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Effect of Metallisation of Basalt Fabric on Its Surface Resistivity

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Abstract
The main aim of the research was to carry out the surface modification of basalt fabric in order to improve selected thermal properties of the material, i.e., resistance to contact and radiant heat, to apply it in a hot work environment. Attention was focused on checking whether the metal coating deposited does not cause too high conductivity, which is dangerous to human health. The process of magnetron sputtering with the use of chromium and aluminum of various thickness values was applied to basalt fabrics. Based on scanning electron microscope analysis, it was found that none of the aluminum or chromium coatings covered 100% of the basalt fabric surface. Results of the surface resistance analysis of four out of the six samples of basalt fabrics tested indicate that they belong to anti-static materials. The metallic coatings obtained can potentially be used to improve the thermal properties of basalt fabric mentioned.

Key words: *basalt fabric, magnetron sputtering, surface metallisation, SEM analysis, surface resistivity.*

Introduction

Currently, on the textile and clothing market, the development of new techniques and technologies related to the production of increasingly innovative products is observed. New technologies in the clothing industry are designed to provide the user with, first of all, comfort of use and functionality under certain conditions. The eco-friendly mineral raw material which is basalt fibre is primarily used in the textile industry for the production of technical products and clothing resistant to thermal and mechanical factors and its components [1] as well as personal protective equipment, for example, protective glove elements [2]. These fibres are produced by the process of melting rock basalt, which occurs at a temperature of about 1450 °C. The basalt fibres obtained are an alternative to some high-performance fibres. An additional advantage is the lower cost of their production compared to glass fibres, which are also used to produce clothing that protects against high temperatures [3-5]. Basalt fibres are eco-compatible materials characterised by easier recyclability if compared, for example, to glass fibres. Basalt textiles are used for the reinforcement of concrete and for the production of composites. In addition, they can be used in the form of filtration materials, mainly in the chemical, petrochemical and oil industries [6]. Fabrics made of basalt fibres are used as acoustic, thermal and fireproof curtains due to their fire retardant properties [7]. They also serve as a reinforcement of road surfaces; and geotextiles and nettles made of them are used in the structure of concrete constructions.

Surface engineering is a part of material engineering, which covers issues related to the formation of coatings on and under the surface of selected materials. Surface coatings have different properties from the basic material [8-11]. Surface modification of material can be carried out using the process of Physical Vapor Deposition (PVD), consisting of creating a coating on a specific substrate as a result of the physical application of particles, ions or atoms of selected chemical compounds [8-12]. This process is carried out in a vacuum. By applying the PVD process, it is possible to cover almost all materials. The PVD process methods allow to obtain coatings with different thicknesses and various physical and chemical properties [11]. Coatings deposited as a result of selected PVD process methods are characterised by a column structure of the coating, which is responsible for the high resistance to thermal stress [13]. In order to carry out the metallisation of a basalt fabric surface by depositing metals, the process of PVD was applied [10-11]. Experiments showed that chromium and aluminum deposition are possible on a basalt fabric surface. Chemical Vapor Deposition (CVD) is a process whereby a solid material is deposited from vapour by a chemical reaction occurring on or in the vicinity of normally heated substrate surface. The resulting solid material can be in the form of thin film [12]. The process can be applied to basalt fabric, for example, in order to graft carbon nanotubes onto the material [14].

Reactive magnetron sputtering (RMS) is a method of PVD enabling in particular depositing metals on the surface of basalt fabric [9-12]. RMS is a method of spraying a material that is the substrate of a coating produced by gas ions obtained in the area between the plasma and the batch. The atomised ions then pass through the plasma, undergoing ionisation or reactions containing ions and atoms of the reactive gas during this time, which leads to coating deposition on the material surface. With the selected technique of PVD, thin coatings of one-component materials as well as chemical compounds or alloys are obtained. An important advantage of using magnetron sputtering is the possibility of high-performance and at the same time the uniform production of coatings on surfaces of various sizes and shapes. The reactive magnetron sputtering method has largely replaced methods from the PVD and CVD groups. Its basic advantages include material versatility and flexibility of applications, and the ability to apply coatings on substrates of various sizes and properties, including substrates requiring low temperatures in application processes.

