Deformation and Heat-Insulating Characteristics of Light Concrete on Porous Burned Binder Under Heating

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Abstract. It is known to use heat-insulating materials with a rigid cellular structure on cement, gypsum, liquid glass binders as a constructive fire protection. The described technologies for the manufacture of such materials do not allow combining a binder with an aggregate. The use of frame technology made it possible to obtain a material with an aggregate on a porous burned soda-lime-silicate waterproof binder. The article presents the results of studies of the fire-proof properties of samples from the obtained material. The deformation and heat-insulating characteristics of the material when heated are used as criteria for fire-proof properties. Volumetric heating of the material to a maximum temperature of 892 °C was carried out when studying the deformation characteristics. The heat-insulating characteristics of the material were studied during one-sided heating of a material sample in the form of a tile to a maximum temperature of 1050 °C. The characteristics obtained indicate that the material can be used for fire-proof lining of building structures and as for the construction of screen walls and as a filling material in fire barriers.

Keywords: coarse aggregate, liquid glass, soda-lime-silicate glass, burned binder, fire-proof characteristics of the material

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Introduction

An effective way to protect geometrically simple building structure elements from fire is by covering them with fire-resistant materials with low thermal conductivity.

The commonly used measure is to use insulation materials with low thermal conductivity to delay the increase in the structure's temperature. This phenomenon prevents structural failures due to fire, including intumescent fire-proof paints [1,2], coatings and plasters of semiliquid compositions [3], and insulation boards with a rigid structure [3,4].

Fire protection thermal insulation materials with a rigid structure can be bricks, stones, sheets, slabs, panels, and shells. The materials are fabricated from cement [5], gypsum [4,6], sodium silicate [7], calcium silicate [8,9,11,16,17], magnesium oxide [10,11], and vermiculite [12]. The polarization of the binder reduces the density and thermal conductivity of the material [4,13] and the inclusion of porous fire-resistant aggregates in its composition [14], for example, perlite and vermiculite [15-17].

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Cellular silicate glass or glass foam is recognized for its unique operational and thermal insulation properties and desirable fire resistance. Using different wastes in glass foam production reduces material costs and makes it competitive. The literature [18-20] indicated the critical parameters for material selection and sintering of glass foams produced using waste glass and the glass foam properties. Technologies for obtaining cellular glass from the recycled waste of soda-lime-silicate glass formed in the sphere of consumption have been developed and discussed by several researchers [21-26]. Most conventional soda-lime-silicate glasses have a sufficient high softening temperature of 550-700 °C, so cellular silicate glass can also be used as a fire-retardant material, but with the inclusion of fire-resistant fillers in its composition are not currently implemented.

A study examined the glass-ceramic foams for thermal insulation based on alkaliactivation and sintering of zeolite-poor rock [27,28]. The alkali-activation and reactive sintering technology produced the acquired foams by adding powdered eggshell or zeolite-poor rock. A similar technology obtained foam glass from solid waste of flat glass and exhausted alkaline batteries [29].

The frame technology is proposed [30,31] for producing fire protection material with expanded clay as a coarse aggregate. A glass powder was selected for forming a fired porous vitreous binder. The glass powder was obtained by grinding the utilized container and building glass and liquid sodium glass. The obtained material had water resistance, and fire safety properties make it possible to use it as a rigid insulating or structural-insulating construction material. However, the physical and mechanical characteristics of the material have not been studied in detail.

Therefore, a review of existing technologies for manufacturing fire-retardant heatinsulating materials with a rigid structure shows that, at the moment, there are no scientific results and technologies that allow combining the cellular structure of coarse aggregate with a binder.

This study aims to obtain a material with expanded clay filler on a porous calcined sodalime-silicate waterproof binder and determine its fire-retardant properties. The deformation and heat-insulating characteristics of the samples under heating evaluated the fire-proof properties.

Materials and methods

Expanded clay with a fraction of 5-10 mm with a bulk density of 530 kg/m³ was used as a filler for the developed material on a firing binder. The expanded clay fraction with the smallest grain size was selected to reduce the stresses at the interfaces between the grains of the filler and the binder since these stresses increase with an increase in the grain size.

