

Design and research of thermal protective material from short basalt fibres

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Abstract. Design and manufacture issues regarding highly porous thermal protection coatings of products by means of liquid filtration of short basalt fibres and mineral binder are considered. The technological process of manufacture of thermally loaded products from the short basalt fibres of thermal protective material (TPM) in the form of tiles and rings, was developed based on a liquid filtration method. The structural and mechanical properties of the highly porous TPM technological modes were determined. The thermal testing of the pipe model samples was carried out on a thermal bench, which showed the temperature on the coating reaching less than 60°C during a hot air run through the pipe at 400°C.

1. Introduction

Rocket and spacecraft structures are under considerable thermal load both during operation in orbit, and passage through dense atmospheric layers. To prevent rocket and spacecraft structures from overheating, the complex system of thermal protection, consisting of 4 layers, is used: a solid layer of metal or composite materials; a layer of material executing heat insulation at the expense of low heat conduction; a layer of char-forming material in which endothermal reactions take place; and a layer of material which is carried away with incoming flow and creates radiative energy flow from rocket and spacecraft surfaces. Therefore, the creation of highly porous, low-density, and environmentally friendly thermal insulation from available and low-cost basalt fibres for rockets and spacecraft operation at temperatures up to 700°C, is an important scientific and engineering task [1, 2].

As a material for heat insulation coating of rockets and spacecraft items operating for a long time at temperatures up to 400°C and more, the thermal-protective material of TZMK-10 grade can be used [1]. The material is based on fine quartz fibre with a diameter of 1.5 – 2.0 µm, and was developed for the heat protection tiles for the spacecraft “Buran”. The disadvantages of the material TZMK-10 are the high cost of its production and the high coefficient of thermal conductivity (0.06 W/(mK)) at normal temperature.

A cheaper but no less effective heat insulation can be produced with short basalt fibre. This thermal-protective material (TPM) is comprised of superthin (diameter 0.5 – 3.0 µm) basalt fibres (BSTF) with alumina binding material, and its heat insulation is based on glass microspheres of MC-БП-A9 [MS-VP-A9] grade and organosilicate compositions OC-11-07 [OS-11-07]. It was deduced from experiments [3 – 5] that when heated to temperatures of 400-500°C, this material would have a low coefficient of thermal conduction, in the mean equal to 0.041-0.042 W/(mK).

Spheroplastics have double the density in comparison with the heat insulation made of the short basalt fibres with mineral binder, but at high temperature and over long-term heating, the



spheroplastics are charred and cracked. In this case, TPM based on basalt fibre, demonstrates high cycle fatigue strength, maintains its shape quite well, and its chemical stability is significantly higher in comparison with that of heat insulation made from spheroplastics. The main method of manufacture of short basalt fibres based TPM is a method of fibre filtration deposition from liquid suspension (pulp), which is used in production with thermal protective tiles and short cylindrical rings [2, 6].

The objective of this paper is to determine parameters for the manufacture process of high porosity, thermal insulating coatings of spacecraft structures, from short basalt fibres and mineral binder, by filtration deposition.

2. Filtration analysis

The technology for the production of short-fibre heat-insulating coatings involves a combination of individual operations including cleaning and fragmentation of the original fibres, preparation of hydro-mass (pulp) from the short fibres, and liquid formation and thermal processing of the product. In this case, the process of filtration deposition of fibres onto a production rig determines the coating material structure and, consequently, its thermo-physical and mechanical characteristics.

The filtration rate depends on the hydro-mass properties – the temperature and operating fluid viscosity, the sizes of the fibres, and the shape of the solid phase particle – binder. The filtration conditions are determined by the hydrostatical pressure value, the filtrate chamber vacuum treatment, and the cake layer porosity value. Utilization of low pressures for pulp with low fibre content improves the structural, thermal and physical characteristics of the moulded product, at the expense of the fibre distribution over material volume.