Currently, research is being conducted all over the world on the use and application of selected methods of the PVD process in the field of textronics, mainly in the field of conductive materials and e-textiles. Textile diodes, conductive paths, antennas and detector systems are currently being produced on the surface of textiles, which are used to record physicochemical changes occurring in the environment [15]. Korzeniewska et al. [16] conducted research on the resistance of metallic coatings deposited on Cordura composite material used in textronic

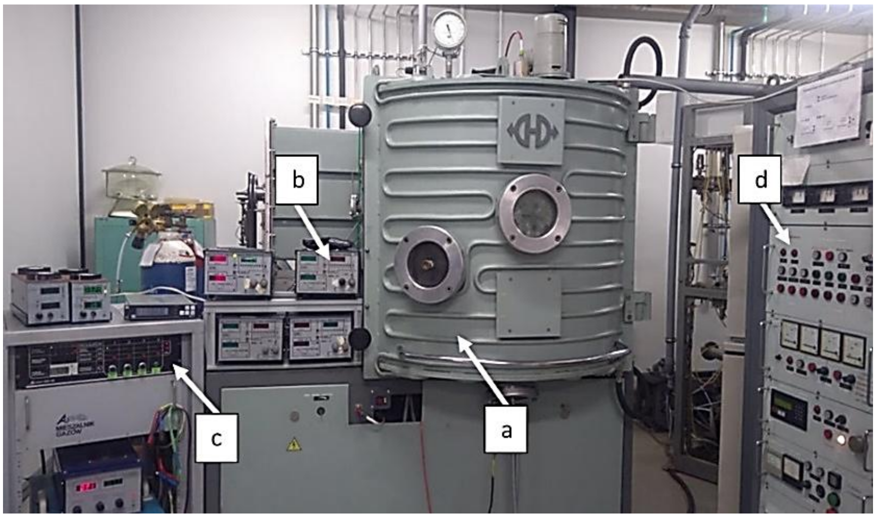


Figure 1. *Stand for depositing coatings using the magnetron method: a) vacuum chamber, b) power magnetrons and power supplies for vacuum pumps, c) working gas mixer, d) control cabinets for the set of pumps and vacuum valves [21].*

systems for mechanical deformations. It was observed that thin electrically conductive coatings obtained during vacuum deposition without protecting their surface against effects of destructive factors showed high resistance to cyclical mechanical deformations. Research was also carried out on the deposition of pure gold and silver coatings on the same composite material [17]. It was found that there was high resistance of the deposited coatings to tensile stress. In addition, the gold coating had a very good chemical resistance. Other studies were related to the optical and electrical properties of polyester fabric with a silver composite coating containing titanium dioxide which was produced by magnetron sputtering. Coatings were obtained using direct current (DC) magnetron sputtering and radio frequency (RF) alternating current. The Ag/TiO_2 composite coating was produced as follows: a thin silver coating was deposited on a polyester fabric using constant current magnetron sputtering, while another titanium dioxide coating was produced on a previously coated silver fabric using alternating magnetron sputtering. The antistatic and anti-UV properties of the modified fabric were found to be improved [18].

The main aim of the current research was to carry out the surface modification of basalt fabric in order to improve selected thermal properties of the material, i.e. resistance to contact and radiant heat, and apply it in a hot work environment. Attention was focused on checking whether the deposited metal coating does not cause too high electrical conductivity, which is dangerous to human health. The idea of the research was born after the project named "Baglo" carried out by the Central Institute for Labour Protection and Lodz University of Technology. The project developed new solutions for protective gloves designed to protect against hot agents, providing protection against mechanical factors at the same time. Research aimed at improving the thermal properties of basalt fabrics has been continued [19, 20]. However, a new solution deserves attention. The construction of new type of protective glove uses basalt fabric, to which aluminum foil is glued with the help a special glue adhesive [21]. The third level of radiant heat resistance and the first level of contact heat resistance for a contact temperature of 100 °C were obtained for the composite.

