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Examination of Selected Thermal Properties of Basalt Composites

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Abstract
A composite based on basalt fabric was produced because of its good thermal and mechanical properties. As a result of the reactive magnetron sputtering technique, a layer of aluminum and zirconium (IV) oxide 200 nm thick was deposited on Mylar film, which was adhered to the surface of the basalt fabric using a special adhesive glue and silicone. The variants of composites prepared were subjected to contact resistance tests at a contact temperature of 100 °C and 250 °C, as well as to resistance to thermal radiation tests. The tests carried out on the composites obtained showed an improvement of tested parameters.

Key words: *magnetron sputtering, basalt fibers, hot working environment, physical vapour deposition, contact heat, radiant heat.*

Introduction

Currently, the textile market is noteable in the development of innovative technologies related to the production of technical products, including accessories and specialist clothing. New solutions appearing in the textile industry are primarily intended to provide the user with comfort and functionality of the product under specific environmental conditions.

Basalt fibres, which have recently been used in the production of specialist clothes protecting against the effects of high temperatures, are characterised primarily by good mechanical and thermal properties. Basalt is a volcanic solid rock, mainly of a fine-grained structure and gray, green or black colour [1]. Fibres therefrom are produced by the process of melting basalt rock, which occurs at a temperature above 1450 °C. These fibres belong to the mineral chemical group, and products made of them, including accessories and specialist clothing, are used at temperatures up to 700 °C [2].

Basalt fibres are an alternative to some high-performance fibres due to their good resistance to high temperatures and various chemicals. Basalt textiles are widely used in many fields. They are used in sound insulation, in construction, mainly for the reinforcement of concrete, and in the chemical and petroleum industry as filter materials. These fibres are also used as enhancers for polymer composites and polymer fillers [3]. In addition, they are utilised to reinforce road surfaces, in shipbuilding, as well as in the petrochemical industry, mainly as filter materials, and serve as fireproof curtains.

In addition, basalt fabrics are characterised by a lower manufacturing cost, which makes that the final product obtained from basalt fabrics can be at a lower cost, as compared to other fabrics, such as aramid or glass [4].

Textile products made of basalt fibres, due to their excellent thermal and mechanical properties, are an alternative to the materials and systems of materials intended for the construction of protective gloves currently used.

Work with exposure to hot agents is associated with the occurrence of a hot microclimate, where the air temperature is in the range of 25-60 °C, with a relative humidity of 10% to 80%. Long-term work in a hot environment causes noticeable thermal stress and fatigue, which may increase the risk to workers in the hot environment. Hence, special attention in these working conditions should be devoted to ensuring the safety and 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, i.e.:
- splashes of molten glass or metals,
- splinters of hot glass or metal,
- contact with flames,
- contact with various hot items,
- strong thermal radiation, which can be a danger to the skin and eyes.

In order to prevent the various types of hazards to which an employee working in a hot microclimate environment is exposed, certain personal protective equipment is used: equipment for the face and eye protection, protective clothing, protective gloves and safety footwear. Gloves used in conditions of a hot microclimate in a particular workplace are primarily to protect the user's hands against heat and fire [5, 6].

The BAGLO project, conducted by the Central Institute for Labor Protection – National Research Institute, in cooperation with Lodz University of Technology, concerned the development of a textile package for protective gloves against hot factors, while also providing protection against mechanical factors. In order to develop a new type of protective glove fabric, basalt fibres were used as well as their modification. The modification consisted in covering the basalt fabric with aluminum foil. Uncoated basalt fabrics and basalt fabrics to which aluminum foil was bound by glue were used. None of the selected fabrics intended for protective gloves achieved contact heat resistance for a 250 °C contact temperature. The highest level of effectiveness of protection against radiant heat was obtained for aluminised basalt fabrics. An additional disadvantage of the aluminized basalt fabric produced was the abrasion and cracking of the layer applied [7, 8].

Due to the above-described negative aspects of the research conducted, this work concerned the modification of Mylar film by depositing on its surface aluminum and zirconium (IV) oxide with a thickness of 200 nm by means of magnetron sputtering, and then the basalt composite preparation.