The mathematical modelling of the moulding process of the short-fibre material heat-insulating structure should be based on the physical principle of liquid flow through a porous body. In this case, the model constants, dimensional or non-dimensional parameters should be used. They are easily determined experimentally and are dependent on the physical properties of the liquid and structural characteristics of fibrous material.

The liquid moulding process for the manufacture of rocket and spacecraft pipes comprised of short basalt fibre heat insulation material is time consuming. The determination, expressed in analytical form, of factors affecting the filtration rate and their inter-dependence, is an important task for the development of the manufacturing process of products based on the short-fibre compositions.

The basis of filtration physico-mathematical analysis is a description of the liquid flow (filtrate) into the pores between fibre layers. The true speed of fluid flow into the pores between fibre layers and binder particles, is difficult to determine since it is impossible to take into account the size and configuration of each pore. Therefore, instead of the true speed of the liquid flow, its conditional value, called the filtration rate, is used.

The filtration rate, or fluid flow, $u_f(t)$, through the porous layer of the already deposited fibres (figure 1), is determined by Darcy's law [7]

$$u_f(t) = \frac{K_p p}{\eta_{liq} h_{layer}}, \quad (1)$$

where K_p (m^2) is the coefficient of liquid permeability through a porous medium; p (Pa) is the pressure differential on the fibre layer; η_{liq} (Pa·s) is the dynamic viscosity of the filtered fluid; h_{layer} (m) is the thickness of the fibre sediment layer.

In equation (1), if we substitute the pressure, p , for the sum of the liquid column height and vacuum pressure Δp of the filtrate chamber, and the current set of the sediment layer thickness via liquid volume filtered through the perforated partition $h_{layer} = c_w(H - h)/\rho_{layer}$, then we obtain a differential equation that determines the filtration time of the pulp:

$$u_f(t) = \frac{K_p(\rho_p g h + \Delta p) \rho_{\text{layer}}}{\eta_p c_w (H - h)} = \frac{dh}{dt}. \quad (2)$$

Here ρ_p (kg/m³) is the density of the fluid (pulp); g (m/s²) is the acceleration due to gravity; h (m) is the current height of the liquid column; ρ_{layer} (kg/m³) is the density of the dry sediment, for the average layer thickness; c_w (kg/m³) is the weight content of fibre in the pulp; and H (m) is the initial height of the liquid column (hydro mass, suspension, pulp).

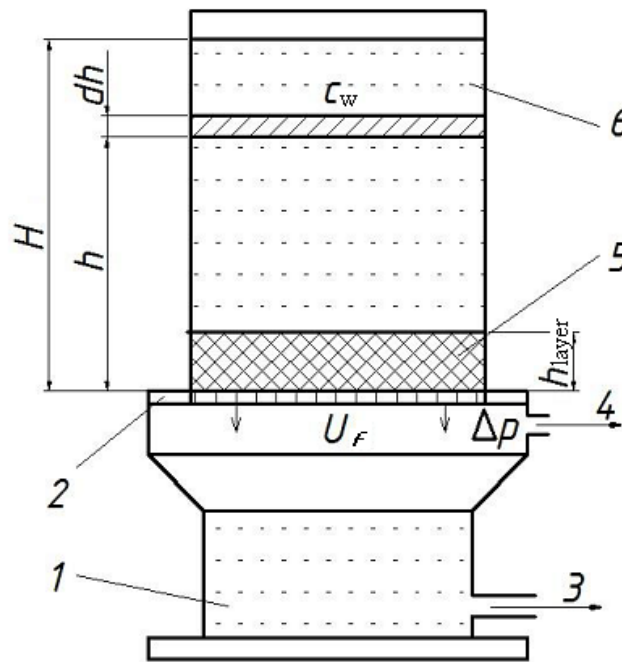


Figure 1. Schematic showing the filtration deposition of short basalt fibres of pulp onto a perforated mandrel: 1 - filtrate; 2 – perforated partition; 3 – filtrate drain; 4 – chamber vacuum treatment; 5 – fibrous sediment layer; 6 – pulp

After the integration of equation (2) we investigate the time required for the deposition of fibres from the pulp according to the scheme shown in Figure 1:

$$t_f = \frac{\eta_p h_{\text{layer}}}{K_p \rho_p g} \left[\frac{h_{\text{layer}}}{H} + (1 + \Delta p_H) \ln \left(\frac{1 + \Delta p_H}{h_{\text{layer}}/H + \Delta p_H} \right) - 1 \right]. \quad (3)$$

In equation (3) $\Delta p_H = \Delta p / H \rho_p g$.