Surface modification of basalt fabrics can improve their thermal, chemical and mechanical properties. Basalt fabrics also show a significant advantage in electrical resistivity. Experimental investigations were carried out to check the electrical behaviour of hybrid and non-hybrid woven structures composed of basalt fibres [22]. These textile fabrics were evaluated by methods measuring the surface and volume resistance and resistivity. A surface resistivity of 10^{12}-10^{14} Ωm was obtained for woven fabrics composed of basalt/polypropylene, basalt/polyester, and basalt/jute fibres with different weaves. Electrical properties are influenced by the size of the electrostatic charge, which is directly related to the size of the electrical and thermal resistances of textile fabrics [23]. The high resistivity of material allows large electrostatic charges to accumulate. Antistatic materials have much reduced resistivity, so that the electrostatic charge may be dissipated by leakage currents. Additionally, basalt fabrics can be efficiently used in electrical resistance applications, replacing, for example, conventional polymers, which as synthetic materials are very sensitive to static electricity. In the article, the chromium and aluminum of different coating thicknesses were used in the process of magnetron sputtering. The effect of metallization of basalt fabric with metals on its surface resistivity is presented from the point of view of use as a protective material in the hot environment. It is important to keep the anti-static properties of the coated basalt fabric. The materials are expected to protect, for example, the user's hands and prevent an electrostatic discharge. Quality of coatings was assigned using the scanning electron microscope (SEM) analysis.

Materials

A commercially available fabric made of basalt yarns (Basaltex a.s., *Šumperk*, Czech Republic) was selected for tests. Basalt fabric is a good non-flammable thermal insulator. The material presents particular advantages when it comes to resistance against: water, most chemicals and other aggressive substances, mostly alkali, organic and inorganic acids, organic solvents and UV radiation. The basalt fabric chosen is characterised by a twill 2/2 weave with the following structural parameters: mass per square meter – 398 g/m^2 and thickness – 0.55 mm.

In order to improve the protective properties of basalt fabric, the metallization process by means of magnetron sputtering was used. Moreover, without a proper coating, basalt fabrics can cause eye and respiratory irritation as well as, above all, skin redness and irritation. The textile substrate was metalised with the use of chromium or aluminum. The resistivity of the metals is about 10^{-8} Ωm. Both metals can be used as thin film coating materials and can improve the mechanical and thermal properties of a textile substrate. It is expected that chromium and aluminum coatings will improve the resistance to heat radiation of basalt fabric**,** while maintaining the anti-static properties of the coated material.

Five rectangular samples with dimensions of 21 x 8 cm were cut from the basalt fabric.

A device for depositing coatings using the magnetron method was used to deposit coatings on the basalt fabric surface [21, 24]. The magnetron deposition stand consists of the following components: vacuum chamber, power magnetrons and power supplies for vacuum pumps, working gas mixer, control cabinets for the set of pumps and vacuum valves (see ***Figure 1***).

Moreover, a set of vacuum pumps and an electric power box are also parts of the stand.

One-sided coating was created directly on the fabric surface. The vacuum unit was pumped to a base pressure of $\sim 3 \cdot 10^{-3}$ Pa. Next, argon (5 N) was introduced to the chamber in order to obtain a working pressure of about 0.5 Pa. During one-axis rotation of the basalt fabrics, metallic coatings were deposited with a power of 1 kW. A standard, balanced WMK-100 magnetron was used with chromium or aluminum targets 100 mm in diameter. The thickness is dependent on the time of deposition, therefore it was decided to obtain 1 µm, 5 µm and 20 µm for the chromium coating as well as two variants of thickness: 1 µm and 5 µm for the aluminum. No additional heating was used during the deposition.

Using an Olympus SZX10 stereo microscope and Olympus Stream Start software, images of the basalt fabric and coated basalt fabrics with a selected coating thickness of 5 µm were captured at a total visual magnification of 9.5×, presented in ***Table 1***.