Soda liquid glass with a density of 1.48 g/cm^3 and a suspension of finely ground cullet from the secondary waste of soda-lime-silicate glass were used as components for synthesizing the firing binder. The fineness of glass grinding was characterized by a residue on grid No. 0063 of 5-6%. The chemical composition of glass is presented in Table 1.

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	Mn ₃ O ₄	TiO ₂	BaO
60.4	11.8	1.4	8.0	0.3	13.9	2.5	0.3	1.3	1.4	-

Table 1. Chemica	l composition	ı of glass,	%
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The frame technology [30,31] was developed by the authors and used to prepare the samples. The preparation of frame molding technology includes the following operations. Initially, the aggregate's grains were mixed with sodium liquid glass. Then the aggregate with a liquid glass-coated surface was placed in the mold. Further, the mold with the aggregate was placed in an electric airing cupboard and exposed to heat treatment at a temperature of 60-80°C, during which the adhesive bond made of liquid glass hardened. The glued hardened frame of

aggregate grains was removed from the mold and soaked with a water slurry of finely ground cullet from the secondary waste of soda-lime-silicate glass. If the volume of intergranular voids of the cured framework was filled partly with an impregnating suspension of finely ground glass, then the materials macrostructure turned out to be large-porous (Fig. 1, a). A combined macrostructure sample cross section with two layers (monolithic and large-porous) was obtained by completely filling the framework voids on one side with glass suspension and not completely on the other side (Fig. 1, b).



Fig. 1. The sample of a large-porous structure size $42 \times 41 \times 167$ mm (a) and a combined structure sample with large-porous and monolithic layers size $84 \times 84 \times 40$ mm (b)

The advantage of the board material of the combined structure is the increased adhesion of the coarse-porous layer to the solution of the connecting layer due to the mechanical engagement of the coarse-porous side. After impregnating the glued frame suspension of finely ground glass, it was dried repeatedly at a temperature of 60-80 °C. After drying, the molded samples were burned in an electric furnace on a refractory pallet. The frame molding technology burns a raw product without heat-resistant forms. The firing temperature was limited to 780 °C.

The material's porous burned binder with the aggregate was synthesized in successive drying and burning operations of the molded sample from two components – sodium liquid glass and a suspension of finely ground cullet. The binder synthesis mechanism obtained by firing including the following stages. The hardening of the frame's liquid-glass glue from aggregate grains during drying is accompanied by the appearance of supersaturated metastable solutions, followed by the transition of hydrate forms of sodium silicates when moisture evaporates into a vitreous state and polymerization of silicon-containing chemical bonds with removing hydroxyl-hydrogen groups with the formation of a silicon polymer frame of a volumetric mesh structure with cells of the type:

$$- \stackrel{|}{\underset{i}{\operatorname{Si}}} - \stackrel{|}{\underset{i}{\operatorname{O}}} - \stackrel{|}{\underset{i}{\operatorname{Si}}} -$$

After soaking the glued hardened frame with a suspension of cullet, during the initial drying period at the interface of the phases. The partial dissolution of the liquid-glass binder of the frame occurs, accompanied by the transition of sodium cations into the solution due to its

solvation interaction and hydrolysis of the binder's anionic skeleton, leading to the release of monomeric and polymer anions of hydrated silica. At a particular stage of drying, the decrease in the pH of the medium and the increase in the concentration of hydrated silica leads to polymerization of the latter with the formation of viscous gel layers that slow down the dissolution of the liquid-glass binder of the frame. Slowing down the binder's dissolution process contributes to preserving a certain strength by the glued frame and allows the frame to dry off the soaked frame without mold. Simultaneously with the interaction of the suspension of finely ground cullet with the frame's liquid glass glue, leaching and watering of the initial glass structure of the suspension particles occur, the mechanism of which is based on the interaction of aqueous solutions of alkalis with silica. Glass watering occurs during its hydration and hydrolysis. The glass watering is accompanied by the adsorption of hydrated alkali metal cations on the active areas of the silica surface that arise during glass grinding, followed by depolymerization of silica due to the hydrolysis of ≡Si–O–Si≡ bonds with the formation of silanol groups Si-OH. Hydrated silica is transferred during drying to the surface of glass particles. With the increase in its concentration during drying, silicic acid polymerization occurs with a thick elastic gel film with astringent properties. As free water is removed from the dried material, sodium hydro silicates are formed in the volume of the binder from the degradation products of the frame's adhesive binder and the glass particles of the suspension. The free water is remaining after drying forms hydrogen bonds with silanol water. The dried semifinished product's heated up to 400 °C is accompanied by water removal from sodium hydro silicates. Further increasing the temperature to the limiting burning temperature of 740-780 °C resulting the silicic acid bonds are destroyed, the silicic-oxygen tetrahedra polymerize, and water is released during the dehydration of the hydroxyl cover of the glass. In the same temperature range, the eutectic mixture of the Na₂O-CaO-SiO₂ system formed from the components of the complex binder ensures that during melting, the accumulation of a significant amount of melt with the necessary pyroplastic mobility and the formation of closed pores in the volume of the firing binder. The coincidence of the processes of gas release and the appearance of closed pores in the melt creates conditions for the swelling of the binder due to a sharp increase in vapor pressure in the pores. It is not advisable to increase the firing temperature above 780 °C due to a decrease in the binder viscosity and disturbance of the pore formation process. According to the results of the microscopic examination, the diameter of the pores from the fired binder ranged between 0.02 and 0.5 mm. The swelling of the binder leads to an inevitable volume increase, and the aggregate's grains move apart without changing the product's shape.