If the filtrate chamber vacuum treatment is neglected, *i.e.* $\Delta p = 0$, then from (3) one can conclude

$$t_f = \frac{\eta_p h_{\text{layer}}}{K_p \rho_p g} \left[\frac{h_{\text{layer}}}{H} + \ln \frac{H}{h_{\text{layer}}} - 1 \right]. \quad (4)$$

According to the filtration equation the pulp volume is determined and the productivity of the product moulding process of the designed manufacturing equipment is calculated. The majority of the filtration time corresponds to operation at lower concentrations of pulp and a lower hydraulic pressure.

In equations (1-4) the coefficient of permeability K_p is the permeability relative to fluid through a porous medium under the action of an applied pressure gradient, it has the dimensions of area, and a value of the order of the square of the size of a typical pore. The coefficient of permeability is a structural feature by its nature, and is determined mainly by the geometry of the pore space. According to Kozeny [8], it depends on the specific volume of the open pores $n = V_p/V_{\text{layer}}$ cubed, divided by the square of the fibre specific surface s_y , slowly streamlined by liquid:

$$K_p = \frac{cn^3}{s_y^2} = \frac{cn^3}{s_0^2 v_f} = \frac{cd_f^2 n^3}{16(1-n)}. \quad (5)$$

Here V_p (m^3) is the volume of pores in the sediment layer; c is a dimensionless quantity: for cylindrical pores $c = 0.5$ and for pyramidal pores $c = 0.597$; $n = 1 - p_{\text{layer}}/p_f$ defines the porosity of the fibrous media; p_f (kg/m^3) is the density of the fibre; s_0 (m^2) is the specific surface of the fibre unit length, $s_0 = s_f/V_f = 4/d_f$; d_f (m) is the average (effective) diameter of the fibres; and $v_f = 1 - n$ is the fibre volume content in the sediment layer. In equation (5), recorded later by Karman [9], the value of $(1 - n)$ was taken in the second degree. Although squared, in our opinion, it should only take account of the specific surface of the fibre s_0 , but not their volume fraction in a porous material. We have observed that, when a large porosity, for example, $n = 0.9K_p$, is calculated according to the Karman formula (equation 5) and determined experimentally, there is a difference of almost an order of magnitude. For porosity of $n = 0.30 - 0.35$, specific to structural TPM, these differences are not more than 30% and with some variations of fibre diameters in composite material, they become hard-to-find in K_p calculations.

To assess the applicability of formula (5), we have conducted experimental studies to determine the permeability coefficient of the fibrous samples, using service water on a special plant. TPM model samples of $A = 100$ (cm^2) of the same surface density (weight) $\gamma^* = p_f(1 - n)h_{\text{layer}} = 0.60$ (g/cm^2) and made of quartz zero-twist yarns with a linear density of 104 (mg/m) and where the in yarn fibre diameter is 12 microns. Short fibres were produced by cutting yarn in a special device into segments with a length of 6.7 mm, 13.4 mm and 20.1 mm. The quartz fibre density was $p_f = 2.13$ (g/cm^3).