Research methodology

Quality of coatings were assigned using SEM analysis. A scanning electron microscope was used for determination of the content of individual elements on the modified surface of the basalt fabric. SEM is a type of electron microscope that produces images of the sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. A JEOL scanning electron microscope, model JSM-6610 LV with special software EDS AZtecEnergy, owned by the Institute of Materials Science and Engineering of Lodz University of Technology, was used (***Figure 2***).

Technical data of the scanning electron microscope are as follows:
- HV resolution mode: 3.0 nm (30 kV), 8 nm (3 kV), 15 nm (1 kV),
- magnification from 5× to 300000×,
- maximum sample: 200 mm diameter,
- automatic functions: contrast, sharpness, brightness.

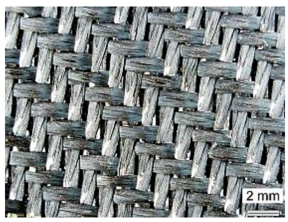
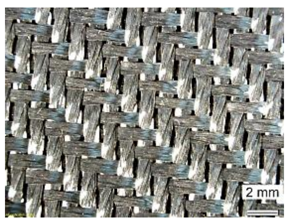
It is important to keep the anti-static properties of the coated basalt fabric from the point of view of use as a protective material in a hot environment. Due to this requirement, appropriate surface resistance is necessary. The surface resistivity of the coated basalt fabrics was determined according to the ASTM D257-14 standard [25], which makes it possible to test samples with a much smaller surface than those mentioned in the European standard [26]. The test method covers direct-current procedures for the measurement of DC insulation resistance, volume resistance and surface resistance. From the measurements and geometric dimensions of the material sample and electrodes, the surface resistivity of the material can be calculated. In this study a Keithley Electrometer/High Resistance Meter, Model 6517A, 5½ digit resolution, was used. The model can make surface resistivity measurements from 10^3 Ω to 10^{17} Ω. The resistance was measured by applying a voltage potential across the surface of the insulator sample and measuring the resultant current using a Test Fixture Model 8009 equipped with three concentric electrodes (***Figure 3***). In accordance with the ASTM D257-14 standard [25] a voltage of 500 V was applied for an electrification time equal to 60 s.

Figure 2. *Scanning electron microscope.*

Surface resistivity ρ_s, displayed on a front panel, is automatically calculated according to the following formula:

$$\rho_s = \frac{\pi D_0}{g} R \qquad (1)$$

Table 1. *Characteristics of selected materials*

Symbol	Fabric description	Microscopic images
V^o	Fabric made of basalt fibres	
V_1^1	Basalt fabric metallisation with a chromium coating of 1 µm thickness	
V_5^1	Basalt fabric metallisation with a chromium coating of 5 µm thickness	
V_{20}^1	Basalt fabric metallisation with a chromium coating of 20 µm thickness	
V_1^2	Basalt fabric metallisation with a chromium coating of 1 µm thickness	
V_5^2	Basalt fabric metallisation with a chromium coating of 5 µm thickness	

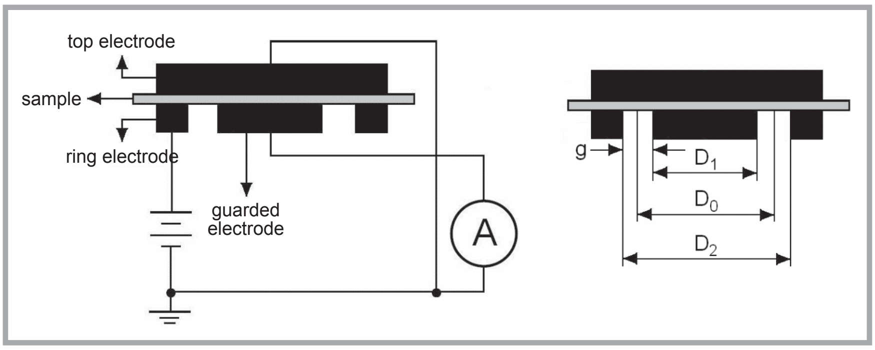


Figure 3. *Surface resistivity measurement technique and electrode dimensions.*

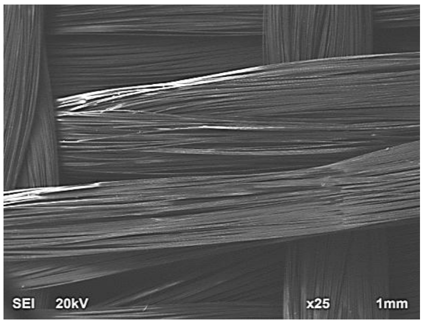


Figure 4. *View of 1 µm chromium coating deposited on the surface of basalt fabric.*