Surface engineering, as a part of material engineering, is a scientific discipline that covers technical and scientific issues that are related to the formation of layers on

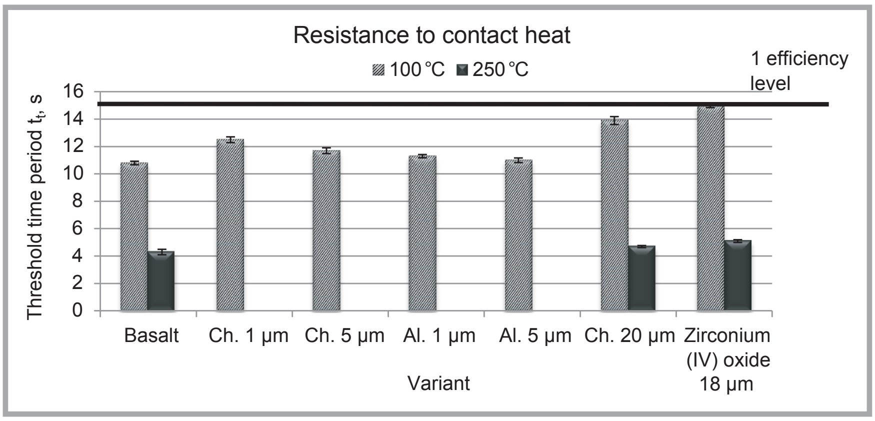

Figure 1. *Values of resistance to contact heat for contact temperatures of 100 °C and 250 °C of samples tested – first stage of testing.*

the surface of selected material as well as under its surface. The surface layer has different properties from the deposited material [9].

The processes of the physical deposition of gas phase coatings are permanently associated with the development of the vacuum technique. They use, in their basic versions, two basic methods for changing the physical state of the coated material, namely evaporation or sublimation (usually thermal) and spraying, occurring under the influence of, other than thermal ones, physical stimuli. Performance characteristics of the coatings applied depend on individual physical properties and a proper adhesion to the substrate. Achieving good coating adhesion to the substrate in the standard methods of the physical vapour deposition process requires very precise surface cleaning, since the substrate temperature during the deposition of coating material is not high. By the term coating, we mean applying a layer of a specific substrate that has completely different properties, structure, and chemical composition from the coated material [10]. In addition, the magnetron sputtering method enables the production of coatings improving the tribological, decorative, corrosive and other properties of products that are used in various conditions of use. An important advantage of using magnetron sputtering is the possibility of high-performance, even the production of coatings on surfaces of various sizes and shapes.

Heat transfer through textile substrates is a very complicated phenomenon. In 2015, research was carried out on the use of magnetron sputtering on the surface of protective fabrics in order to change their thermal properties. An aluminum (Al) coating was deposited on the surface of aramid fabric, as well as a two-layer coating: aluminum (Al) with silicon dioxide (SiO_2), and a three-layer coating: silicon dioxide (SiO_2)/aluminum (Al)/silicon dioxide (SiO_2). The metal coating (aluminum) on the surface of the fabric was deposited by the DC (direct current) magnetron sputtering technique, while the non-metallic coating (silicon dioxide) by RF (radio frequency) magnetron sputtering technique. It was observed that all three types of coated structures on the surface of aramid fabric form continuous and dense layers. It was shown that the resistance to heat radiation was improved for three types of coatings applied on the aramid fabric [11].

Further research concerned the thermal and physical properties of nylon fabric subjected to metal sputtering, i.e., aluminum, copper and nickel. The basic material in the research was the nylon fabric. Mylar foil was also used, one side of which was conductive, as a result of aluminum spraying. The thickness of the deposited metal coatings was about 600 nm. Examination by X-ray spectrometry of an energy dispersion showed an over 80% content of individual metals on the surfaces of the samples tested. Results of the shear test were comparable for uncoated and coated samples. The conductive fabric showed the highest stiffness, because it was more tightly woven [12].

Heat transfer in sputtered aluminum textiles was also investigated. The metal was sprayed on four textile substrates: nylon, polyester, a cotton blend with 50/50 polyester and polyurethane with a shape memory, which was produced by the authors and named SMPU (shape memory polyurethane). The thickness of the coating applied increased linearly with the spraying time. The spraying time for aluminum on selected substrates was 1, 3, 5, 10 and 25 minutes, which allowed coatings with a thickness of 67.5, 145.8, 254.0, 378.8 and 884.1 nm to be obtained. Aluminum was only sprayed on one side. It was confirmed that basic fabrics and those modified with aluminum coating showed different values of thickness, thermal conductivity and heat emissivity. To identify heat transfer properties, aluminum was applied to selected samples for 10 minutes. The heat transfer coefficient of the aluminum coating increased with its thickness. The cotton/polyester blend (50/50) showed the lowest heat transfer coefficient [13].