During liquid impregnation of the fibrous samples, the pressure difference across the layer Δp was measured by vacuum gauge. The liquid volumetric flow rate Q was measured by flow meter and layer thickness h_{layer} was also measured, and the porosity of the sample was calculated according to formula

$$n = 1 - M_{\text{sample}}/(Ap_f h_{\text{layer}}), \quad (6)$$

where $M_{\text{sample}} = A\gamma^*$ is the sample mass. According to the water flow experimental results, the experimental values of the permeability coefficient were calculated by the equation (1), and can be written as:

$$K_p = \frac{Q\eta_{\text{liq}}}{A(\Delta p/h_{\text{layer}})}. \quad (7)$$

3. Experiments

Figure 2 presents the analytic dependence of the permeability coefficient of the porous fibrous material, calculated using formula (5), and experimental values of the porosity n and the coefficient K_p , calculated using formulas (6) and (7). Figure 2 demonstrates that the dependence calculated according to equation (5) is reasonably consistent with the results of the fibrous samples obtained from the water flow experiment. In figure 3, the experimental dependence of samples porosity of the cut

fibre length is presented. From figure 3, it is shown that the shorter the fibres, the greater material porosity.

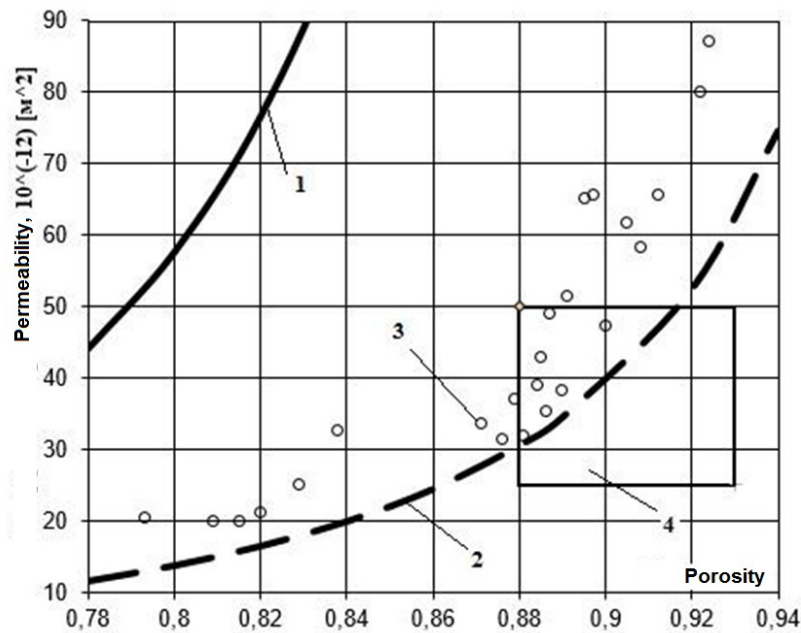


Figure 2. The dependence of the permeability coefficient K_p of the cut fibre samples on the material porosity n : 1 – by the formula of Kozeny-Karman; 2 – by formula (5); 3 – experimental values; 4 – the results area of experimental studies on glass fibre samples [8]

