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Thin-Film Deposition Systems for Obtaining Conductive Textiles

Emilia Visileanu*, Alexandra Gabriela Ene, Cătălin Marian Grosu, Razvan Victor Scarlat and Laurentiu Dinca

The National Research and Development Institute for Textiles and Leather 16, Lucretiu Patrascanu Street, sector 3, zip code 030508, Bucharest, Romania

ABSTRACT

Smart textiles are fabrics with technological components that perform different wearer functions. Functions can vary, but examples of use are garments that can heat cool or change colour. The idea of our project was to create a device that would stop blood flow without the injured person's intervention. The detection of the wound is realised using a conductive textile structure made of a thin metal film on its surface. When a circuit break occurs, it will generate an electrical signal interpreted by a control unit to operate an air pumping system that activates a pneumatic tourniquet that stops the flow of blood. To accomplish conductive textile structures the EB-PVD method and the Sputtering method were applied and different metals were used, such as Cu, Ag etc. The amount of metal deposition was 4,0-7,00µg/cm2. Samples were studied on the surface and in sections by scanning electron microscopy in Low Vacuum mode, using the backscattered secondary electron (ABS) detector and the energy dispersive spectroscopy (EDS) detector. The electrical resistivity (Ω) was determined and conductivity (S/m) was calculated for initially treated textile structures and after washing and abrasion tests.

***Corresponding author**

Emilia Visileanu, The National Research and Development Institute for Textiles and Leather 16, Lucretiu Patrascanu Street, sector 3, zip code 030508, Bucharest, Romania.

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Introduction

Conductive Textile Market Size was valued at USD 2.7 billion in 2022. The conductive textile industry is projected to grow from USD 3.1 Billion in 2023 to USD 10.5 billion by 2032, exhibiting a compound annual growth rate (CAGR) of 16.30% during the forecast period (2023 - 2032). High demand from the military and defence sectors, as well as a growing market for smart fabrics, is the key market driver enhancing the market growth. The military & defence segment has a significant market share, because the use of conductive wearables, which can monitor the battlefield, track soldier health, and assist communications and temperature monitoring, is expanding. Furthermore, political uncertainty, combined with the advancement of advanced technology, is projected to enhance demand for products used in military and defence applications.

The idea of the research project was to create a device that would stop blood flow without the injured person's intervention. The detection of the wound is realised using a conductive textile structure made of a thin metal film on its surface.

Conductive textile materials are, strictly speaking, not intelligent. They do not react to their environment, but they make many smart textile applications possible, especially those that monitor body functions. They are widely used in smart textile applications such as sensors, communication, heating textiles and electrostatic discharge clothing. Electroconductive materials are required in sensors, actuators and heating panels, and the best-suited materials are highly conductive metals such as copper, silver and steel [1].

Conductive fabrics can be produced by coating the non-conductive fabric with conductive materials, using coating processes such as: spraying, electroless plating, sputter coating, plasma treatment, vacuum metallisation, in situ polymerisation, chemical vapour deposition, textile coating/printing, etc.

Physical vapour deposition, often referred to by the abbreviation "PVD", is a process where the material is vaporized as a target in the process chamber and applied as a thin film to the surface to be coated (substrate). The most frequently used and, from an economic standpoint, most important PVD process is sputtering. In the PVD process, the material – known as the target – is kept in solid form in the system for subsequent thin film coating. This material is then vaporized and grows as a thin film on the substrate surface. Various methods can be deployed when vaporizing the material, e.g. with a laser, an electric arc or by bombarding it with particles.

There are also PVD techniques in which the material is transferred into a gas state by heating (thermal vapour deposition). Molecular beam epitaxy and ion beam-guided deposition also belong to the group of PVD processes. The resulting coatings are particularly pure, and uniform and achieve a very high level of adhesion to the substrate. PVD coatings, therefore, offer an environmentally friendly alternative to conventional electrochemical processes for numerous areas of application.

This article presents the obtaining conductive textile structures via EB-PVD and the hybrid PVD by DC/RF sputtering method with copper and silver targets and analysis of them from the structural and electrical properties point of view.

