STUDY ON THE USE OF AEROGEL ON THE SURFACE OF BASALT FABRIC

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E-mail: pamela_miskiewicz@wp.pl**Abstract:**

The layer of aerogel was applied to the surface of basalt fabric due to the possibility of improving a fabric protecting against the influence of hot environmental factors. The analysis of aerogel surface roughness and thickness of the obtained sample, resistance to contact heat for the contact temperature between 100°C and 250°C, and tests of resistance to the penetration of thermal radiation were carried out. In addition, thermal conductivity, thermal resistance, thermal diffusion, thermal absorption, and surface roughness were determined. The obtained results indicate the unevenness of aerogel application on the surface of basalt fabric. For this reason, work should be carried out on an appropriate technology that will allow them to be applied evenly on the surface of the fabric. The parameters tested and the results obtained are promising in terms of the possibility of using the fabric obtained in protective gloves.

Keywords:

Aerogel, basalt fabric, protective gloves, hot work environment, surface roughness, CIELAB

1. Introduction

Currently, in the textile market, the development of innovative technologies related to the production of technical products is visible, including accessories and specialist clothing. New solutions appearing in the textile industry are to guarantee the user's comfort and functionality of the product under specific environmental conditions.

Aerogels are highly porous materials that consist of 95% air and 4% silicon dioxide. They are characterized primarily by a low density of 2–150 mg/cm³, a low coefficient of thermal conductivity, and a small refractive index [1–4]. In addition, aerogels exhibit good mechanical properties; they are resistant to stretching and compression and are optically transparent. Currently, chemical methods are used for obtaining aerogels [1, 2, 5]. Most of them are made of silica, due to which they are stable up to 1200°C, which corresponds to the melting point of the silica. Properties that show aerogels show that it is an ideal material for the construction of spacecraft. Moreover, aerogels have been used in space suits as an insulation layer. Increasingly, they are used in aviation in the form of thermal insulation in the aircraft. An extremely important feature of aerogels is their very low thermal conductivity compared with standard insulation materials [5]. The low thermal conductivity affects the limitations of heat loss, which is very important for materials with protective properties that guarantee the insulation against hot and cold environments. An important disadvantage in the application of silica aerogels is their fragility as well as time consumption and energy consumption in the production process.

In the field of textiles, the use of aerogels has also been noticed. So far, attempts have been made to exploit the excellent insulating properties of aerogels, and attempts have been made to apply them to materials that protect people against hot or cold environments. The vast majority of research on the introduction of aerogels into textiles was carried out for applications in nonwovens [1, 2, 5–7] and less frequently for woven fabrics [7–9].

Basalt is a volcanic solid rock, mainly of fine-grained, gray, green, or black color, and can be used for the production of basalt fibers [10]. These fibers are produced in the process of melting rock basalt, which occurs at the temperature above 1450°C. The obtained fibers are an alternative to some of the high-efficiency fibers as they show unique physicochemical properties, such as an exceptional resistance to low and high temperatures and resistance to alkali and acids. They show a good resistance to vibrations, dirt, abrasion, corrosion, UV radiation, and a good durability, are fatigue-free, do not require any chemical additives in the production process, are safe for the humans, and are environmentally friendly. Fabrics made from basalt yarns are distinguished by a good resistance to thermal and mechanical factors [11, 12]. Basalt textiles are used for reinforcement of concrete and the production of composites. In addition, they can be used as filtration materials in chemical, oil, and petrochemical industries [13]. The fabrics are also used as acoustic, thermal insulation, and fireproof curtains [14]. The new mineral raw material, which is basalt fibers in the textile industry, is used primarily for the production of personal protective equipment [15–17].

Work in exposure to hot agents is associated with the occurrence of hot microclimate when the air temperature is in the range of 25–60°C, with a relative humidity of 10–80%. Long-term work in a hot environment causes noticeable thermal stress and fatigue, which may increase the risk of workers in the hot environment. Hence, special attention in these working conditions should be devoted to ensuring safety, the selection of personal protective equipment that guarantees protection at the appropriate level and the highest possible level of thermal comfort.

Performing work in a hot microclimate is associated with the exposure of employees to various dangers, that is:

- splashes of molten glass or metals,
- splinters of hot glass, metal, and slag,
- contact with the flame,
- contact with various hot items, and
- strong thermal radiation that can cause skin and eye danger.