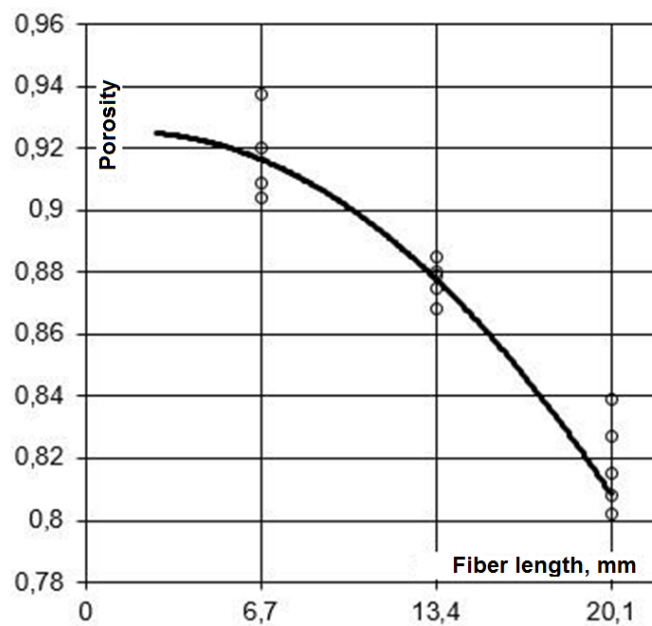


Figure 3. The dependence of the sample porosity on the length of the short fibre

At the same time the fibre length cannot be too small, less than $l_f = 1.0 - 1.5$ mm, because during the filtration deposition of very short fibres they can partially lie down at an angle or perpendicular to the sample or product plane. This reduces the strength of the composition for compression, and the material porous structure becomes unstable. On the other hand, with fibres of decreasing length (figure 3) the TPM porosity increases, thereby reducing the material thermal conductivity of the material. To obtain a fibre length of $1.0 - 1.5$ mm, a further shortening of the original cut fibres is carried out in propeller stirrer. The relationship between fibre length and processing time is shown in figure 4. When the concentration of fibres in water is $c_w = 15 - 20$ (g/l) and the rotation speed of mechanical stirrer is 1200-1500 rpm, the disintegration time of the quartz fibres and basalt fibres is 3 – 5 minutes and 8 – 15 minutes, respectively.

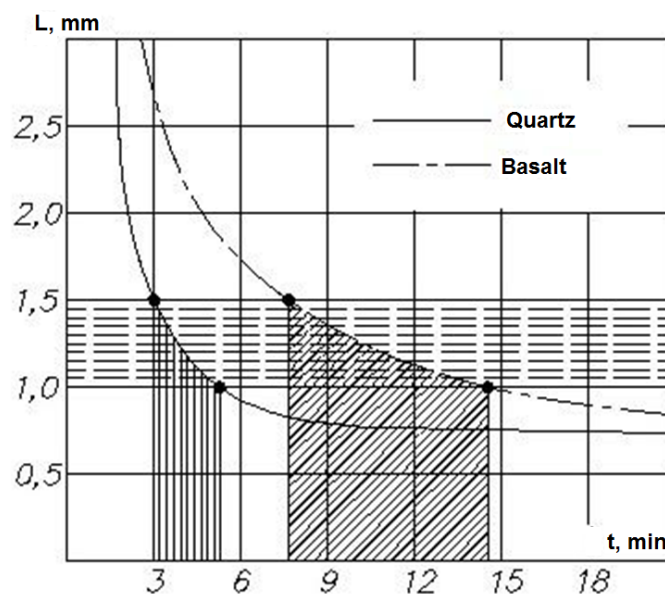


Figure 4. The relationship between fibre length and processing time:
1 – silica fibre; 2 – basalt fibre

The choice of the minimum possible diameter of fibre for the basalt TPM manufacturing is constrained by the fact that for a diameter of less than $3 \mu\text{m}$ [10] the thermal conductivity becomes too low (figure 5). Besides, with small diameters, the thermal resistance of the material increases at the expense of the specific surface increase; however, when the fibre diameter is less than $d_f \leq 1.5 - 2.0 \mu\text{m}$, and when the wavelength of the thermal spectrum becomes comparable to its diameter, the electromagnetic wave can bend around the fibre. As a result, the composition becomes transparent for thermal radiation and consequently the fibrous composition thermal conductivity on the whole will be increased. With this in mind, for spacecraft structures with insulated coatings the basalt superthin fibre-canvas BSTF "MINOL" [11] with the fibres diameter $d_f = 0.5$ to $3.0 \mu\text{m}$ and the fibres length in the canvas $l_f = 50-60$ mm is selected.