Genesis of the study

In the first stage of research presented in [1-2], a one-sided coating was created directly on the surface ofbasalt fabric. Chrome, aluminum and zirconia were deposited on fabric at various values of thickness: 1.5 and 20 μm thick chrome, 1 and 5 μm aluminum, and 18 μm zirconia. The modified basalt fabric samples were tested for contact heat resistance (***Figure 2***) and radiant heat resistance (***Figure 3***).

For ***Figure 2***, the following symbols were used:

- Ch. 1 μm – basalt fabric modified with a chrome layer of 1 μm thickness,
- Ch. 5 μm – basalt fabric modified with a chrome layer of 5 μm thickness,
- Al. 1 μm – basalt fabric modified with a aluminum layer of 1 μm thickness,
- Al. 5 μm – basalt fabric modified with a aluminum layer of 5 μm thickness,
- Ch. 20 μm – basalt fabric modified with a chrome layer of 20 μm thickness,
- Zirconium (IV) oxide 18 μm – basalt fabric modified with a zirconia (IV) layer of 18 μm thickness.

As a result of the direct coating of basalt fabric, the first level of protection efficiency against contact heat for a contact temperature of 100 °C was obtained only for the 18 μm zirconia (IV) coating. Tests of contact heat resistance at 250 °C were performed for samples of basalt fabric, and basalt fabric coated with 20 μm chromium and 18 μm zirconia (IV), because the remaining coatings did not achieve satisfactory results in the test for a contact temperature of 100 °C.

Only selected samples were tested for resistance to thermal radiation. The best result was obtained for a 20 µm thick basalt fabric sample coated with chromium. It has been observed that covering by metals causes low resistance to contact heat, while the fabrics tested herein show better resistance to thermal radiation; although the opposite situation is noted for zirconia (IV) oxide coating.

Due to the fact that results obtained by depositing the coating directly on the surface of basalt fabric were not satisfactory, basalt composites as described in the Chapter 4 and 5 were prepared [4].

The article presents results for the resistance to contact heat and radiant heat of basalt composite fabrics. The authors strived to achieve the highest possible resistance to contact heat while not neglecting other thermal properties, i.e., radiant heat resistance.

The results of the resistance to contact heat shown above for a contact temperature of 100 and 250 °C and the resistance to heat radiation obtained for basalt fabric directly coated with metal and ceramic were not satisfactory for the authors. Therefore, a basalt composite was produced to improve the thermal properties selected.

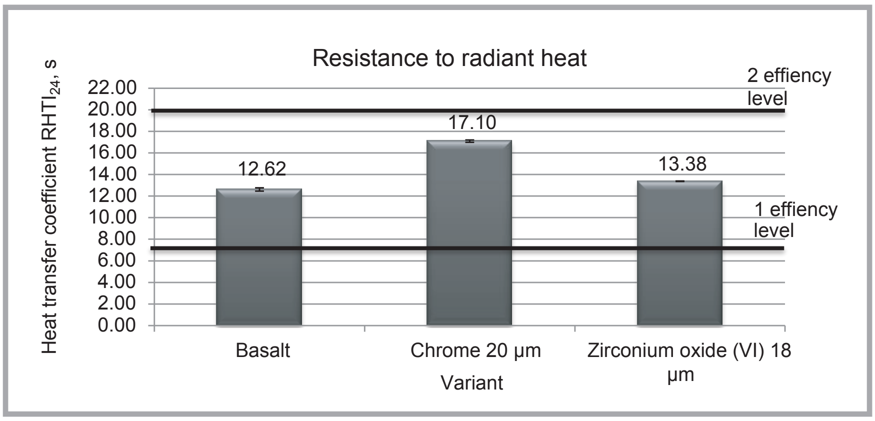

Figure 2. *Results of thermal radiant resistance of samples tested – first stage of testing.*

