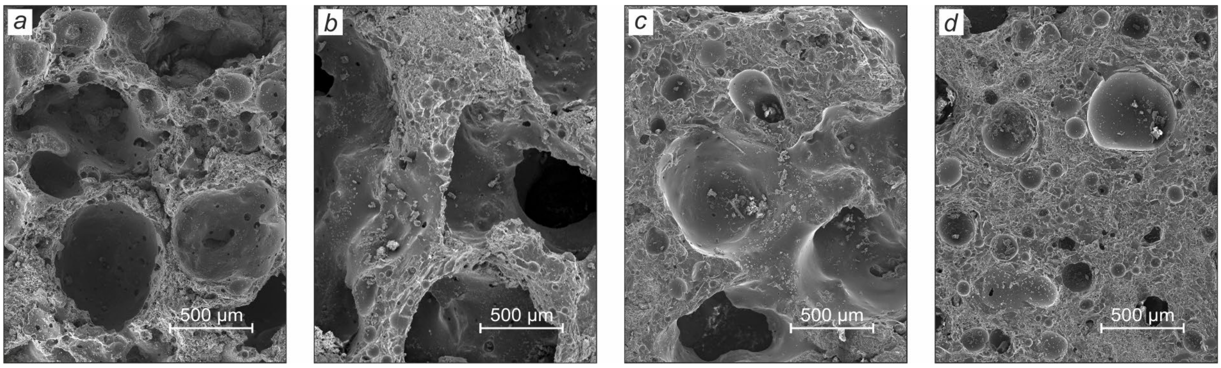

Fig. 2. Microstructure of cellular ceramic samples from granulated mixture based on GFG and loam; SEM, firing temperature, ° C: *a* – 850; *b* – 900; *c* – 950; *d* – 1000

SEM studies complement optical microscopy and show the detailed structure of interporous partitions. It is also occurs the enlargement and connection of pores with an increase of the temperature and firing time (Fig. 2, b, c). As a result, oblong pore-cells are formed with a shape factor of 2–3 or more. At the same time, the thickness of interporous partitions increases from 30–50 μm to 300–800 μm (Fig. 2, c). At the temperature of 1000 ° C, solid areas of solidified melt are observed with an abundance of small (≤ 100 μm) spherical closed pores (Fig. 2, d). At the micro level, the structure of interporous partitions changes.

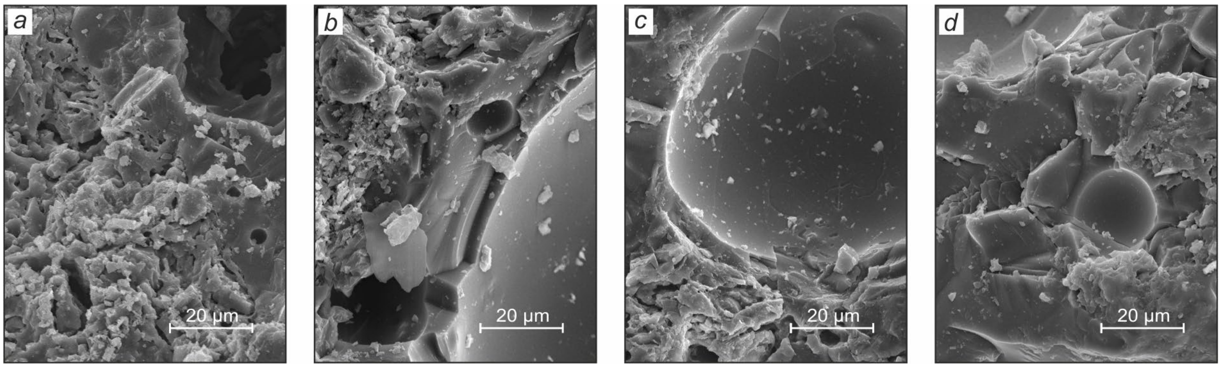

Fig. 3. Micrographs of the glass-ceramic phase at the interface of cellular ceramic samples, SEM, calcination temperature, ° C: – 850; *b* – 900; *c* – 950; *d* – 1000

Druze microstructure consisting of small cryptocrystalline particles (Fig. 3, a) melts and forms a glass-ceramic microstructure with an abundance of glass phase (Fig. 3, d). Spherical micropores with a diameter of 1-20 microns are formed in the solid phase during firing (Fig. 3).

Conclusions

It have been carried out the studies of firing of cellular ceramics on the basis of granulated foam glass.

It was established that the optimal cellular structure of ceramics is formed by pressing a granular mixture of composition of 25% loam and 75% GFG and roasting with an exposure time of 80-90 minutes at a temperature of 900-930 ° C. Under these conditions, the best balance between strength (18-24 MPa) and average density (1000-1100 kg / m3) of ceramic material is ensured.

It have been established the effect of connecting small cells of the gas phase with each other, their enlargement, migration and exit from the three-phase ceramic system at a temperature of more than 950 ° C. As a result, the average density of cellular ceramics increases 1.4-1.5 times.

The formation of a solid glass-ceramic shell along the inner surface of the cells of the gas phase was established, which ensures low water absorption of the ceramic material (6.5-7%).

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