where, R – measured resistance, Ω; $D_0 = D_1 + g$; D_1 – diameter of the guarded electrode, mm; g – distance between the guarded electrode and ring electrode, mm; and

$$g = 0.5(D_1 - D_2) \quad (2)$$

where, D_2 – inner diameter of the ring electrode, mm.

Results and discussion

On the scanning electron microscope, images of the metallised surfaces of the basalt fabrics were taken. Content analysis of individual elements present in each coating deposited was conducted, shown in ***Table 2***.

As seen in ***Table 2***, the coating of chromium of 1 µm thickness allowed for determination of the chemical composition of the basalt fabric with the use of the EDS method. The chromium content is small (0.1 at. %). Similar thickness of Al coating did not allow for efficient fabric metallisation. The surface of this sample was charged, hence no image or chemical composition could be registered. In the case of thick coatings, due to the depth of electron penetration, the chemical composition showed a huge amount of chromium (93.7 or 96.7 at. % for 5 or 20 µm, respectively) and only 77.5 at. % of aluminum. This can be explained by the well-known dependence that electrons penetrate elements with a low atomic mass more easily (Al in this case), which should be treated as an expected phenomenon.

Figure 4 shows the basalt fabric surface after metallisation with a chromium coating of 1 µm thickness, which was obtained using the scanning electron microscope.

As is shown, the colour of deposited coating is missing due to its very small thickness. The thin chromium coating is barely noticeable.

Table 3 presents quantitative and colour maps of the content of the main, individual chemical elements (oxygen, silicon and chromium) on the basalt fabric surface covered with a 1 µm thick chromium coating. The rest of the constituents can be checked in ***Table 2***. The colour analysis using the organoleptic method was based on observing the colour of sample. The appropriate colour was specified for each chemical element in the coating. The analysis confirmed a slight chromium content in the coating, below 1% (the green colour) (***Table 3***). The red colour indicates a high content of oxygen. The blue colour shows almost 16% percent of silicon in the coating.

The basalt fabric surface after metallisation with a chromium coating of 5 µm thickness is shown in ***Figure 5***.

In this case the results of analysis show almost 94% of chromium content (the green color) in the coating (***Table 4***). Oxygen (the blue colour) constitutes over 4% of the total composition.

Results of basalt fabric metallisation with the chromium coating of 20 µm thickness is shown in ***Figure 6***. The predominant colour is red, indicating very high content in the chromium deposit.

The quantitative and colour analysis showed almost 97% chromium content.

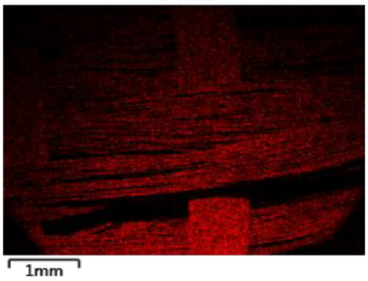
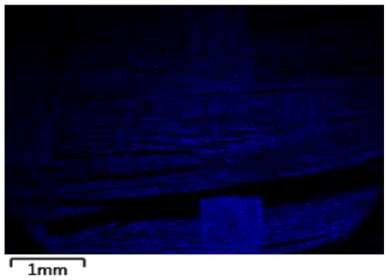
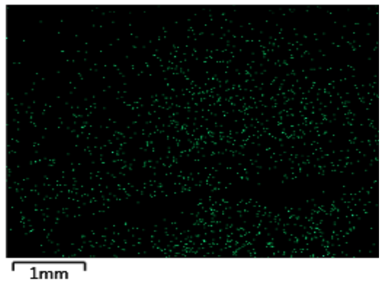
The aluminum coating on the surface of 1 µm thick basalt fabric did not possess an electric charge that could allow for analysis by scanning electron microscope of elements contained therein. In the case of aluminum coating with a thickness of 5 µm (***Figure 7***), no colour analysis was obtained. However, it was possible to analyse any chosen point of aluminum deposited on the basalt fabric surface. The 5 µm thick aluminum coating consisted of almost 78% aluminum, 15% oxygen and 6% silicon.

