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Thermal resistance of fire retardant materials

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Abstract. The results of thermal resistance of fire-retardant materials "OGNEBASALT" PMBOR and OGNEZA-GT sealant in the form of a mineral wool heat-insulating plate are presented. To study the behavior of fire-retardant materials, the method of TG-DSC synchronous thermal analysis (thermogravimetry in conjunction with differential scanning calorimetry) was used on a NETZSCH thermal analyzer. The incombustible properties of "OGNEBASALT" PMBOR have been confirmed, which showed a decrease in the mass of the material by 21 % when heated to 1000 °C. The thermo-expanding sealant OGNEZA-GT reduced its weight at the same heating with the fire-basalt material by 64 %. For a 10 % weight loss, the two test specimens require different temperatures of 395 and 262 °C, respectively. Time interval of "OGNEBASALT" PMBOR weight loss from 99.6 % to 77.6 % – from 12 minutes to 27 minutes of the experiment; for OGNEZA-GT sealant, from 99.8 % to 54.7 % – from 8 minutes to 18 minutes of testing. The best thermo-resistant properties have been revealed for the fire-retardant material "OGNEBASALT" PMBOR, which makes it possible to recommend its widespread use as a material for passive fire protection. The research results can be used to justify the choice of fire protection in buildings of various functional classes of fire hazard.

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1. Introduction

One of the elements of fire protection are refractory materials used to increase the fire resistance of building structures in the event of a fire. The objects of study were samples of refractory materials – fire retardant material "OGNEBAZALT" PMBOR (LLC "OG-NEBAZALT MSK", Moscow region, Balashikha) and fire retardant thermo-expanding sealant OGNEZA-GT in the form of a mineral wool heat-insulating plate (LLC "Ogneza", g . St. Petersburg).

A number of publications provide the results of designing and increasing the fire resistance of wood [1–12] and metal structures [13–16], studies to reduce the flammability of synthetic materials [17, 18], imparting fire retardant properties to cellulosic textile materials [19, 20]. Research work is mainly aimed at studying the properties of fire retardants, products of pyrolysis and combustion of materials, the mechanism of slowing down combustion, increasing the fire resistance of materials, assessing the flammability and fire-retardant efficiency of coatings for metal structures, intumescent fire-retardant materials [21–23]. However, data on the thermal properties of fire-resistant materials are scarce [24]. There is especially insufficient information on the properties of Russian-made refractory materials.

Purpose of the research: diagnostics of behavior under thermal influence of modern fire-resistant materials of Russian production.

Research objectives:

- study of the weight loss of fire retardant materials when exposed to temperature;
- determination of temperature ranges of sample weight loss;
- study of the microstructure of samples before and after exposure to temperature;
- determination of materials with the best heat-resistant properties.

Fig. 1 shows the investigated samples of refractory materials – the fire-protective material "OGNEBASALT" PMBOR and the fire-retardant thermally expanding sealant OGNEZA-GT in the form of a mineral wool heat-insulating plate.

a)

b)

c)

Figure 1. Investigated samples of fire retardant materials: a) side view and top view of the fire-retardant material "OGNEBASALT" PMBOR (thickness – 8 mm); b) side and top view of fire retardant thermally expanding sealant OGNEZA-GT (thickness – 30 mm); c) side view of both samples.

Fire-retardant material "OGNEBASALT" PMBOR is a fire-retardant roll-stitching material made of super-thin basalt fiber with a thickness of 5.0 to 16.0 mm, laminated with aluminum foil on one side (TU 5769-004-52876233-2009). Manufacturer – LLC "Epoch-Basalt", Bryansk region [25].

Fire-retardant basalt material PMBOR is a layer of super-thin basalt fibers, randomly arranged and bonded together in a natural way, without adding glue, but stitched with a knitting-stitching method. PMBOR is used for fire protection (building structures, air ducts) and for thermal insulation (pipelines, boilers, boiler building structures, residential and industrial buildings and structures, attic floors of houses).

The study used a sample "OGNEBASALT" PMBOR 8 mm thick. According to the manufacturer's data [25], the fire resistance limit of "OGNEBASALT" PMBOR is EI 90.