Equipment for Accomplishing Thin Films

EB-PVD

Electron beam deposition installation (BP-PVD) Torr-Model No: 5X300EB-45KW was used. The system is designed to be able to combine several layers, having 4 guns with electron emission, with a power of 10 kW/ton,1000 mA. Each gun is equipped with a carousel system with 4 "pockets", in which 4 crucibles with a volume of 75 cm3 each can be placed, thus allowing an almost continuous evaporation of up to 16 different materials from all 4 guns. The installation has the possibility of deposition on rotating 3D plates or cylinders with a maximum length of 350 mm; simultaneous or multilayer deposition of thin films; heating of the substrates during deposition; melting and deposition of grains of metals and alloys (W, Ni, Co, Cr, Al, Y), dielectric materials (Al203, Si02, Zr02, etc.) and compounds [2].

The textile materials used as the substrate were fixed on the support of the deposition installation (Figure 1).

For the EB-PVD deposition of copper and silver on textile materials, a crucible was loaded with Cu material and Ag of 99,99% purity.

Working parameters: starting vacuum: 10-6 Torr, working vacuum: 5x10-5 Torr, deposition speed (Å/s):10, maxim power e-beam: 100 KW, the amount of deposition: Cu: 4- 5µg/cm2 and for Ag:6-7 µg/cm2.

The technological flow included the following phases: preparation of the substrate: cleaning and degreasing of the surface, loading of substrates and deposition materials into the vacuum chamber, vacuuming of the chamber (advanced vacuum up to 10-7 Torr), evaporation of material: Ag, evacuation and ventilation of the chamber, extraction of samples .

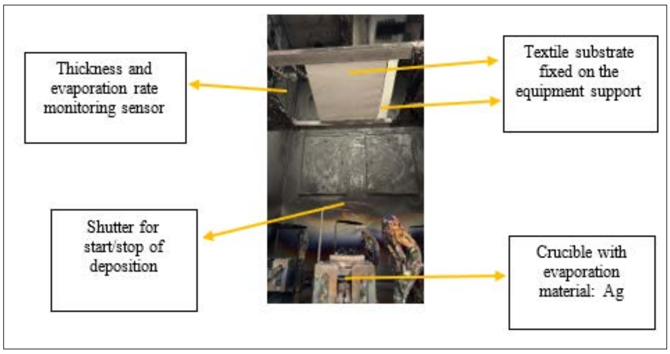


Figure 1: Sistem EB-PVD

Sputtering

The sputtering phenomenon occurs when energetic particles of a gas or plasma (incident ions) bombard a material, also known as the target. One of the most used sources for the incident ions is plasma. Magnetron sputtering, even with direct current (DC) or radio-frequency (RF) uses a magnetic field and an electric field to confine particles near the surface of the target, leading to the growth of the ion density and generating a high rate of sputtering [3].

Thin film deposition was performed on a hybrid PVD (Physical Vapor Deposition) system with a DC/RF Magnetron Sputtering module. The equipment is manufactured by Kurt J. Lesker and consists of a 2-inch diameter TORUS magnetron sputtering cannon/circular box, a PD500x DC source with a maximum power of 1500W, and an R301 RF source with a maximum power of 300 W (Figure 2a). Commercial Cu target (99.99%) and Ag target (99,99%) with a diameter of 2 inches and thickness of 3 mm were used for deposition and were mounted on the magnetron sputtering cathode (Figure 2b). Starting vacuum: 9*10 e^{-6} mbar, working vacuum: 1.9*10e^{-5} mbar, flow Ar: 50 ml/min, power source DC: 75 W, deposition time: 120 min, distance between crucible and substrate: 0.8 m, Ar flow: 50 ml/min.

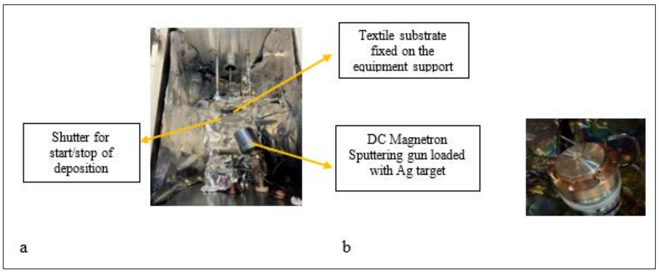


Figure 2: (a) Sputtering System; (b) Target

Materials and Methods

Textile Substrate

For the experiment, a woven textile substrate made of 100% cotton, with a density of 610 yarns/10cm (warp) and 360 yarns/10 cm (weft) and a mass of 207 g/m2. The textile structure was treated (padding technologies) with 3 different substances: ITOBINDER-Acrylate and PERMUTEX-EX-RU-Urethane to obtain in the deposition process of metal ions a continuous film on their surfaces, which ensures continuity from the view of electrical properties. Previous research has revealed the tendency of metal ions to penetrate between the fibres of the textile substrate so that their deposition didn't form a continuous film (Figure 3).