The developed technology offers a waterproof material with a large-pore structure and a combined structure with large-pore and monolithic layers with a density of 567 and 710 kg/m³, respectively, Fig. 1. The compressive strength of the material in large-pore structure is 1.5 MPa. Prismatic samples of size $42 \times 41 \times 167$ mm (Fig. 1, a) were used to determine the deformation of the longitudinal bending under the action of their dead weight during heating in an electric resistance furnace, Fig. 2, a. The increase in temperature rate in the furnace was 14.0 °C/min at the beginning of heating and 4.5 °C/min at the end.

Samples in the form of tiles of a combined structure with large-porous and monolithic layers $84 \times 84 \times 40$ mm in size (Fig. 1, b) were used to determine the heat-insulating characteristics of the sample under the influence of temperature from one side. A unilateral effect on the temperature sample was carried out by sealing the opening of the electric resistance furnace with a tile so that the coarsely porous layer of the tile was facing inside the furnace, Fig. 2, b. After sealing the furnace opening with tiles, the temperature in the furnace was raised. The temperature change on the surface of the tile facing outward was recorded with a pyrometer. The heat-insulating characteristics of the tile sample were evaluated by the temperature on the surface of the tile facing outward.



Fig. 2. Testing samples for deformation of the longitudinal bending (a) and the heat-insulating characteristics (b) in electric resistance furnaces

Results and discussion

The following results were obtained for the deformation of longitudinal bending of the samples under study. Visual observation of the samples during their heating in an oven for 70 min up to a temperature of 788 °C; no changes were found in the samples. With a further rise in temperature for 10 min. from 788 °C to 835 °C, buckling deformations of the samples developed due to a decrease in the viscosity of the vitreous binder and its viscous flow. The growth of deformations ended with the loss of stability of the samples at a temperature of about 835 °C, Fig. 3, a. It should be noted that the temperature of 788 °C at the beginning of sample deformation is higher than the temperature range of 550–700 °C at the beginning of softening typical soda-lime-silicate glasses. The interaction of the binder with the surface of the filler, which resists the viscous flow of the binder, contributes to more excellent temperature resistance.





Therefore, the filler increases the material's resistance to deformations under heating. With a further rise in temperature for 20 minutes from 835 °C to 892 °C, no visible changes in the shape of the deformed and buckling specimens were observed (Fig. 3, b, c). Therefore, the resistance of the material of prismatic samples to deformation, caused by the binder's softening

during heating, is maintained up to a temperature of 788 °C, which exceeds the softening point of typical soda-lime-silicate glasses by at least 100 °C. The integrity of the material is preserved when it is heated to even higher temperatures, at least up to 892 °C, under the termination of the longitudinal force from its dead weight on the sample. The preserving of integrity indicates the positive effect of the filler on the increase in the structural strength of the material when heated due to the interaction of the binder with the surface of the filler. Table 2 shows the heat-insulating characteristics of a combined structure tile (Fig. 1, b).