To prevent various types of hazards, to which an employee working in a hot microclimate environment is exposed, certain personal protective equipment is used such as equipment for the face and eye protection [17], the protective clothing [15], the safety footwear [18], and the protective gloves [16, 17]. Gloves used in conditions of hot microclimate in a particular workplace are primarily to protect the user's hands against heat and fire.

Research regarding the application of aerogel to the surface of basalt fabric to improve the thermal properties of the fabric and the possibility of using it in protective gloves intended for the use in the hot environment was carried out.

2. Materials

The fabric was selected from the fabrics made of basalt fibers. Basic parameters of the woven fabric were determined and given in Table 1.

Image of basalt fabric was captured using Olympus SZX10 stereo microscope at the total visual magnification of 9.5× and presented in Figure 1a.

The SZX10 microscope is equipped with 1× and 1.5× objective lens for the high magnification and resolution. The microscope covers a magnification range of 0.63× to 6.3× with its 1× objective and 0.95× to 9.5× with its 1.5× objective. It is designed to provide a completely natural view of the sample with a good stereo and color representation.

The process of applying aerogel to the surface of basalt fabric was made at the Central Institute for Labor Protection, National Research Institute (CIOP-PIB) in Lodz. The composition of the multilayer product was as follows: fabric, aerogel, glue, humidifier, and flame retardant mean. Descriptions and contents of individual components could not be given due to patent actions. Image of basalt fabric covered with aerogel was captured using the stereo microscope described above and presented in Figure 1b. The same total visual magnification was applied.

3. Methods

3.1. Determination of resistance to contact heat

Resistance to contact heat was tested according to the standard PN EN ISO 12127-1:2016 [19], using the OTI device for testing the thermal insulation (Figure 2), for contact temperatures of 100°C and 250°C.

Table 1. Structure parameters of basalt fabric

Weave	Thickness (mm)	Mass per unit area (g/m ²)	Apparent density (kg/m ³)	Thread count (threads/cm ²)
Twill	0.55	398	724	72

Figure 1. (a) Basalt fabric before applying aerogel. (b) Basalt fabric after applying aerogel

Figure 2. OTI device owned by the Central Institute for Labor Protection-National Research Institute

The principle of the test relies on subjecting the test sample, which is placed on the calorimeter, to contact with a heating cylinder, heated up to the temperature from 100°C to 500°C. The contact temperature is selected depending on the expected application of gloves at a particular workplace. To carry out the test, three samples should be prepared, with a diameter of 80 mm, and 24 h before the measurement, they should be acclimated in the following conditions: temperature $20 \pm 2^\circ\text{C}$, relative humidity 65%. During the test, the threshold time period t_t is measured, which is the time between the first contact with the heating cylinder and the moment, when the temperature of the calorimeter increases by 10°C compared with the initial value. In the case of gloves, the tests are carried out for samples, and the arithmetic mean is calculated of the three obtained values of the threshold time period t_t . Based on test results, the gloves are classified into the appropriately chosen efficiency level.

The standard PN EN 407:2007 [20] defines the resistance ranges to contact heat and four efficiency levels, to which gloves are classified based on laboratory tests. The efficiency levels are shown in Table 2.

3.2. Determination of resistance to radiant heat

Radiation heat resistance was determined by the test method described in the PN EN ISO 6942:2005 [21] standard (method B), taking into account the guidelines from the PN EN 407:2007 standard [20]. The principle consists in subjecting the sample of the tested material, placed on a suitable holder, to thermal

radiation with a flux density of 20 kW/m^2 at a given time. In the case of protective gloves, the increase of temperature of the calorimeter by 24°C is recorded. It is expressed in the form of heat transfer coefficient t_{24} . The tests are performed for two samples, and as a result of the test for gloves, the arithmetic mean calculated from the two heat transfer times t_{24} (this is the so-called RHTI_{24} coefficient), on the basis of which the gloves are classified to one of the four levels of effectiveness, according to the standard PN EN 407:2007 (Table 3).

The radiation heat resistance test for selected fabric variants was made using a device for measuring the thermal radiation transfer coefficient using a copper calorimeter (Figure 3).

