Advanced Physical Research
Vol.6, No.1, 2024, pp.63-72
https://doi.org/10.62476/apr61.72

STUDY OF BRITTLE FRACTURE AND TEMPERATURE RESISTANCE IN THE RANGE OF 20-1100°C OF AUTOCLAVED CELLULAR REINFORCED MATERIALS

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Abstract. In this paper, the study of cellular autoclaved reinforced material resistant to brittle fracture and temperatures in the range of 20-1100°C. The analysis of the results shows that the studied cellular autoclaved reinforced materials by the value of F (brittle fracture criterion) will not brittle fracture under conditions of a sharp change in temperature from 20 to 300 °C because the value of F ≤ 4. It was determined that on the wall system made of blocks of the material in question with a density of 400 kg/m^3, 10 days after the completion of masonry under standardized fire exposure for 180 minutes in the temperature range of 20-1100 °C, there was no loss of thermal insulation, bearing capacity and integrity of the samples. During the tests, the maximum temperature values in the middle of the cross-section of the samples did not exceed 97°C. The values of As, Af, Amin for the test time of 182 minutes were 175074, 175212, 168560 °C·min, respectively, which meets the criterion requirements of DSTU B V.1.1-19:2007 and ensures the fire resistance class REI 180.

Keywords: *Autoclaved, cellular, crack, material, fiber, resistance, temperature.*
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Received: *2 November 2023;* ***Accepted:*** *12 January 2024;* ***Published:*** *19 February 2024.*

1. Introduction

The study of the durability of autoclaved cellular materials over time is possible by the following indicators: bending strength; frost resistance; linear shrinkage, crack resistance and resistance to variable temperature fields, for example, in the event of a fire. As they known from the works (Recommendations, 1979; Gorchakov, *et al.*, 1968; Regel, *et al.*, 1974; Cherepanov, 1974; Vyrovoy, 1988), crack resistance is an important characteristic that reflects the actual physical process of their destruction. The quantitative assessment that determines the resistance of such materials to crack development is the invariant material constant K_{si}, the critical stress intensity factor at tear.

The process of main cracks development and fracture in the macrostructure of the cellular material occurs in different ways, mainly due to the main initial structural defects - cracks at the interface "matrix - filler - pore - gas". Cracks occur during shrinkage and

How to cite (APA):
Lapovska, S.D., Konoplya, M.S., Chernenko, M.V. & Guzii, S.G. (2024). Study of brittle fracture and temperature resistance in the range of 20-1100°C of autoclaved cellular reinforced materials. *Advanced Physical Research, 6*(1), 63-72 https://doi.org/10.62476/apr61.72

are associated with the crystallisation of newly formed structures or are of sedimentary and force origin, so fracture (crack development) occurs along the contact zone with their subsequent release into the matrix.

The contact between the aggregate (sand) and the porous cement stone is the strongest point of the structure, because they determined by the best conditions for cement hydration due to the aggregate releasing water during the concrete maturation process. In addition to releasing water, the aggregate creates a vacuum in the aggregate, which ensures self-evacuation of the contact layer and a natural increase in its strength (Komokhov *et al.*, 1992). Therefore, in the process of fracture, the main role we played by the initial (technological) cracks in the structure of the aggregate and matrix. Moreover, these initial cracks will develop in different ways.

Firstly, cracks tend to penetrate easily from a harder material to a less hard material, and the reverse phenomenon they not observed, cracks developing taken together we mostly be stopped at the interface with the matrix and will not have a dominant effect on the crack resistance of the concrete. Secondly, cracks developing in the matrix will penetrate relatively easily into the aggregate and will play a major role in the failure of cellular concrete. They will "jump" through the sand aggregate, so the main cracks in cellular concrete have a straight-line trajectory, and an increase in the number of pores per unit volume of concrete will lead to a decrease in crack resistance and will contribute to the damping of elastic energy.

The physical nature of elastic energy attenuation is ambiguous. It can be caused by processes occurring both in the solid phase of cement stone neoplasms and as a result of thermoelastic relaxation at the boundaries of phases and aggregates, diffusion of the liquid phase, i.e. its viscous movement in the pore space initiated by alternating stresses of the structure formation process. Plastic deformation, in front of the growing crack front, can develop because of a decrease in the free surface energy of the solid phases of neoplasms in cement stone. The observed plastic deformation is one of the forms of manifestation of the P.A. Rebinder effect (Regel, *et al.*, 1974; Cherepanov, 1974). According to (Vyrovoy, 1988), the cracks that appeared in the cement matrix are new distribution surfaces, on which the next volumetric deformations we detected. A complete redistribution of deformations in the structural microspace occurs, contributing to the development of cracks. If the crack they slowed down by an elastic damper (air in the pore), the effort to further advance it will increase and to start its movement (acceleration), energy will be required at least sufficient to relax the loading elastic impulse. For porous materials, this process we repeated many times.