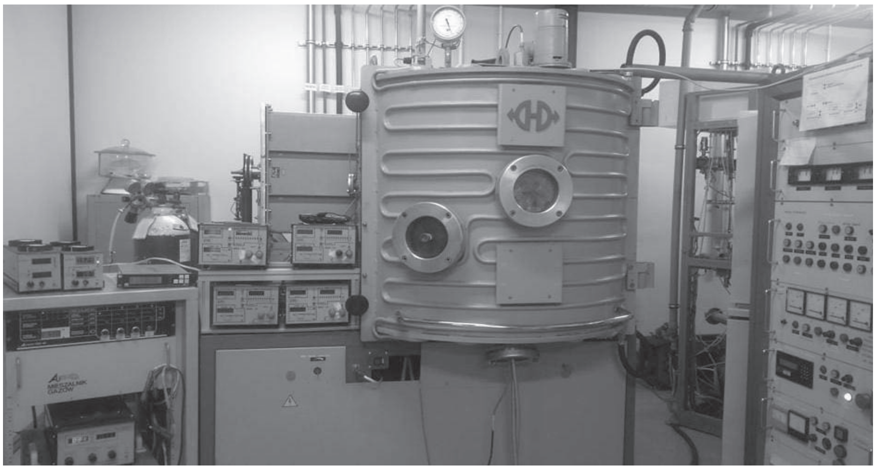

Figure 3. *Stand for depositing coatings using the magnetron method.*

Research methodology

Coating

The main factor in the application of new material is primarily the ability to create a bond between the coating and substrate. The quality of the coating depends on the preparation of the substrate surface before the coating process. By the term coating we mean applying a layer on a specific substrate that has completely different properties, structure, and chemical composition from the coated material.

In the Physical Vapour Deposition (PVD) process, we distinguish the following stages:

- transfer of the material to a steam state,
- transport of the material in the steam state to the object surface,
- material condensation on the object and coating growth [14, 15].

The technique selected for the deposition of individual chemical compounds on the surface of Mylar film was reactive magnetron sputtering, which involved spraying the material, being the substrate of the coating produced by gas ions obtained in the area between the plasma and the batch. The atomised ions then passed through the plasma, undergoing ionisation or reactions containing ions and reactive gas atoms during this time, leading to the deposition of the coating on the material. Magnetron sputtering of the coatings was carried out on a B-90 device (***Figure 3***).

Coatings deposited by reactive magnetron sputtering should cause:

- an improvement of decorative, tribological and anti-corrosive properties of the product operating in
- various conditions,
- the same (not worse) mechanical properties of the particular substrate,
- a very strong connection, so that the adhesion force compensates the stresses that appear in the coating,
- the occurrence of compressive stresses in the coating [9, 14].

Determination of resistance to contact heat

Resistance to contact heat was tested according to the standard PN-EN ISO 12127-1: 2016 [16], using an OTI device for testing the thermal insulation at contact temperatures of 100 °C and 250 °C. The principle of the test relies on subjecting the test sample, which is placed on a calorimeter, to contact with a heated cylinder, heated up to a temperature in the range of 100 to 500 °C. The contact temperature is selected depending on the expected application of gloves at a particular workplace.

In order to carry out the test, three samples should be prepared, with a diameter of 80 mm, and 24 hours before the measurement they should be conditioned in the following conditions: temperature 20 ± 2 °C & relative humidity 65 ± 5%. During the test, the threshold time period t_t is measured, which is the time between the first contact with the heated cylinder and the moment the temperature of the calorimeter increases by 10 °C compared

Table 1. Variants of composites prepared for testing.

No.	Variant of composite	Composite photo	Symbol
1	Fabric made of basalt fibers of twill wear 2/2		B
2	Fabric made of basalt fibers + glue		BK
3	Fabric made of basalt fibers + silicon		BS
4	Fabric made of basalt fibers + glue + foil with aluminum		BKAl
5	Fabric made of basalt fibers + silicon + foil with aluminum		BSAl
6	Fabric made of basalt fibers + glue + foil with zirconium(IV) oxide		$BKZrO_2$
7	Fabric made of basalt fibers + silicon + foil with zirconium(IV) oxide		$BSZrO_2$

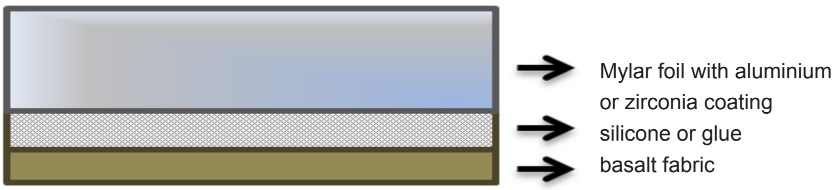

Figure 4. Scheme of basalt composite produced.

to the initial value. In the case of gloves, tests were carried out for three samples, and the arithmetic mean was calculated from the three values of the threshold time period t_t obtained. On the basis of the test results, the gloves are classified to the appropriate efficiency level.