3.3. Determination of thermal insulation properties of improved basalt fabric destined for the protective clothing

The thermal insulation properties of textiles are one of the most important features of flat textile products. They primarily determine the elementary functions of clothing. In addition, thermal insulation properties are an important factor in assessing the comfort of the user in clothing.

The Alambeta testing device was used to determine the thermal insulation properties of the basic and modified basalt fabrics [22]. It was constructed and manufactured by the Czech company Sensora, and it is an automatic device used primarily to obtain dynamic and statistical evaluations of the thermophysical properties of textile materials [23]. Performing the test relies on measuring the amount of heat penetrating the tested sample – fabric, which is placed between the upper plate with a temperature of 32°C (it should more or less correspond to the human body temperature) and the lower plate reaching the

Figure 3. Stand for testing the resistance to the penetration of thermal radiation

Table 2. Efficiency levels for the resistance to contact heat according to the standard PN-EN 407:2007 [20]

Efficiency level	Contact temperature (T_c , $^\circ\text{C}$)	Threshold time period (t_t , s)
1	100	≥ 15
2	250	≥ 15
3	350	≥ 15
4	500	≥ 15

Table 3. Efficiency levels for the resistance to radiation heat according to the standard PN EN 407:2007 [20]

Efficiency level	Heat transfer coefficient t_{24} (RHTI_{24}), s
1	≥ 7
2	≥ 20
3	≥ 50
4	≥ 95

ambient temperature. During the measurement, metal plates adhere to the tested sample with a pressure of approximately 200 Pa. The device is primarily used in scientific research, and its important advantage is the short time of measurement. As the only instrument in the world, the device allows conducting a simultaneous examination of seven parameters of the selected material, which define its thermal insulation properties.

Using the Alambeta device, it is possible to determine the following parameters, which describes the thermal insulation of textile materials:

- *Thermal conductivity λ* – defined as the ability of the tested material to conduct heat and expressed in $\text{Wm}^{-1}\text{K}^{-1}$. About textiles, this parameter depends primarily on the fiber arrangement.
- *Thermal diffusion A* – called the temperature compensation coefficient, expressed in m^2s^{-1} . This parameter allows determining how quickly the tested material reacts to temperature changes.
- *Thermal absorption b* – defined as the heat absorption coefficient of tested material, expressed in $\text{Wm}^2\text{s}^{1/2}\text{K}^{-1}$. It determines the surface property resulting in the sensation during the contact between the human skin and the material.
- *Thermal resistance r* – defined as a barrier against the heat penetrating through the tested material, considering the difference in temperatures and expressed in W^{-1}Km^2 .
- *Sample thickness h* – the device enables the automatic measurement of the thickness of the tested sample, expressed in mm.

3.4. Determination of surface roughness of aerogel-coated basalt fabric

Sample of basalt fabric covered by an aerogel was taken into account. Grayscale map of the sample surface was obtained. Line profiles depicting the dependence of the grayscale values of the pixels on the distance were determined to roughness profile measurements for the sample. The gray level range was 0–1023. The distance was defined as a distance from the beginning of the line. Three horizontal (H1, H2, and H3) and three vertical (V1, V2, and V3) roughness profiles were determined on a square sample with sides of length 1 cm. The square covers part of the aerogel-coated basalt fabric (Figure 4). Stream motion software was used to find line profiles. The Stream software enables step-by-step workflows to acquire sharp, crisp images that are ready for quantitative measurements and professional reporting based on the latest standards.

Measurements of surface unevenness of aerogel applied to the basalt fabric were carried out. Surface texture R parameters (profile parameters) were chosen based on the standard ISO 4287:1997 [24]:

Figure 4. Line profiles arrangement on sample surface

- *Arithmetic mean deviation R_a of the assessed profile* – indicates the average of the absolute value along the sampling length. R_a is used as a global evaluation of the roughness amplitude on a profile. R_a is meaningful for the random surface roughness. The parameter does not detect irregularities or shape of the profile.
- *Root mean square deviation R_q of the assessed profile* – corresponds to the standard deviation of the height distribution, defined on the sampling length. The parameter provides the same information as R_a .
- *Maximum profile peak height R_p* – indicates the point along the sampling length, at which the curve is highest.
- *Maximum profile valley depth R_v* – indicates the point along the sampling length, at which the profile curve is lowest.
- *Total height of the profile R_t* – the vertical distance between the maximum profile peak height and the maximum profile valley depth along the sampling length, wherein