Due to the introduction of fibre fibres, additional contacts they created between the fibre and the newly formed structure of the nasal sclerosis. Studies (Weidemann *et al.*, 2007; Pehlivanl *et al.*, 2016; Lapovska *et al.*, 2020) have shown that the vast majority of reinforcing fibres are located perpendicular to the direction of expansion (swelling) of the mixture.

This arrangement of fibres leads to a several-fold increase in the bending tensile strength due to the absorption of tensile forces that occur in the tensile zones of the material. In works (Pehlivanl et al., 2016; Ng, 2010; Narayanan *et al.*, 2020), it was noted that fibre in the areas of contact between the fibre and the material matrix promotes additional formation of tobermoryt crystals.

This leads to an increase in the solid phase in the material and a decrease in destructive loads (Othuman *et al.*, 2011; Khan, 2002; Brelak *et al.*, 2017), which significantly affects the K_{si} value, allowing for a predictive assessment of durability based

on the criteria of brittle fracture, temperature distribution, and development of main cracks.

Therefore, it is advisable to investigate the reinforced cellular autoclave material for brittle fracture and temperature resistance in the range of 20-1100°C.

2. Materials and test method

The blocks with dimensions of 600x200x200 mm were manufactured using injection moulding technology under autoclave conditions by YDK LLC (Dnipro, Ukraine) from cellular mix.

Mix composition per $1m^3$: 205 kg of river quartz sand; 50 kg of Portland cement M500-I; 125 kg of quicklime; 6 kg of gypsum; 0.6 kg of aluminium paste; 0.05 kg of surfactant (sulphanol); 0.62 kg of fibre (cellulose fibre); the rest is water.

The autoclaved cellular material was characterised by an average density of 400 kg/m^3, flexural strength of 1.2 MPa, compressive strength of 2.5 MPa, thermal conductivity of 0.089 W/m·°C, and frost resistance of F25.

To determine the value of K_{si}, cubes with an edge of 70 mm with two cuts - cracks in the same plane and one cut - we cut from the block massif according to the method of GOST 29167-91 (Figure 1, a, b).

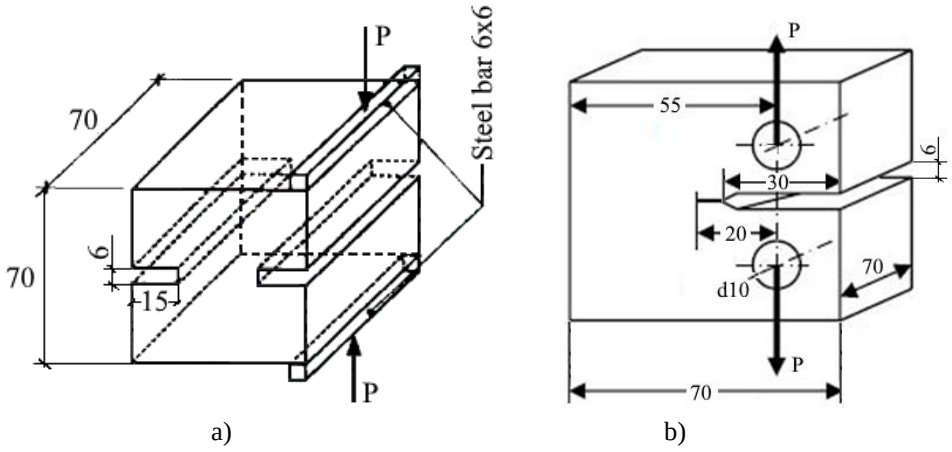
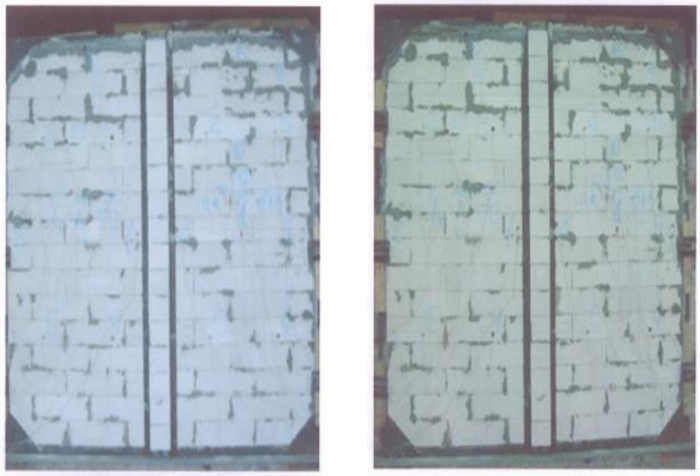