Determination of resistance to radiant heat

The radiant heat resistance was determined in accordance with the test method described in the PN-EN ISO 6942: 2005 [17] standard (method B), taking into account the guidelines from the PN-EN 407: 2007 standard [18]. The principle consists of subjecting the sample of material tested, placed in a suitable holder, to thermal radiation with a flux density of 20 kW/m^2 for a given time period. In the case of protective gloves, an increase in the calorimeter temperature of 24 °C was recorded, which is expressed in the form of the heat transfer coefficient t_{24}. Tests were performed for two samples, and from the results of the test for gloves, the arithmetic mean was calculated from the two heat transfer time values t_{24} (the so-called $RHTI_{24}$ coefficient), on the basis of which the gloves were classified to one of the four levels of efficiency, according to the standard PN-EN 407: 2007. A radiant heat resistance test for selected fabric variants was made using a device for measuring the thermal radiant transfer coefficient using a copper calorimeter.

Materials

Aluminum and zirconium (IV) oxide layers were put on Mylar foil – the sprayed coatings reached a thickness of 200 nanometers.

Four variants of composites were made for the study. Each composite consisted of fabric made of basalt fibre yarn and Mylar foil. Depending on the variant, an adhesive or silicon was used, and Mylar foil was coated with aluminum or zirconium(IV) oxide (***Figure 4***).

Characteristics of individual components of the composite:

- **Basalt fabric:** mass per unit area 398 g/m^2, thickness 0.55 mm, twill weave.
- **Mylar film:** a flexible, strong and durable film for industrial applications. It is a thin translucent film with a thickness of 100 μm and tensile strength of 190 MPa. It exhibits resistance to moisture and most chemicals. Moreover, it is resistant to temperatures from -70 to 150 °C. Due to the fact that the film does not contain plasticisers, it is not brittle. The film can be recycled.
- **Technicqll adhesive**: a polyurethane, colorless glue with high strength parameters for permanent bonding and repair of products made of soft PVC and other synthetic materials, also of fabric undercoat fabric. Resistance to water and temperature in the range from -30 to 80 °C.
- **Silicone technicqll**: a high quality single component silicone adhesive. It creates a layer that remains flexible

over the entire service life at -60 to +300 °C, temporarily up to + 360 °C. Perfectly combines smooth surfaces like, glass, ceramics, plastics, marble and mineral products.

The composites created consisted of the following layers (***Figure 4***).

Test results

Results of contact heat resistance tests

Standard PN-EN 407: 2007 [18] defines the range of resistance to contact heat. We distinguish four levels of efficiency to which the samples of materials subjected to testing are classified, shown in ***Table 2***.

Tables 3 and ***4*** present the results of the resistance to contact heat at a contact temperature of 100 °C and 250 °C, respectively.

Figure 5 shows a comparison of contact heat resistance results for contact temperatures of 100 °C and 250 °C for all samples tested.

Subsequent layers of the composite affect its thickness. Basalt fabric has a thickness of 0.49 mm and basalt fabric covered with glue – 0.57 mm, while for basalt fabric covered with silicon it is 1.17 mm. The adhesive penetrated the basalt fabric structure, while the silicone did not. In the case of composites, the basalt fabric with glue and modified foil of both aluminum and zirconia reached a thickness of 1.29-1.34 mm, whereas for the composite with silicone it was 1.20-1.80 mm.

Analysis of contact heat resistance test results for a contact temperature of 100 °C showed that four composite variants achieved the first degree of protection efficiency, namely: the silicon coated fibre basalt fabric, basalt fabric with silicone and aluminum coated foil, basalt fabric with glue and foil with zirconium (IV) oxide applied, and basalt fabric with silicon and foil with the zirconium (IV) oxide applied.

The resistance to contact heat of the composite variant consisting of basalt fabric, glue and foil coated with zirconia oxide at a contact temperature of 100, achieved the result of 15.1 seconds, and belongs to the 95% confidence interval of the average value. The graph shows an increase in resistance to contact heat for a contact temperature of 100 °C for all variants of samples tested in relation to the reference sample – fabric made of basalt fibre yarn. The highest efficiency of protection against contact heat at a contact temperature of 100 °C was shown by the composite of basalt fabric with silicone and foil with zirconium (IV) oxide applied, for which the threshold time of the sample was 22.2 seconds.