To determine the thickness of the insulation coating for the spacecraft, the average coefficient of the TPM thermal conductivity, is required and this depends on the porosity of the fibrous structure. Neglecting the Stefan-Boltzmann radiant heat loss, and heat loss for calm air in the insulating material pores, its thermal conductivity coefficient can be written based on the mixtures principle

$$\lambda(n, T) \cong \lambda_{TPM}^{ev} = n\lambda_{air}^{ev} + (1 - n)\lambda_{BF}^{ev}, \quad (8)$$

where the average values of the coefficients $\lambda_{BF}^{ev} = 0.0587 \text{ W/(m}\cdot\text{K)}$ and $\lambda_{air}^{ev} = 0.0413 \text{ W/(m}\cdot\text{K)}$ for temperature range from 60°C to 400°C are defined in [3]. Then, for material with porosity $n = 0.92$ to 0.94 , the average coefficient of TPM thermal conductivity is $\lambda_{TPM}^{ev} = 0.0425 \text{ W/(m}\cdot\text{K)}$.

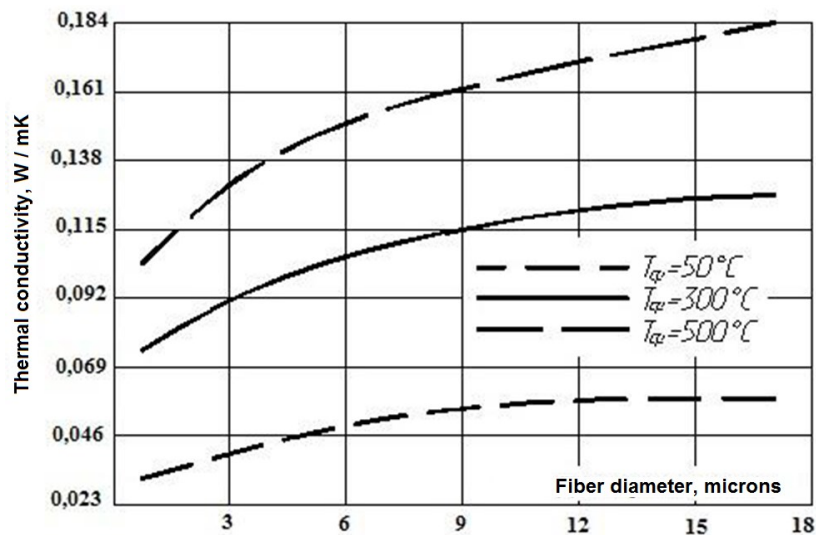


Figure 5. The dependence of the basalt fibre thermal conductivity of their diameter at different temperatures

Considering that most of rockets and spacecraft have a cylindrical form, and accepting that the heat flow along the tube length of $q_1=150 \text{ W/m}$ is inconsequential, the required HIC thickness of the basalt fibres can be pre-calculated using the formula

$$\frac{q_1}{\pi d} = \frac{2 \lambda_{TPM}(T_1 - T_2)}{d \ln \frac{d+2h_{TPM}}{d}}, \quad (9)$$

where d is the rocket or spacecraft outer diameter, $d = 60 \text{ mm}$; $T_1=400^\circ\text{C}$ – the temperature on the inner wall of rockets and spacecraft; $T_2=60^\circ\text{C}$ – the temperature on the outside of the basalt cover; and h_{TPM} is the thickness of the heat insulation of the spacecraft. By substituting λ_{TPM}^{ev} from (8) into formula (9), we will define the thickness of spacecraft structure heat insulation: $h_{TPM} = 25\text{mm}$.

To increase the compressive strength of the high-porosity fibrous TPM, the high-temperature mineral binder is used, which fixes the fibres at the sites of their contacts. The selected binder, aluminium oxide Al_2O_3 , is derived from salts of aluminium sulphate in aqueous solution of ammonia, presented in hydro mass in a colloidal condition, which increases the liquid pulp and the filtrate viscosity.

The hydro mass prescription composition, for the manufacture of thermal insulation products, is as follows: 0.95 kg of the short basalt fibre, 0.4 l of 27 % $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ solution of aluminium sulphate, and 0.07 l of 25 % $6\text{NH}_4\text{OH}$ aqueous ammonia solution. The formation of aluminium oxide occurs in the process of drying and heat treatment of the product at a temperature of 300°C . In the process of filtration deposition the amount of pulp acidity must be strictly controlled: it is necessary to perform hydrolysis and deposition of the fibres at $pH = 7.6$ to 8.4 . In this case, one can ensure the calculated amount of aluminium oxide in the heat insulation material in the form of binder, while maintaining a sufficiently high filtration rate.