Table 1 shows the characteristics of the material "OGNEBASALT" PMBOR [25].

Table 1. Technical characteristics of the fire-retardant material "OGNEBASALT" PMBOR.

Indicator	Value
Surface density, g /m^2, no more	1800
Density, kg /m^3, no more	140
Humidity,%, nomore	2.0
Thermal conductivity, at a temperature of 25 ± 5 °C, W / (mK), no more	0.038
Application temperature, °C:	
– without lining	from –260 to +900
– covered with fiberglass	from –260 to +400
– withoutlining	NG

Fire-retardant thermally expanding sealant OGNEZA-GT in the form of a mineral wool heat-insulating plate is made on the basis of basalt rocks, has a density of 100 kg/m^3 and belongs to the fire hazard class KM0 [26]. The material is used for fire protection and thermal insulation of reinforced concrete floor slabs, for sealing expansion (structural joints), cable and ventilation ducts of communications through walls and ceilings. The principle of operation of the sealant is based on its ability to expand under the influence of high temperatures (from 200 °C) and create a dense non-combustible layer of coke, which prevents the penetration of fire and smoke into adjacent rooms.

2. Methods

To study the behavior of fire retardant materials under temperature exposure, experiments were carried out using the method of synchronous thermal analysis (STA) (Russian State Standard GOST R 55134-2012), including differential scanning calorimetry (DSC) and thermogravimetry (TG).

The thermal analyzer is a thermal balance (digital, high-sensitivity, high-resolution) with top loading of the sample and direct measurement of the temperature on the sample. In these devices, a sample of a substance (mg) is heated at a given temperature regime, at a given rate in an inert gas atmosphere, the decrease in the mass of the substance (thermogravimetric analysis, TG or TG) and exo- and endo-effects (differential thermal analysis, DTA or DTA (DSC – Differential Scanning Calorimetry)).

The study by the STA method was carried out at the Department of Fire Safety of the Federal State Budgetary Educational Institution of Higher Education "Academy of Civil Protection" on a thermal analyzer STA 449 F3 Jupiter of the German company "NETZSCH" (Fig. 2).

Figure 2. Synchronous thermal analyzer STA 449 F3 Jupiter from NETZSCH.

The main technical characteristics of the thermal analyzer are shown in Table 2.

Table 2. Specifications STA 449 F3 Jupiter.

Indicator	Value
Temperaturerange, °C	–150 – 2400
Maximumsampleweight, g	35
Heating and cooling rate, °C/min	0.001 – 50
TG resolution, µg	0.1
DTG resolution, µW (depending on the type of sensor)	< 10

The results of the analysis of the thermal analyzer are recorded in the form of a graphical dependence:

- TG (integral curve) – "change in sample mass,%, – heating duration, min";
- DSC (thermal effects) – "exo- and endo-effects, microV/mg, – heating duration, min". The latter effects can arise as a result of chemical processes, phase transitions, thermal destruction of a substance, etc. in the sample under study;
- dDSC – "rate of change in the mass of samples under thermal exposure, microV/mg/min – heating duration, min".

The method of differential scanning calorimetry (DSC) allows to study with high accuracy the properties of substances under thermal influence [27].

Characteristics of the experimental conditions:

- measurement mode – TG / DCS / dDSC;
- heating rate: 20 °C/min;
- heating – up to 1000 °C;
- atmosphere – N_2.

The microstructures of the samples of fire retardant materials before and after exposure to temperature were investigated using an electron microscope of the Department of Chemistry and Materials Science of the FGB-VOU VO "Academy of Civil Protection".

3. Results and Discussion

Fig. 3–6 show the TG /DCS /dDSC curves of the investigated fire-retardant materials, reflecting the rate of weight loss of the samples and the accompanying thermal phenomena, obtained using a NETZSCH STA 449 F3 Jupiter thermal analyzer. Fig. 3, 5 show the dynamics of the processes occurring with the samples in relation to temperature; Fig. 4, 6 – in time.