$$R_t = R_p + R_v \quad (1)$$

- *Maximum height of the profile R_z* – the mean value of the individual profile peak heights obtained between the maximum profile peak height and the maximum profile valley depth along the sampling length. Usually, sampling length is divided into $n = 5$ evaluation lengths; hence

$$R_z = \frac{1}{5} \sum_{i=1}^{n=5} (R_{pi} + R_{vi}) \quad (2)$$

This parameter is frequently used to check, whether the profile has protruding peaks that might affect static or sliding contact function.

3.5. Assessment of the quality of layer covering basalt fabric

The following assumptions have been made: The sample in square shape lies on a plane XY in such a way that its center corresponds to the coordinates (0,0). The area of a square

sample with sides of length 3 cm was divided into nine squares (A, B, C, D, E, F, G, H, and K) with sides of length 1 cm (Figure 5).

Each square covers part of the aerogel-coated basalt fabric. Colors of two neighboring squares were compared based on the instrumental color measurement. Instrumental color measurement can be used as the method provides sufficient accuracy and repeatability for the objective color assessment of uniformity of layer covering basalt fabric. DigiEye is a noncontact digital color imaging system. It is useful especially in the case of measurements of fabric textures [25, 26]. CIE 1976 $L^*a^*b^*$ (CIELAB) color space was defined by International Commission Illumination and used to express color as three numerical values [27]:

- *Lightness L^** – the percentage of chromatic colors.
- *Axis a^** – the green-red component with green in the negative direction and red in the positive direction; values are often within the range of –100 to 100.
- *Axis b^** – the blue-yellow component with blue in the negative direction and yellow in the positive direction; values are often within the range of –100 to 100.

Parameters L^* , a^* , and b^* combine Cartesian coordinates and form 3D color space in the cylindrical coordinate system. The system provides the prediction parameters such as:

- *Chroma C^** – the percentage of hue in color.
- *Hue angle h* – the achromatic point in the a^*b^* plane.

CIE standard is to define procedures for calculating the coordinates of the CIELAB color space and the Euclidean color difference values based on these coordinates.

Difference ΔE between two colors B1 (L_1^* , C_1^* , h_1) and B2 (L_2^* , C_2^* , h_2) can be expressed by the following dependency:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta C^*)^2 + (\Delta H)^2} \quad (3)$$

wherein:

$$\Delta C^* = C_1 - C_2 \quad (4)$$

$$\Delta L^* = L_1 - L_2 \quad (5)$$

$$\Delta H = 2\sqrt{C_1 - C_2} \sin(\Delta h) \quad (6)$$

$$\Delta h = h_1 - h_2 \quad (7)$$

Differences ΔE determined for selected pairs of squares were used to assess the quality of layer covering basalt fabric. CIE illuminant D65 was used.

4. Results and discussion

4.1. Results of contact heat resistance tests

The results of contact heat resistance tests and the thermal radiation transmission were analyzed in terms of requirements for the use in protective gloves. The results of contact heat resistance measurements for the contact temperature of 100°C and 250°C for the fabric made of basalt fiber and the fabric of aerogel-coated basalt fiber yarn are listed in Table 4.

In Table 4, three statistical parameters are given: $\bar{x}$ – the arithmetic mean, SD – the standard deviation and SD^2 – the coefficient of variation.

Figure 6 shows the results of resistance to contact heat at the contact temperature 100°C and 250°C for all tested variants.

The tests of resistance to contact heat at the contact temperature 100°C and 250°C show a significant difference between the values of contact heat for basalt fabrics and the basalt fabric with aerogel. The value of the tested parameter for the fabric made of basalt fibers for the contact temperature of 100°C is on the level of 10.6 s, while for the contact temperature 250°C, it is in the range of 4.7 s. The highest resistance to contact heat for both contact temperatures was obtained by basalt with the aerogel. For the contact temperature of 100°C, it achieved 1 level of protection efficiency, while at the contact temperature of 250°C, it reached a resistance of 9 s to contact heat. The results shown in Table 4 are divergent among themselves due to the uneven coverage of the surface of basalt fabric by aerogel.