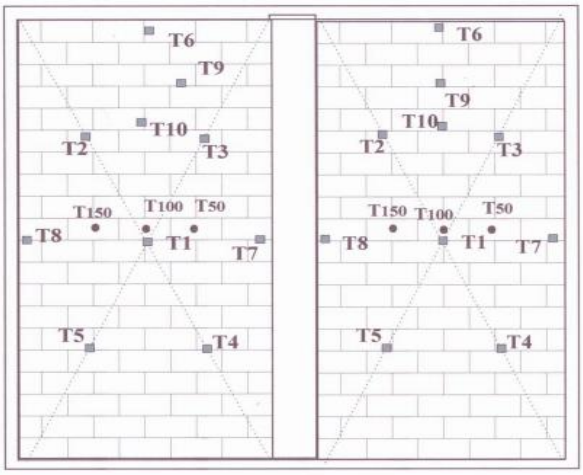
Figure 1. Schemes of testing samples of autoclaved aerated concrete: a - by the criterion of brittle fracture; b - by the development of a main crack

Two wall specimens with actual design dimensions of 3235 x 1460 mm and a thickness of 200 mm were tested. The specimens we mounted in a supporting structure made of steel channel No. 20 (Figure 2). The specimens we separated by blocks with a cross section of 300x200 mm without ligation. The appearance of the samples before and after the tests, the layout of the thermocouples on the samples they shown in Figure 2 and Figure 3.



a) b)

Figure 2. View of samples on the test furnace: a - before the test; b - after the test.



Sample No. 1 Sample No. 2

Figure 3. Layout of thermocouples on samples

The specimens we mounted on an adhesive mixture for aerated concrete UDK TVM C1.MR2 according to DSTU B V.2.7-126-2006, manufactured by LLC "YDK" (Dnipro, Ukraine).

On the surfaces of the samples opposite to the fire action, 10 TCA thermocouples (T1-T10) we installed (see Figure 3) in accordance with DSTU B V.1.1-19:2007. To obtain the temperature distribution values along the cross-section of the samples, three thermocouples we installed on each sample. Thermocouple T50 they installed in the middle of the sample at a distance of 50 mm from the heating surface, thermocouple T100 - at a distance of 100 mm from the heating surface (middle of the sample thickness) and thermocouple T150 - at a distance of 150 mm from the heating surface.

The resistance to brittle fracture of the cellular autoclaved material in the temperature range 20-300°C we determined by **F** the brittle fracture criterion (BFC) (Recommendations, 1979; Guzii et al., 2023). Its value was calculated using formula (1). The formation of main cracks under the action of cyclic loads and as a result of shrinkage phenomena occurring in the structure of the cellular autoclaved material was calculated using formula (2) (Kozhabekov *et al.*, 2000; Pak *et al.*, 1978):

$$F = a\frac{\alpha_{bt}E_{bt}\rho}{K_1\lambda P}W_o^{vom}, \quad (1)$$

where α_{bt} – is the coefficient of linear temperature deformation of the cellular material, 1/°C; E_{bt} - modulus of elasticity of heated cellular material, mN/m^2; ρ - density of cellular material in dry state, kg/m^3; α- a proportionality factor of 1.16·10^{-2} W·m$^{5/2}$/kg; K_1– is the coefficient of pseudo-intensity of stress of a heterogeneous material, mN m$^{-3/2}$;

$$K_I = \frac{P}{BW^{\frac{1}{2}}}((29.6\left(\frac{a}{W}\right)^{\frac{1}{2}} - 185.5(\frac{a}{W})^{3/2} + 655.7(\frac{a}{W})^{5/2} - 1017(\frac{a}{W})^{7/2}) \quad (2)$$

P - total porosity, m^3/m^3; λ - is the thermal conductivity of the cellular material, W/(m·°C); W_o^{vom}- volumetric operational moisture content of the cellular material, m^3/m^3.