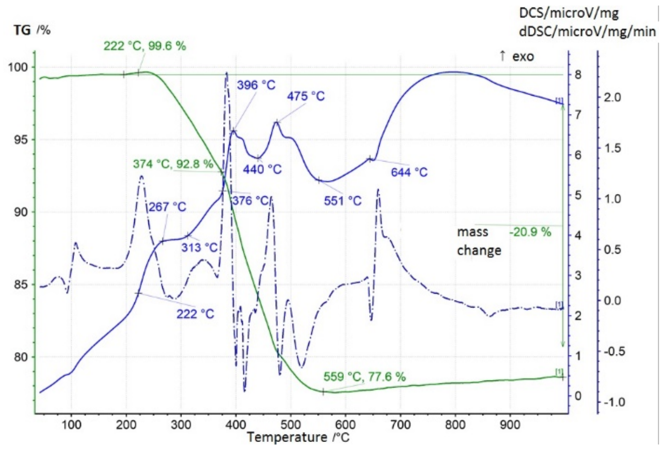


Figure 3. Thermogram of the fire retardant material "OGNEBASALT" PMBOR (by temperature).

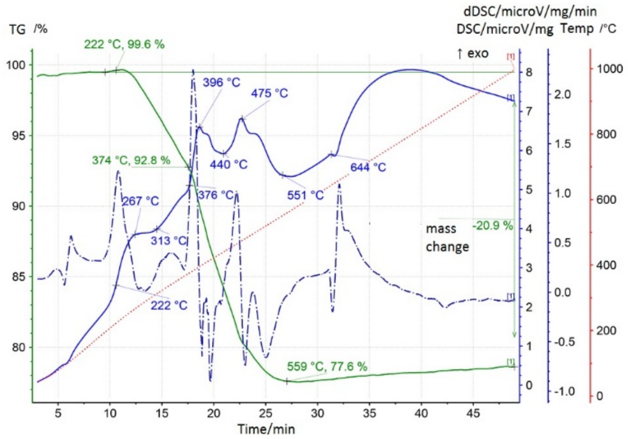


Figure 4. Thermogram of fire-retardant material "OGNEBASALT" PMBOR (by the time of temperature exposure).

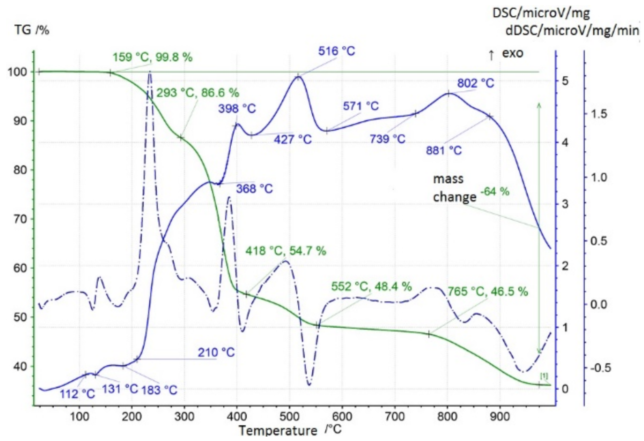


Figure 5. Thermogram of fire retardant thermally expanding sealant OGNEZA-GT (by temperature).

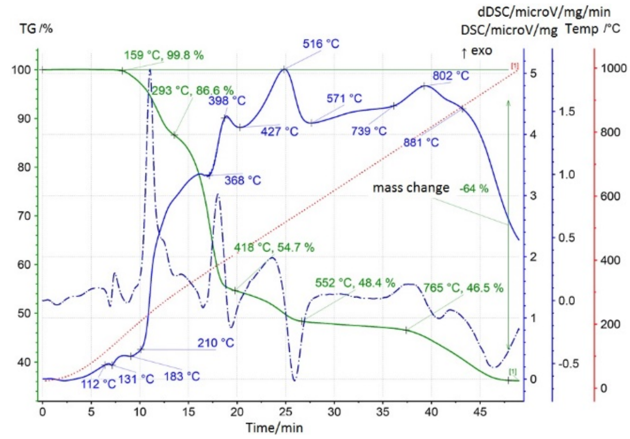


Figure 6. Thermogram of fire retardant thermally expanding sealant OGNEZA-GT (by the time of temperature exposure).