During fibre-free filtration deposition from pulp, the time for the accumulation of a sediment layer of height up to 100 mm (to the cylindrical bushing) is not less than 90 min, according to formula (4),

and in this case, the problem of material thickness equidensity and the product surface geometry is not fully solved. The dynamics of the hydro mass column reduction as a function of time is presented in figure 6.

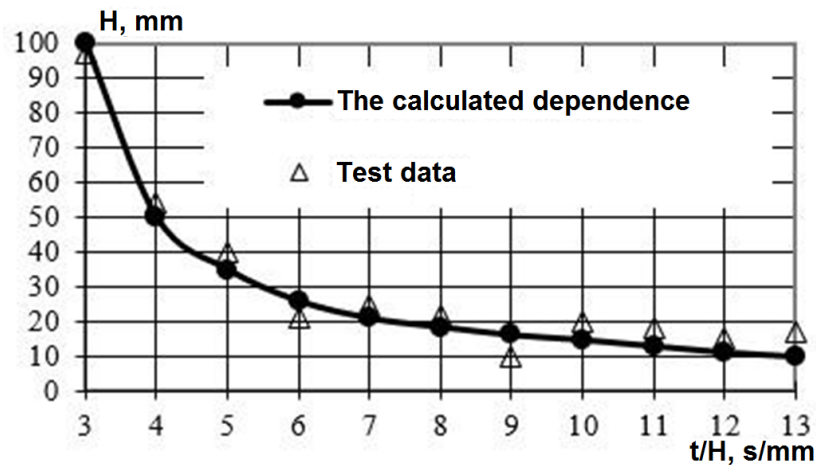


Figure 6. The dependence of hydro mass column height of the time during free filtering

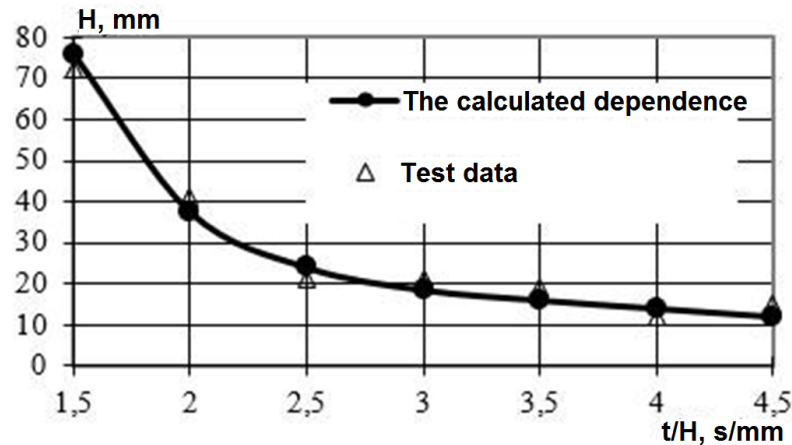


Figure 7. The dependence of hydro mass column of the time with vacuum treatment and pre-moulding

The efficiency of the process increases significantly as a result of filtrate chamber vacuum treatment (figure 7). In this case, the value of Δp in formula (3) is a free parameter. The pressure of vacuum treatment should be chosen in such a way that the thickness and density of the sediment layer would not be less than the designed (accepted) values. In this case, the fibre filtration deposition time should be less than that of the hydro mass layering on liquid and solid phases. In fact, equation (3) can be considered as the time control law for sediment layer filtration and density for the manufacture of products from fibrous heat insulating material with a predetermined thickness h_{layer} .