The resistance to temperatures in the range of 20-1100°C of the cellular autoclaved material was determined in accordance with DSTU B V.1.1-19:2007. This method consists in determining the time interval from the beginning of the test at a standard temperature of wall samples installed in a vertical opening of a refractory furnace to the onset of one of the fire resistance limit state (Stanescu et al., 2021). Are standard for load-bearing walls in terms of loss of continuity, thermal insulation capacity or load-bearing capacity.

The limit state based on loss of integrity **E** they considered a state in which one of the following ignition conditions we met:

- Flame burning or smouldering from the glow of a cotton swab brought to the unheated surface of the sample at the crack area at a distance of 20 to 30 mm for at least 30 s;

- Flame on the unheated surface of the sample we observed for at least 10 s;

- the appearance of a crack (or hole) through which a probe with a diameter of 6 mm can be freely inserted into the furnace and moved along the crack for a distance of at least 150 mm.

The limit state in terms of loss of thermal insulation capacity **I** is:

- Exceeding the average temperature on the unheated surface of the sample over the initial average temperature of this surface by 140 °C;

- The temperature at any point of the unheated surface of the sample exceeds the initial temperature at this point by 180 °C.

The limit state because of loss of bearing capacity **R** is the state in which one of the following conditions they met:

- The value of the longitudinal displacement of the loaded end of the specimen exceeds the value C = h/100 mm;

- The vertical strain growth rate exceeds dC/dt = 3h/1000 mm/min.

For walls with a load-bearing structural and thermal insulation frame (without a frame), the fire resistance limit for loss of load-bearing capacity can we taken equal to the fire resistance limit for loss of thermal insulation capacity. The temperature in the middle of the specimen is less than the critical temperature of~ 500ºC, taking into account the values of the temperature distribution across the specimen cross-section obtained by the furnace during the test.

The fire resistance limit of a structure they determined by the formula

$$t_{fr} = t_{mes} - \Delta t, \quad (3)$$

where t_{fr} – fire resistance limit, min; t_{mes} – is the minimum value of the time from the beginning of the test to the achievement of the fire resistance limit state determined by the results of tests of two identical samples, min; Δt – test error, min.

The error value Δt is determined by the formula

$$\Delta t = (0{,}015 t_{mes} + 3)\,(A_s - A_f)/(A_s - A_{min}), \quad (4)$$

where A_s, A_f, A_{min} – are the integral values (areas under the curves) of the standard temperature, the average temperature in the furnace, and the minimum permissible temperature in the furnace, respectively, ºC·min, If $A_f > A_s$, then $\Delta t = 0$.

A standard fire in the temperature range developed according to equation (5) and its evolution in time they shown graphically in Figure 4. The standard fire is an empirical model for assessing the fire resistance of structural elements of buildings:

$$\theta g = 345 \cdot \log 10(8t + 1) + \theta \quad (5)$$

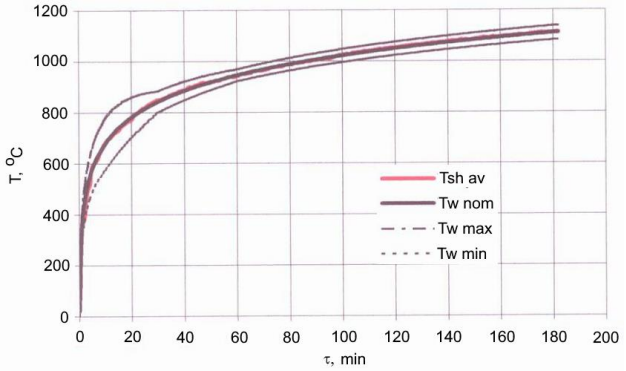


Figure 4. Temperature in the furnace

Samples made of cellular autoclaved reinforced material were tested for brittle fracture and crack resistance in the temperature range of 20-300°C using a Shimadzu Autograph AG-1C 20 kN universal testing machine equipped with a thermal chamber with a measurement error of 0.5%.

The resistance to temperatures in the range of 20-1100°C of masonry wall samples made of cellular autoclaved reinforced material we carried out in a vertical test furnace GDP-1 (Test Centre Test, LLC and Ukraine).