As can be seen from Fig. 3-6, the behavior of fire-retardant materials under temperature exposure differs from each other, however, the non-combustible properties of the fire-retardant material "OGNEBASALT" PMBOR, declared by the manufacturer, are confirmed as a non-combustible material, in which the mass of material when heated to 1000 °C decreases by 21 %. The thermo-expanding sealant OG-NEZA-GT reduced its weight with the same heating as the fire-basalt material by 64 %.

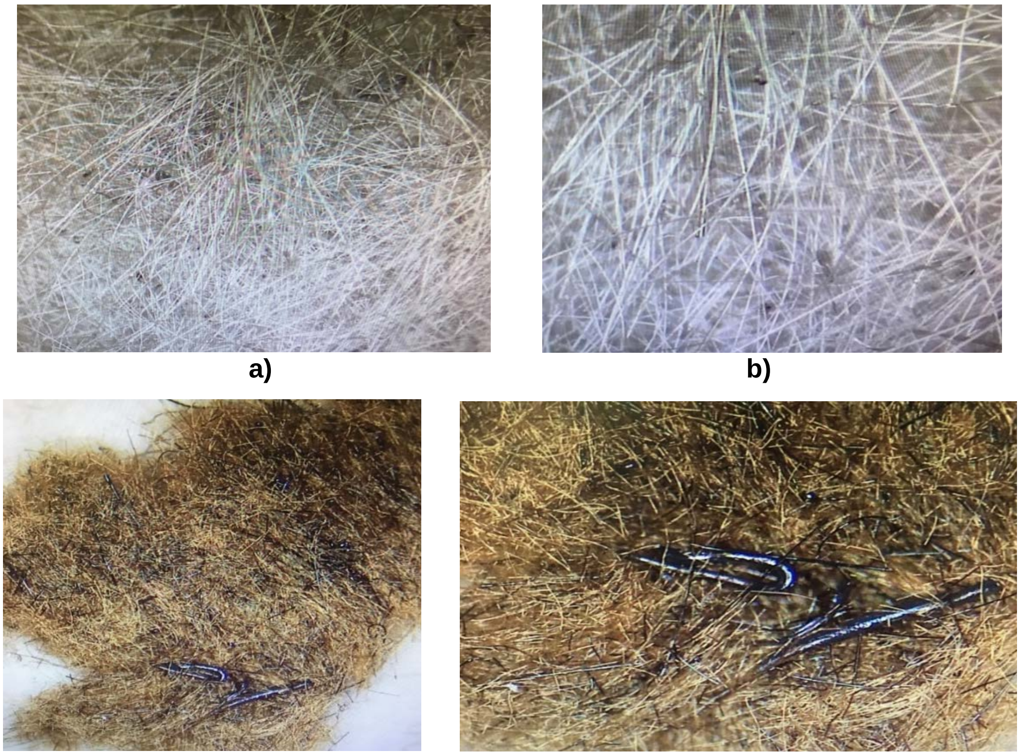
On the curve of the thermogram of the fire-retardant material "OGNEBASALT" PMBOR (Fig. 3), it can be seen that up to a temperature of 222 °C, the sample weight decreases by only 0.4 %, while a slight exothermic effect is observed, the maximum of which is at 267 °C. Upon further heating to a temperature of 313 °C, a series of small exo effects begins with maxima at 396 °C and 475 °C and an endo effect with a minimum at 551 °C, and then an exo effect begins at 644 °C. Thus, it can be assumed that in the temperature range of 222–559 °C, thermal oxidative destruction of the organic part of the sample occurs, followed by spontaneous ignition of its combustible part, accompanied by a decrease in mass by 21 %; however, upon further heating, no loss of mass occurs, which is due to the mineral composition of the sample (silica based material).

Fig. 4 allows you to trace the behavior of the fire-retardant material "OGNEBASALT" PMBOR under temperature exposure in time: the weight loss of the sample by 0.4 % occurs from 12 minutes of the experiment, when the temperature of 222 °C is reached, and up to 27 minutes (weight loss 22.4 %) ... By the 50th minute of the experiment, the material retains 79.1 % of its mass.