4.2. Results of radiation heat resistance tests

Figure 7 presents the results of thermal radiation resistance measurements for the fabric made of basalt fiber yarns and the aerogel-coated basalt fabric.

Based on the test results presented in Figure 7, it is visible that all tested fabrics have the first level of efficiency of protection

Figure 5. Squares arrangement on sample surface

Table 4. Measurement results of contact heat resistance for the contact temperature of 100°C and 250°C

Statistical parameters	Resistance to contact heat 100°C, t_p , s		Resistance to contact heat 250°C, t_p , s	
	Basalt	Basalt+Aerogel	Basalt	Basalt+Aerogel
	10.90	24.73	4.50	9.41
	10.60	27.03	4.10	9.22
	11.00	30.03	4.30	7.90
$\bar{x}$	10.833	27.263	4.300	8.843
SD	0.208	2.658	0.200	0.822
SD ²	1.9%	9.8%	4.7%	9.3%
Efficiency level	0	1	0	0

Figure 6. Comparison of contact heat resistance for all tested materials for contact temperatures of 100°C and 250°C **Figure 7.** Comparison of radiation heat resistance for all tested materials

against radiation with the 20 kW/m² flux density. The highest resistance of the tested parameter was achieved for the basalt fabric with aerogel.

4.3. Results of thermal insulation properties tests

Measurements of thermal insulation properties were done on the fabric made of basalt fibers and fabrics made of basalt fibers with aerogel. The test was carried out under normal climate conditions; 10 measurements were taken for each variant. The graphical presentation of the most important parameters of thermal insulation of textile materials is presented below: the thermal conductivity (Figure 8), thermal resistance (Figure 9), sample thickness (Figure 10), thermal absorption (Figure 11), and thermal diffusivity (Figure 12).

Thermal conductivity defines the material ability to conduct heat. This parameter depends on the material porosity and structure. More heat under the same conditions will flow through the material with a higher coefficient of thermal conductivity. As a result of the aerogel application, an increase in thermal conductivity was achieved on the surface of the basalt fabric.

The fabric made of basalt fibers with the aerogel had a higher thermal resistance value equals $28.2 \times 10^{-3} \text{ W}^{-1}\text{km}^2$, so it is the largest barrier to heat that penetrates through the material

Figure 8. Influence of type of material on the effective thermal conductivity

during the test. In addition, the high thermal resistance value is associated with an increase in the heat protection of the tested material. The thermal resistance value increases with the thickness of the textile material, which is consistent, because the basalt sample with aerogel is much thicker than the remaining sample of basalt fabric due to the thickness of aerogel layer (Figure 10).

Figure 9. Influence of type of material on the thermal resistance

Figure 11. Influence of type of material on thermal diffusion

Figure 10. Influence of type of material on the fabric thickness

Figure 12. Influence of type of material on the thermal absorption

The difference in thickness between the two samples is about 1 mm. The thickness of the aerogel layer may influence the results of the other parameters tested.

The fabric samples tested have a differentiated thermal diffusion value. The higher value of the determined parameter has reached the fabric made of basalt fibers with aerogel and then the fabric without the aerogel, which means that it easily dissipates heat and responds quickly to the temperature change.

Thermal absorption makes it possible to evaluate textile materials about the assessment of the feel of their grip/cold feel. When the material is characterized by the high heat absorption, it has a feeling of coolness, when touched. The obtained absorptivity results for the tested fabrics slightly differ from each other. The fabric made of basalt fibers has reached the higher value of thermal absorption than this with the aerogel. It means that at the time of contact with the skin, the fabric will be felt as colder, while the fabric made of basalt fibers with the aerogel will feel warmer in touch due to the lower value of this parameter.

4.4. Results of sample surface roughness analysis

To evaluate the surface roughness of the sample covered by the aerogel, three vertical roughness profiles were determined using Stream motion software. Horizontal roughness profiles are presented in Figures 13–15.

Based on the received roughness profiles, chosen R parameters were determined. Equations (1) and (2) were used to calculate the total height R_t and the maximum height R_z of the profiles and given in Table 5.

Values of coefficients variation of all R parameters indicate the unevenness of the aerogel layer applied to the surface of basalt fabric. The values are in the range of 10–27%. The roughness parameters affect the functional properties of the material.