At the time of testing, the air temperature was 11°C with a relative humidity of 62%. The temperature and overpressure in the furnace met the requirements of the standard. The overpressure in the furnace at the fifth minute was 8 Pa, and starting from the 10th minute - 10 Pa.

3. Results

The analysis of the results given in Table 1 shows that the studied cellular autoclaved reinforced materials in terms of the value of F (brittle fracture criterion) will not brittle fracture under conditions of a sharp change in temperature from 20 to 300 °C because the value of $F \leq 4$ (Recommendations, 1979).

Table 1.

Data for calculation of the brittle fracture criterion based on the development of a main crack for cellular autoclaved materials

Simple*	α_{bt}, 1/°C	E_{bt}, mN/m^2	ρ, kg/m^3	K_1, mN·m$^{-3/2}$	P, m^3/m^3	λ, W/(m·°C)	W_o^{vom}, m^3/m^3	F
UCAM	$0.86 \cdot 10^{-5}$	$1.9 \cdot 10^3$	420	0.61	0.157	0.092	$7.5 \cdot 10^{-2}$	0.43
RCAM	$0.8 \cdot 10^{-5}$	$1.8 \cdot 10^3$	400	0.68	0.142	0.089	$7.2 \cdot 10^{-2}$	0.39

Note*: UCAM - unreinforced cellular autoclave material; RCAM - reinforced cellular autoclave material

It should we noted that the studied compositions of cellular autoclavable materials have a relatively low modulus of elasticity (Table 1), which allows reducing the value of tensile stresses in the outer protection zone associated with one-sided heating of the material.

Thus, during cyclic tests of autoclaved aerated concrete samples (Figure 1, b), at a loading rate of 0.02 m/min, the maximum destructive forces were obtained, respectively, 0.2 MPa and 0.28 MPa. The calculated stress intensity factor was (Formula 2), respectively, 0.61 mN·m$^{-3/2}$ and 0.68 mN·m$^{-3/2}$. After the calculations according to Formula 1, the F values were 0.43 and 0.39, respectively, which is significantly lower than the criterion requirements. Taking into account the data of works (Pak *et al.*, 1978; Lesovik *et al.*, 2000; Lapuk *et al*., 1978; Zaitsev, 1982; Vishnevskiy, 1958), we can say the following that the durability of the considered cellular autoclaved materials in terms of the intensity of crack formation will be approximately 20 years.

The results of temperature measurements on the unheated surface and in the cross-section of the samples we shown in Figures 5-8.

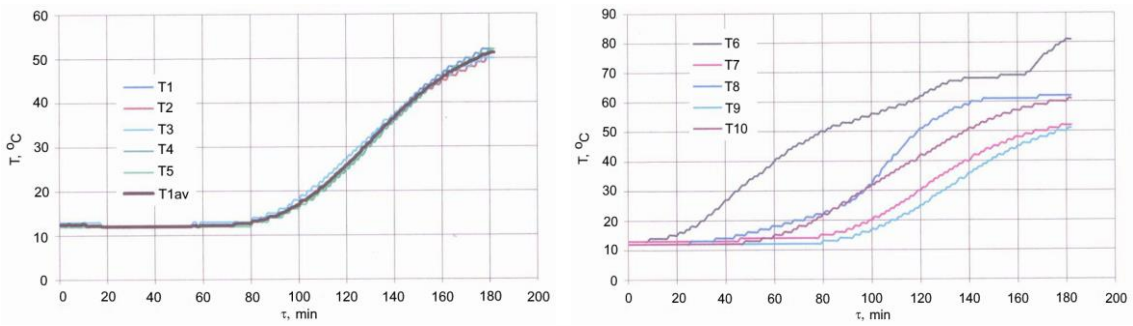


Figure 5. Temperatures of the unheated surface of sample No. 1 (T1-T10)

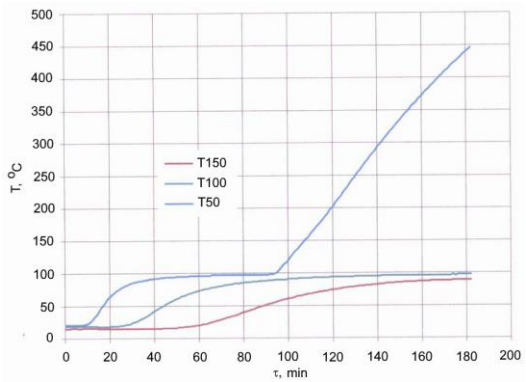


Figure 6. Sectional temperature of sample No. 1 (T50, T100 and T150)