Cylindrical rings, manufactured by fibre-free filtration deposition from pulp, with wall height of more than 50-60 mm, have a material density variation in respect of height of up to 12-15%. To reduce the material inhomogeneity, it is necessary to apply a method of liquid filtration with the filtrate chamber vacuum treatment, and then finished pre-moulding of the sediment layer by means of punch, to form the final geometric size and shape of the product. Constant pressure difference across the sediment layer under vacuum treatment can be steadily maintained in the range of $\Delta p=50$ to 70 kPa.

To reduce the manufacturing time of the long cylindrical shells made of the short basalt fibres by liquid filtration, it is necessary to apply a moulding process using the plane generating cylinder shown in figure 8. Initially, for a short time (7-10 min), the deposition of the main volume of fibres takes place, and then, without interrupting the process, the mandrel-punch is introduced, and fibre filtering and the TPM lock moulding ends. With such a scheme of filtering, the pulp column height and the product manufacturing time decreases sharply. Additionally, the design of a manufacturing jig is simplified significantly.

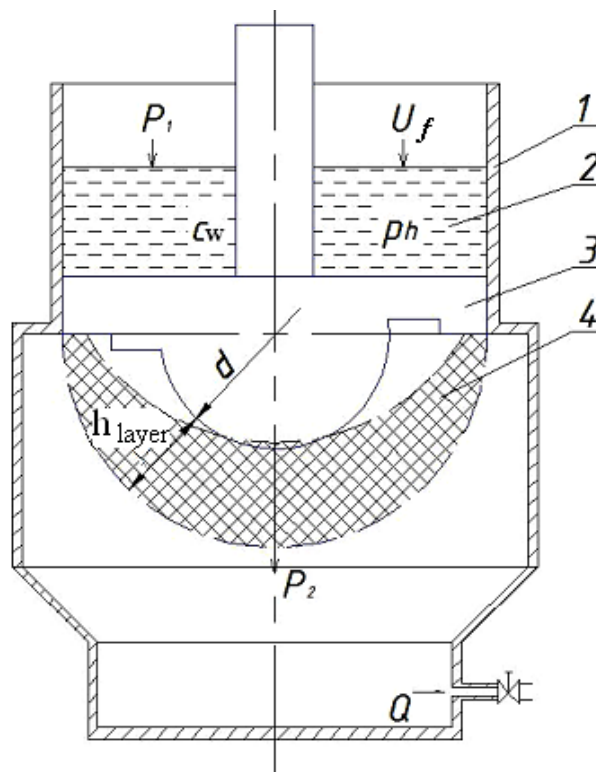


Figure 8. The scheme of cylindrical shells moulding made of the short basalt fibres:
1 – camera; 2 – pulp; 3 – punch; 4 – the initial layer of fibre sediment

To determine the actual density of the “shell” product obtained by the filtration deposition method, measurements of mass and geometric parameters of the product were carried out. Products from four different batches were taken. The geometrical parameters were almost equal, with minor errors, resulting in figure 9: $D_{TPM}=110\pm0.2$ mm; $d=60\pm0.2$ mm; $L_{TPM}=255\pm0.1$ mm; $h_{TPM}=25\pm0.1$ mm. The average weight of the shells was found equal to 0.1327 kg, the material density $\rho_{TPM}=156\pm3.4$ kg/m³, and the fibrous composition porosity was 94 %.



Figure 9. View of the assembled heat insulation basalt shells for rockets and spacecraft

4. Conclusions

The proposed mathematical model for the free filtration deposition of short fibres from liquid pulp describes the dynamics of the heat insulating product formation, and determines rational parameters of the technological process of their manufacture.

To improve product quality and increase the productivity of the deposition process for short basalt fibre filtration, it is necessary to use the filtrate chamber vacuum treatment and sediment layer finish pre-moulding to obtain the final geometric size and shape of the product.

To increase the compressive strength of the highly porous fibrous rings and cylindrical shells, it is necessary to use a mineral binder of 5-7 % by weight, based on oxide Al_2O_3 , that fixes the basalt fibres in the sites of their contact.

The cylindrical shells obtained by the filtration deposition method from the short basalt fibres meet the required quality.

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