Quantitative and colour analysis of the individual chemical element contents in the coating is more accurate the greater the thickness of the coating deposited is. 1 µm thick coatings are too thin to obtain a readable image on SEM. It can be concluded that the magnetron sputtering method allows the production of even coatings on different surfaces and shapes. In addition, the coatings deposited have a column structure.

Table 2. *Chemical composition (at. %) of chromium or aluminum coatings deposited by means of magnetron sputtering onto basalt fabric.* ***Note:*** ** The thickness of this aluminum coating was too small to allow efficient imaging with the SEM technique. The image was charged by an electron beam, hence no determination of chemical composition was performed.*

Coating type	Thickness, µm				
	Chromium			Aluminium	
	1	5	20	1	5
O	**73.3**	**4.3**	2.1	n.a.*	**14.8**
Si	**15.6**	0.8	0.6		**5.8**
Al	7.3	0.3	0.4		**77.5**
Ca	2.0	0.4	0.1		1.1
Fe	1.5	0.4	0.1		0.8
Ti	0.2	0.1	0.0		0.1
Cr	0.1	**93.7**	**96.7**		–

Table 3. Analysis of the chemical constituent present in the 1 µm chromium coating.

O Kα1	Si Kα1	Cr Kα1
		
Coating composition		
Oxygen 73.3%	Silicon 15.6%	Chromium 0.1%

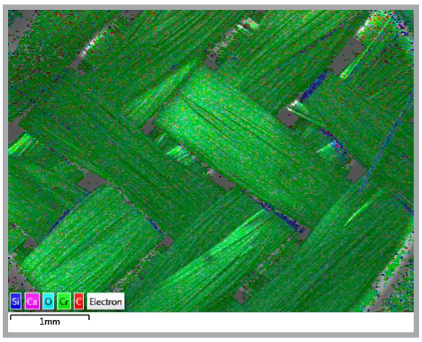


Figure 5. *View of 5 µm chromium coating deposited on the surface of basalt fabric.*

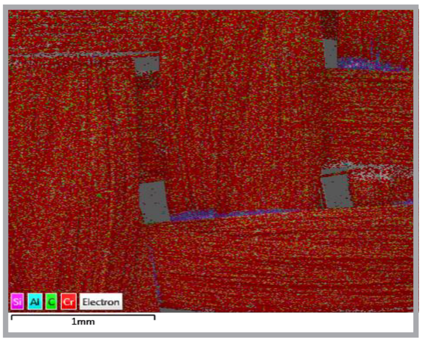


Figure 6. *View of 20 µm chromium coating deposited on the surface of basalt fabric.*

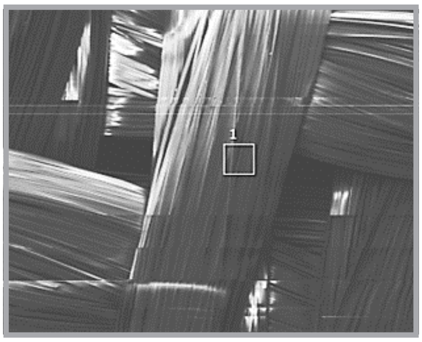
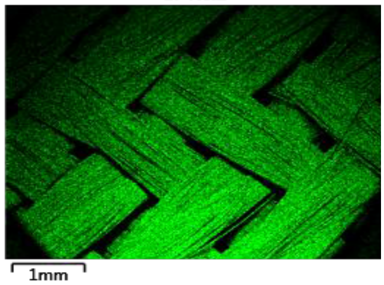
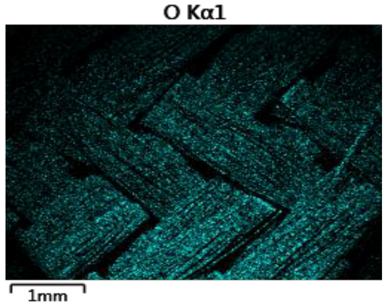