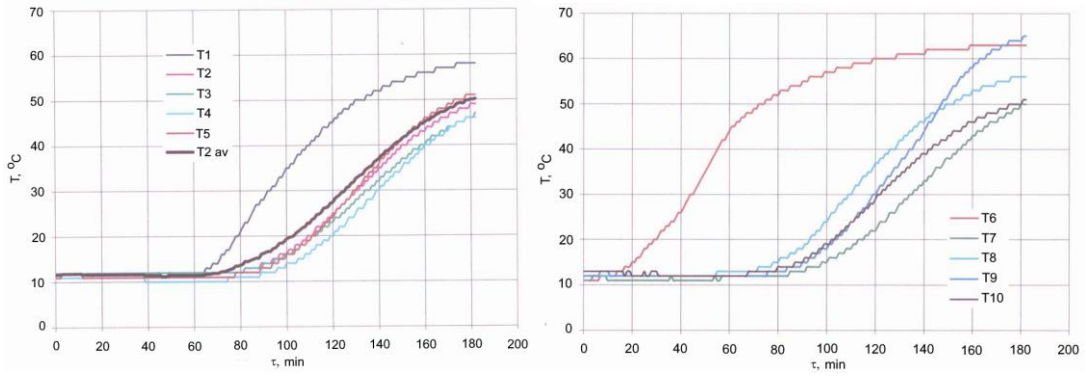


Figure 7. Temperatures of the unheated surface of sample No. 2 (T1-T10)

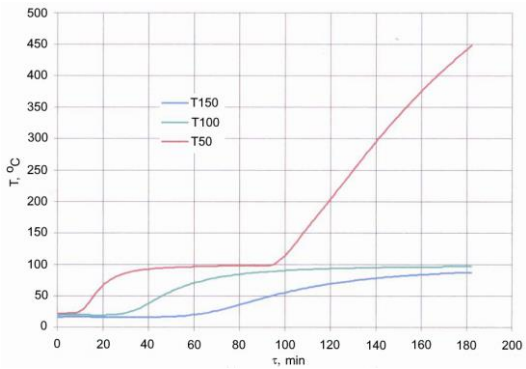


Figure 8. Sectional temperature of sample No. 2 (T50, T100 and T150)

During the tests, the maximum temperature values in the middle of the cross-section of the samples did not exceed 97°C. There was no loss of thermal insulation, load-bearing capacity or integrity of the samples.

The calculated values (Formula 4) are as follows of As, A_f, Amin for a test time of 182 min were 175074, 175212, 168560 °C·min, respectively, which provides fire resistance class REI 180 and meets the criteria of DSTU B V.1.1-19:2007, SR EN 13501-1 and P 118/99. The test error (Δt) according to formula (2) was 0 min.

It is possible to increase the temperature resistance of wall structures made of the material in question to REI 240 and above by using heat and fire-resistant dry mixes based on magnesia cement (Guzii *et al.*, 2021).

5. Conclusion

In this paper, the study of cellular autoclaved reinforced material resistant to brittle fracture and temperatures in the range of 20-1100°C. The analysis of the results shows that the studied cellular autoclaved reinforced materials by the value of F (brittle fracture criterion) will not brittle fracture under conditions of a sharp change in temperature from 20 to 300 °C because the value of $F \leq 4$. The material is characterised by a relatively low modulus of elasticity, which reduces the tensile stresses in the outer protective zone associated with its one-sided heating.

Cyclic tests of the material samples have shown that at a loading speed of 0.02 m/min, the maximum destructive force is 0.28 MPa, the calculated stress intensity factor is 0.68 $mN \cdot m^{-3/2}$, and the calculated value of F=0.39, which is significantly lower than the criterion requirements. Allows us to predict the durability of the developed reinforced

porous autoclaved material in terms of crack formation intensity for 20 years. The 200 mm thick, material is resistant to high temperatures for 182 minutes, which allows it to the classified as REI 180. During the temperature tests, the maximum temperature values in the middle of the cross-section of the samples did not exceed 97°C. There was no loss of thermal insulation, bearing capacity, or integrity of the samples.

The results obtained in this work can be use in the construction of low-rise buildings and the arrangement of walls in monolithic concrete frames.

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