Group of parameters R_a , R_q , and R_t are used to assess the thermal contact of the surface. The arithmetic mean R_a and the root mean R_q do not show large scattering at 10–11% level. The total height of the profile $R_t = 738.3$ is significant in comparison with the upper limit of the gray scale equals 1023. The rough surface of the tested sample is more difficult to keep clean. Roughness promotes the adhesion of the material, in particular, the adhesion of metal splashes of molten metal to the material. Mechanical tightness of the material and its surface reflectivity assessment can be carried out using parameters R_t and R_z . The average value $R_z = 573.0$ calculated for six lines profiles (H1, H2, H3, V1, V1, and V3) in comparison with the upper limit of the gray scale means that the profiles have protruding peaks that might affect static or sliding contact function. It is important from the point of view radiation heat resistance properties of the material. Moreover, a thin coating of aerogel may not fully meet the protective performance connected with contact heat resistance of surface-modified basalt fabric.

Figure 13. Horizontal roughness profile 1

Figure 16. Vertical roughness profile 1

Figure 14. Horizontal roughness profile 2

Figure 17. Vertical roughness profile 2

Figure 15. Horizontal roughness profile 3

Figure 18. Vertical roughness profile 3

Table 5. Roughness parameters

Line profile	R_a	R_q	R_p	R_v	R_t	R_z
H1	372	397	276	579	855	654
H2	384	419	265	639	904	783
H3	413	427	236	322	558	447
V1	483	500	254	540	794	627
V2	385	410	308	399	707	457
V3	370	383	243	368	611	470
Statistical parameters*	R_a	R_q	R_p	R_v	R_t	R_z
$\bar{x}$	401.2	422.7	263.7	474.5	738.2	573.0
SD	42.9	41.0	26.1	128.5	137.0	136.8
SD ²	11%	10%	10%	27%	19%	24%

*Designation of statistical parameters as in Table 4.

4.5. Result of assessment of the quality of layer covering the basalt fabric

Colors of two neighboring squares covering the chosen part of the aerogel-coated basalt fabric were compared. Differences ΔE were determined from formula (3) for selected pairs of squares. To each ΔE , the value the coordinates (x, y) was assigned and presented in Table 6.

Values of ΔE are in the range of [0.68, 4.29]. Values below 2 mean that the average observer is not able to detect the differences in colors of the two selected areas. Values

above 2 indicate the mean that there are visible differences. Measurement results are presented in Figure 19 in the form of surface 3D plots. The distance-weighted least squares method was used to fit a curve to the data.

The obtained measurement results of differences of colors of selected areas of basalt fabric indicate that the aerogel covers the fabric unevenly.

Table 6. Results of instrumental color measurements

No.	1	2	3	4	5	6	7	8	9	10	11	12
Pair of squares	AB	BC	DE	EF	GH	HK	AD	DG	BE	EH	CF	FK
x	-1.0	-1.0	0.0	0.0	1.0	1.0	-0.5	0.5	-0.5	0.5	-0.5	0.5
y	0.5	-0.5	0.5	-0.5	0.5	-0.5	1.0	1.0	0.0	0.0	-1.0	-1.0
ΔE	1.85	0.71	4.29	1.05	1.91	0.68	0.76	0.94	3.48	2.28	2.51	2.24

Figure 19. Color differences observed on aerogel-coated basalt fabric

5. Conclusions

The obtained results confirm the legitimacy of applying aerogels to the fabric structures. As a result of the use of aerogel on the surface of the basalt fabric, the parameters studied, that is, the resistance to contact heat, resistance to radiation heat, and selected thermal parameters are improved. The obtained results confirm the operation of the aerogel layer. The basalt fabric modified by the aerogel was characterized by varying thickness and roughness, which is associated with work on the development of aerogel application technology into the fabric surface. Analysis of colors differences of selected pairs of squares covering chosen part of the aerogel-coated basalt fabric enabled an assessment of the quality of aerogel layer covering the fabric. Received results indicated that the aerogel covers the fabric unevenly. For the use of modified fabric in protective gloves, it would be desirable to apply various aerogel values of thickness to the surface of basalt fabric and extend tests of protective and mechanical properties. The layer should be uniform to fully meet the protective performance. Although the aerogel application on the fabric is very demanding, the results will be continued by the authors.

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