Analyzing the thermogram of the fire retardant thermally expanding sealant OGNEZA-GT (Fig. 5, 6), the following conclusions can be drawn. The sample does not lose weight until it reaches a temperature of 159 °C, while insignificant exothermic effect at 112 °C and an endothermic effect at 131 °C are observed. At 210 °C, a powerful exothermic effect begins with a decrease in weight. At 418 °C, only 54.7 % of the sample weight is retained. It can be assumed that thermal oxidative decomposition of the organic part of the material occurs in the temperature range 159 – 418 °C.

The time interval of weight loss from 0.02 % to 45.3 % was determined from 08 minutes to 18 minutes of testing, which can be presumably associated with the thermal expansion of the structure (which is consistent with [24]). Endoeffects at 427 °C and 571 °C can be explained by the consumption of heat for the process of melting substances. At a temperature of 802 °C, 39 minutes after the start of the test, an exo effect occurs, which is probably due to spontaneous ignition and burnout of an additional 15 % of the sample mass. The total decrease in the mass of the sample by the 50th minute of the experiment was 64 % when the temperature reached 1000 °C (end of the experiment).

Fig. 6 a, b show the images of the original structure of the material "OGNEBA-ZALT", obtained using an electron microscope with a magnification of 15 and 30 times. Fig. 6 c, d show the structure of material residues after a fire exposure of 1000 °C (an increase of 15 and 30 times).



a) b)

c) d)

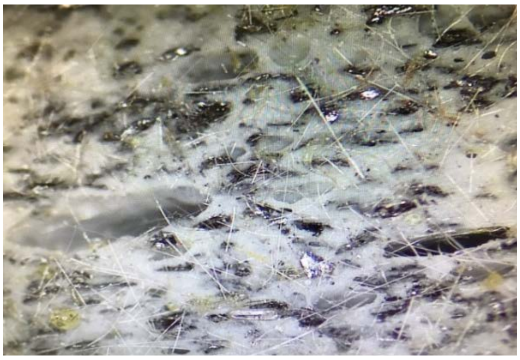
Figure 6. Micrographs of samples of fire-retardant material "OGNEBASALT" PMBOR (a, b) and their residues in the crucible after exposure to fire after (c, d): a – fire retardant material "OGNEBASALT" PMBOR (increase 15 times); b – fire-retardant material "OGNEBASALT" PMBOR (30 times increase); c – the remainder of the sample "OGNEBASALT" PMBOR after exposure to fire at 1000 °C (15 times increase); d – the remainder of the sample "OGNEBA-ZALT" PMBOR (increase in 30 times).

As can be seen from Fig. 6 a, b, the fire retardant material "OGNEBASALT" PMBOR is a chaotically located layers of fibers. After a fire exposure of 1000 °C (Fig. 6 c, d), the fibrous structure of the material is preserved, which confirms the non-combustible properties of the material. In this case, clearly expressed, few, sintered structures are formed (presumably, aluminum foil, which is lined with the original material).

OGNEZA-GT sealant is a two-layer material consisting of dense and fibrous parts. Fig. 7a shows an image of a two-layer structure of the OG-NEZA-GT sealant with an increase of 15 times; Fig. 7 b is a micrograph of the dense part of the OGNEZA-GT sealant (magnification 45 times). Fig. 7 c, e allows you to see the fibrous part of the OGNEZA-GT sealant (magnification 30 and 60 times). Fig. 7 f and e – photomicrograph of the remainder of the sealant sample after exposure to fire at 1000 °C (magnification 45 and 60 times).

Fig. 7a clearly shows the two-layer structure of the fire-retardant thermally expanding sealant OGNEZA-GT. Fig. 7 b shows the porous structure of the dense part of the sealant, presumably of organo-mineral origin. Fig. 7 c, d show the fibrous part of the sealant. After a fire exposure of 1000 °C (Fig. 7e,f), the fibrous structure of the material is preserved, providing the integrity of 36 % of the sealant structure. Presumably, the dense part of the sealant completely burned out, turning into small sintered fragments, clearly shown in Fig. 7e, f. The porous structure of the dense part of the sealant, due to its high permeability, was intensively exposed to temperature until complete burnout.