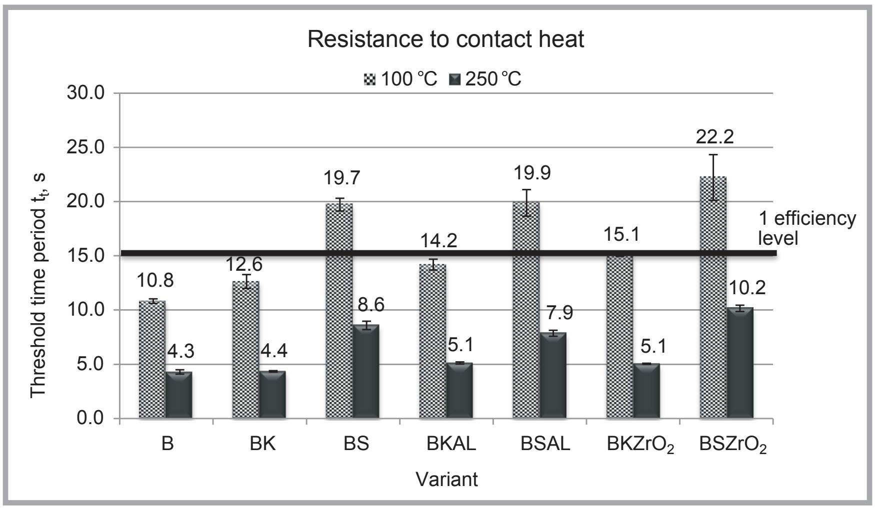

Figure 5. *Values of the resistance to contact heat for contact temperatures of 100 °C and 250 °C of samples tested.*

Table 2. *Efficiency levels for the resistance to contact heat according to Standard PN-EN 407:2007 [18].*

Efficiency levels	Contact tempmerature T_C, °C	Threshold time period t_t, s
1	100	≥ 15
2	250	≥ 15
3	350	≥ 15
4	500	≥ 15

Table 3. *Measurement results of the contact heat resistance for a contact temperature of 100 °C.*

Symbol	B	BK	BS	BKAL	BSAL	$BKZrO_2$	$BSZrO_2$
	Resistance to contact heat 100 °C, t_t, s						
	10.90	13.38	19.30	14.20	20.82	15.00	24.00
	10.60	12.27	19.50	13.70	18.50	15.20	22.80
	11.00	12.28	20.40	14.70	20.35	15.03	19.90
$\bar{x}$	10.833	12.643	19.890	14.200	19.890	15.077	22.233
SD	0.208	0.638	0.586	0.500	1.226	0.108	2.108
CV, %	1.92	5.05	2.97	3.52	6.17	0.72	9.48
Efficiency level	**0**	**0**	**1**	**0**	**1**	**1**	**1**

Where: $\bar{x}$ – arithmetic average, SD – standard deviation, CV, % – variation coefficient.

Table 4. *Measurement results of the contact heat resistance for a contact temperature of 250 °C .*

Symbol	B	BK	BS	BKAL	BSAL	$BKZrO_2$	$BSZrO_2$
	Resistance to contact heat 250 °C, t_t, s						
	4.50	4.28	8.55	5.15	8.05	5.10	10.00
	4.10	4.40	8.23	5.02	7.55	5.00	10.50
	4.30	4.38	9.00	5.21	8.00	5.06	10.00
$\bar{x}$	4.300	4.353	8.593	5.127	7.867	5.053	10.167
SD	0.200	0.064	0.387	0.097	0.275	0.050	0.289
CV, %	4.65	1.48	4.50	1.89	3.50	1.00	2.84
Efficiency level	**0**	**0**	**0**	**0**	**0**	**0**	**0**

Where: $\bar{x}$ – arithmetic average, SD – standard deviation, CV, % –variation coefficient.