Figure 7. *View of the measuring point of 5 µm thick aluminum coating deposited on basalt fabric surface.*

Research on the electro-conductive properties of the coatings deposited on the surface of basalt fabric was carried out. It is necessary to state if the surface modification changes the electrical properties of basalt fabric, which is important from the point of view of the electrical behaviour connected with static electricity generated on the materials. The surface resistivity of the coated basalt fabric, which indirectly describes the ability to conduct electric current on the modified sample surface, was determined. Two samples of each coated and uncoated material were prepared and measurements repeated 3 times for each sample. The mean value of the surface resistivity, standard deviation and variation coefficient were calculated. Results obtained are given in ***Table 5***.

Table 4. Analysis of the chemical constituent contained in the 5 µm chromium coating.

Cr Kα1	O Kα1
	
Coating composition	
Chromium 93.7%	Oxygen 4.3%

An increase in the surface resistivity of the coated basalt fabric in relation to the uncoated fabric was observed. The variation coefficient of the value measured is in the range 21-33%. In the case of fabric metallised by a chromium coating, a thickness of 5 µm was enough to significantly increase the surface resistivity above the range of the measuring instrument. In general, it was observed that the metallisation of basalt fabric caused an increase in surface resistivity even though metals were deposited on the surface. With regard to the reference sample Vo and modified samples: V_1^1, V_1^2, and V_5^2, it can be stated that the deposited metal coating does not cause too high conductivity, which is dangerous to human health. Materials protecting hands from heat in a hot work environment can also guard against electrostatic discharge. Results of the surface resistances of the basalt fabrics indicate that they are within the limit of anti-static materials. It seems that samples V_5^1 and V_{20}^1 should be treated as insulating materials; surface modification requires a repetition. The reason for the increase in surface resistivity of the basalt fabrics observed, despite covering them with excellent conductive materials, is visible in the SEM analysis and coating quality. It was noticed that none of the aluminum or chromium coatings covered 100% of the basalt fabric surface. A lack of coating continuity is seen in the structure of the substrate, which is characterised by its surface roughness.

Table 5. *Surface resistivity of chromium and aluminum coatings.* ***Note:*** ** Model 6517A can make surface resistivity measurements from* $10^3\ \Omega$ *to* $10^{17}\ \Omega$*.*

Symbol	Surface resistivity, Ω	Standard deviation, Ω	Variation coefficient, %
V^o	$1.93 \cdot 10^{13}$	$4.12 \cdot 10^{12}$	21
V_1^1	$2.22 \cdot 10^{14}$	$5.49 \cdot 10^{13}$	25
V_5^1	out of range*	–	–
V_{20}^1	out of range*	–	–
V_1^2	$1.26 \cdot 10^{14}$	$2.70 \cdot 10^{13}$	21
V_5^2	$2.26 \cdot 10^{14}$	$7.48 \cdot 10^{13}$	33

Conclusions

The research results presented led to the following conclusions:

- the metallisation of basalt fabric caused an increase in surface resistivity even though metals were deposited on the surface;
- based on scanning electron microscope analysis, it was found that none of the aluminum or chromium coatings covered 100% of the basalt fabric surface;
- values of the surface resistance of four out of the six samples of basalt fabrics tested (including unmodified basalt fabric) indicate that they are within the limit for anti-static materials; the remaining samples are insulating materials;
- the four basalt composites obtained can potentially be used to improve the thermal properties of basalt fabric investigated, i.e., resistance to contact and radiant heat, and can be applied as a material for use in a hot work environment.

Declaration of conflicting interests

The authors declare no potential conflicts of interest with respect to the research, authorship and/or publication of this article.

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