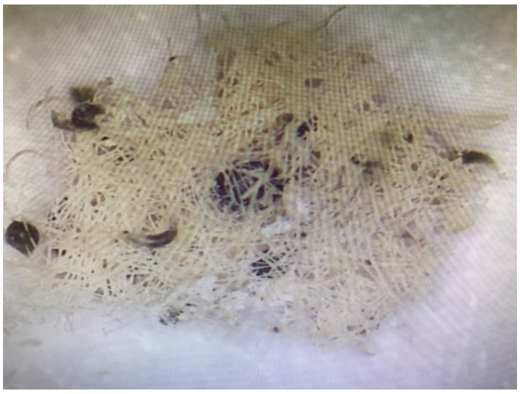
a)



b)

c)

d)



e)

f)

Figure 7. Micrographs of a sample of fire retardant thermally expanding sealant OGNEZA-GT (a, b, c, d) and its remainder in the crucible after exposure to fire at 1000 °C (e, f): a – OGNEZA-GT sealant (15 times increase); b – the dense part of the OGNEZA-GT sealant (magnification 45 times); c – fibrous part of the OGNEZA-GT sealant (increase by 30 times); d – the fibrous part of the OGNEZA-GT sealant (60 times increase); f – the remainder of the sealant sample after exposure to fire (45 times increase); e – the remainder of the sealant sample after exposure to fire (60-fold increase).

Table 3 shows the reduction in the mass of the tested samples of fire retardant materials when heated.

Table 3. Characteristic of reducing the mass of the sample of the sealant under fire exposure.

	Weight loss of samples,% (upon reaching temperature, °C)									
	10	20	30	40	50	60	70	80	90	100
Fire retardant material "OGNEBASALT" PMBOR	395	481	Starting from a temperature of 559 °C, no change in mass occurs. Theoverallweightlossis 21 %.							
OGNEZA-GT sealant	262	338	364	382	516	888		No mass loss occurs. The overall weight reduction of the sample is 64 %.		

Based on the data in Table 3, it can be concluded that for a 10 % weight loss, the two test samples require different temperatures – 395 and 262 °C, respectively. For the sealant, weight loss of 20, 30, 40 % occurs with a "step" of 76, 26, 18 °C, respectively, which adversely affects the fire-retardant characteristics of the material.

4. Conclusions

1. The incombustible properties of the fire-retardant material "OGNEBASALT" PMBOR have been confirmed.

2. The material "OGNEBASALT" PMBOR, when heated to 1000 °C, reduced its weight by 21 %, the OGNEZA-GT sample – by 64 %.

3. The temperature and time intervals of changes in the properties of the investigated fire-retardant materials have been established.

"OGNEBASALT" PMBOR begins to respond to the effect of temperature by weight loss at 222 °C at the 12th minute of the test, OGNEZA-GT – at 159 °C at the 08th minute of the test.

In the temperature range of 222–559 °C (from 12 minutes of the experiment to 27 minutes), thermal oxidative destruction of the organic part of the sample "OGNEBASALT" PMBOR occurs by 21 %. With further heating, the material is resistant to heat and does not lose weight.

In the temperature range 159–418 °C (from 08 minutes of the experiment to 18 minutes), a loss of 45.3 % of the mass of the OGNEZA-GT sample occurs due to thermal oxidative destruction of the organic part. The decrease in the mass of the sample by the 50th minute of the experiment was 64 % when the temperature reached 1000 °C (end of the experiment).

4. The microstructure of materials has been investigated. "OGNEBASALT" consists of randomly arranged layers of fibers. After exposure to a temperature of 1000 °C, the fibrous structure of the material is preserved. OGNEZA-GT sealant is a two-layer material consisting of dense and fibrous parts. After exposure to 1000 °C, the fibrous structure of the material is preserved, ensuring the integrity of 36 % of the structure. Presumably, the dense part of the sealant has completely burned out.

5. The best heat-resistant properties were found in the fire-retardant material "OGNEBASALT" PMBOR, which allows us to recommend it as a material for passive fire protection. The research results can be used to substantiate the choice of fire protection in buildings of various functional classes of fire hazard.

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