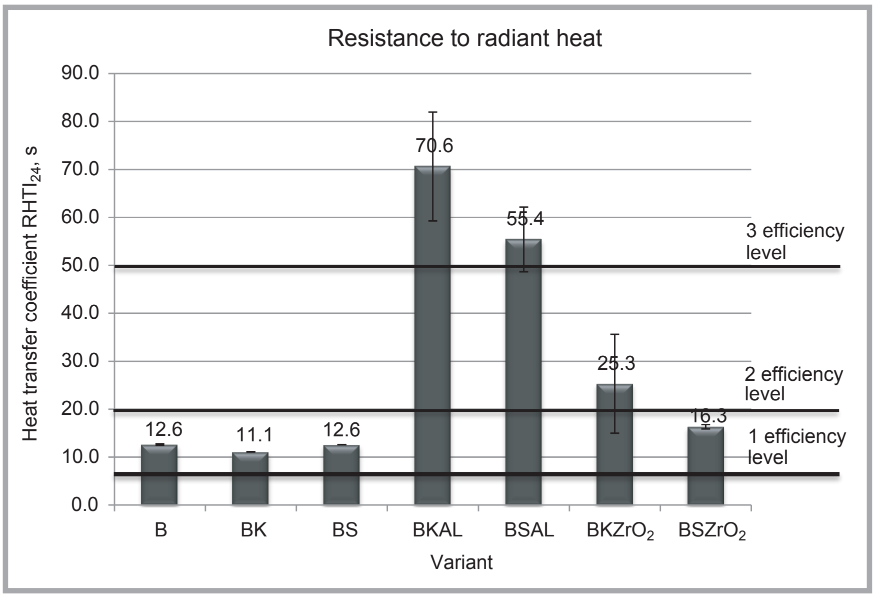

Figure 6. *List of results of thermal radiant resistance of tested samples.*

Table 5. *Efficiency levels for the resistance to radiant heat according to Standard PN-EN 407:2007 [18]*

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

In the case of the resistance to contact heat for a contact temperature of 250 °C, none of the composite variants achieved the first level of protection effectiveness. The highest resistance to a contact temperature of 250 °C was obtained for the sample of basalt fabric with silicon and foil with the zirconium (IV) oxide applied (10.2 s) and for the sample of basalt covered with silicone (8.6 s).

Compared with the first test stage (***Figure 1***), involving the direct deposition of selected chemical compounds on the basalt fabric surface, an improvement in contact heat resistance is visible for both contact temperatures. As in the first stage of the tests, the same as for the composite, the highest resistance to contact heat for contact temperatures of 100 °C and 250 °C was observed for samples containing zirconium oxide.

The research goal was to achieve resistance to contact heat for a contact temperature of 250 °C while maintaining the best resistance to heat radiation.

Results of radiant heat resistance tests

The classification of protective glove protection efficiency depending on the heat transfer coefficient is presented in ***Table 5***.

Figure 6 presents a summary of results for all samples tested for the radiant heat resistance when exposed to thermal radiation at a flux density of 20 kW/m^2. The levels of efficiency are marked in accordance with ***Table 5***.

As was exprected, the third level of radiant heat resistance was demonstrated by the fabric composite made of basalt fibre yarn with an adhesive and foil with aluminium applied, as well as by the basalt fabric with silicone and foil with aluminum applied. The second level of thermal radiant protection efficiency at a flux density of 20 kW/m^2 was obtained for the basalt fabric composite with an adhesive and Mylar foil with zirconium (IV) oxide applied. The remaining samples showed the first level of protection against thermal radiant.

Composite samples showed a similar tendency of resistance to radiant heat to those in the first stage of testing, which means that a content of metals (chromium, aluminum) increases the efficiency of protection against heat radiant.

As expected, in accordance with the results presented in the project [7, 8], basalt fabric with an aluminum coating shows the highest level of protection against heat radition.

Conclusions

The basalt composites presented show an improvement in resistance to contact heat at a contact temperature of 100 °C and 250 °C and in resistance to thermal radiant at a 20 kW/m^2 flux density compared to the first stage of the research [2, 4], which relied on the direct deposition of selected chemical compounds on the surface of basalt fabric. The research shows that when the resistance to contact heat of the samples tested improves, the resistance to thermal radiant deteriorates, and vice versa. However, the authors are striving to achieve resistance to contact heat at a contact temperature of 250 °C, while at the same time they desire to achieve even better, or at least not worse, resistance to thermal radiation.

Achieving resistance to a contact temperature of 250 °C would allow the composite to be used in the palm part of protective gloves, while maintaining a mass per unit area of 298 g/m^2 for basalt fabric.

The authors would like to use the magnetron sputtering method for the creation of a basalt composite intended for gloves protecting against the effects of hot factors, which would be an innovative solution in material engineering, certainly in the textile sector. The further research direction is associated with coating by aluminum and zirconia (IV) oxide of selected thickness.

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