Time from the beginning of the temperature increase in the heating chamber of the furnace, min	0	5	10	15	30	45	60	75	76
The temperature in the heating chamber, °C	25	320	455	730	900	1020	1050	1050	Trial terminated
Facing the outward sample's surface temperature, °C	25	37	56	147	220	286	324	329	_

Table 2. The tile's heat-insulating characteristics

The temperature regime in the heating chamber of the furnace complies with the Russian State Standard GOST 30247.0-94 (ISO 834-75) "Elements of building constructions. Fire-resistance test methods. General requirements" [32], used for testing building structures for fire resistance. This dependence determines the temperature regime:

 $T - T_0 = 345 \log (8t + 1),$

where *T* is the temperature in the furnace corresponding to the time *t*, °C; T_0 is the temperature in the furnace before the start of thermal exposure (assumed to be equal to the ambient temperature), °C; *t* is the time calculated from the beginning of the test, min.

As a criterion for the limiting state of the enclosing structure in terms of the loss of heatinsulating ability, an increase in temperature on the unheated surface of the structure by more than 220 °C was taken following the fire resistance test methods of Russian State Standard GOST 30247.1–94 "Elements of building constructions. Fire-resistance tests methods. Loadbearing and separating constructions" [33]. Experimental data showed (Table 2) that a temperature increase on the tile's unheated surface by more than 220 °C was recorded after the 30th minute of the test. Following the accepted criterion, 30 min is a required time to reach the limit state based on the loss of the heat-insulating ability of the prototype tile. No visible changes were found in the unheated surface of the sample tile during the test. Inspection after the test of the surface of the sample tile facing the heating chamber of the furnace also showed no visible changes in this surface, except for a change in color towards darkening. Insignificant, by 5 °C, increase in the temperature of the unheated surface of the sample tile while maintaining the maximum temperature of 1050 °C in the heating chamber of the furnace for 15 min. (Table 2) testifies to the high heat-shielding characteristics of the material.

Conclusion

The object of the study was material with expanded clay filler on a porous calcined soda-limesilicate waterproof binder (expanded glass concrete). The fire-retardant properties of the material were evaluated by the deformation and heat-insulating characteristics of the samples during heating. According to the research results, water-resistant material with a large-pore structure and a combined structure with large-pore and monolithic layers with a density of 567 and 710 kg/m³, respectively, was proposed. The material of a large-pore structure has a compressive strength of 1.5 MPa. The deformation characteristics of the samples of expanded clay-glass concrete during their heating in an electric furnace were determined by visual observation of the deformations of the buckling of the samples under the action of their dead weight. Deformations develop due to the softening of the porous glassy binder during the heating of the samples. The heat-insulating characteristics of expanded clay-glass concrete were evaluated by the surface temperature of the sample tile during the heating of the opposite tile surface. A hole in the electric furnace was sealed with a sample tile, the temperature in the furnace was raised to 1050 °C, and the temperature was measured with a pyrometer on the surface of the sample facing outward.

The following conclusions are drawn from studying the deformation and heat-insulating characteristics of samples of expanded clay-glass concrete during heating.

1. No deformations were recorded in the samples when it is heated up to 788 °C. When the sample heated above 788 °C, buckling deformations occurred in the samples due to the softening of the porous vitreous binder. The developed deformations lead to the loss of stability of the samples when it reaches a temperature of about 835 °C.

2. The softened glassy binder's interaction with the filler surface leads to an increase in the material's structural strength, as a result of which the temperature of 788 °C at the beginning of the deformation of the samples exceeds the softening point of typical soda-lime-silicate glasses by at least 100 °C.

3. The heating of samples after deformation and stability loss happened at a temperature of 892 $^{\circ}$ C, exceeding the beginning of softening the glassy binder of expanded clay-glass concrete by about 200 $^{\circ}$ C, which does not lead to loss of samples integrity.

4. The limiting state of the sample tile of expanded clay-glass concrete in terms of loss of heat-insulating ability was recorded after the 30th minute of the test. The limiting state criterion is an increase in temperature on the unheated surface of the sample tile by more than 220 °C.

5. Insignificant, by 5 °C, increase in the temperature of the unheated surface of the sample tile while maintaining the maximum temperature of 1050 °C for 15 minutes. The furnace's heating chamber indicates the material's high heat-shielding characteristics. The obtained deformation and heat-insulating characteristics of expanded clay-glass concrete during heating show that this material can be used for fire-protective cladding of building structures and fire-protective material in fire-proof partitions and screen walls.

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