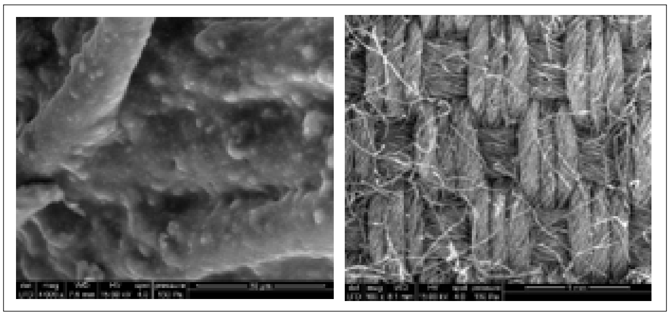


Figure 3: Textile structures, before and after treatments

Washing and Abrasion Tests

The washing test of the coated textile structures was carried out by applying the SR EN ISO - CO 6/2010 standard and using the James Heal Gyrowash - washing and dry-cleaning equipment, measuring range 0-100°C, precision 2°C. ECE detergent, without optical agent and without phosphates, washing temperature 40°C, washing time 30 minutes.

The abrasion resistance test was carried out according to SR EN ISO 12947-2: 2017 and the use of the James Heal Maxi-Martindale Multidirectional Action Device (series 1609/22/1555) model 1609. Abrasion load of 12KPa. Number of cycles: 1000, 3000 and 5000.

Electrical Properties

The electrical properties of textile-based materials are influenced by the elasticity and deformability of textile-based products and unstable conductive coatings [4]. In addition, the washability issue reducing the reliability of e-textiles is always a challenge for electronic textiles in terms of application [5]. The electrical resistivity (Ω) was determined on one inch of the textile surface, using the BX PRECISION 889B Bench LRC/ESR METER and the electrical conductivity was calculated (1):

$$\rho = \frac{U}{I} \cdot \frac{D \cdot g}{L} = R \cdot \frac{D \cdot g}{L} \qquad (1)$$

SEM and EDAX Analysis

The analysis of the 2D morphology of the surfaces before and after the washing and abrasion tests was carried out by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectrometry (EDX) and using the FEI Quanta 200 Electron Microscope with an Ametek X-ray Detection System EDAX Element.

Results and Discussion

Table 1 shows the SEM images of the non-treated textile structure, coated with Cu by the EB-PVD method, and Table 2 shows the surface aspects of the textile structures treated with ITOBINDER-AG Acrylate or PERMUTEX-EX-RU-Uretan and coated with a thin film of with the EB-PVD method, after washing and rubbing tests (organoleptic evaluation).

Table 1

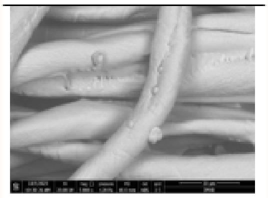
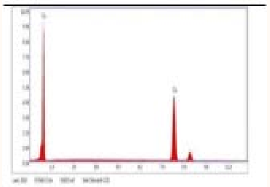
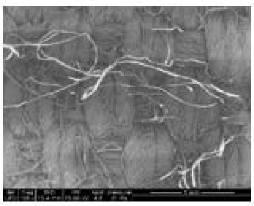
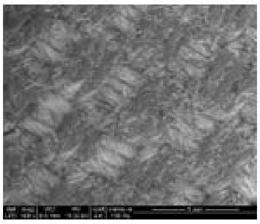
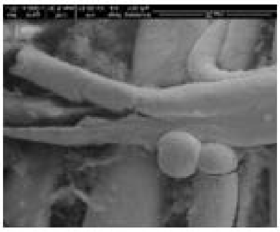
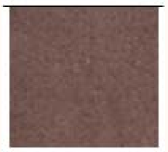
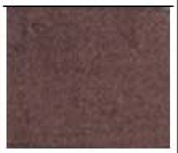
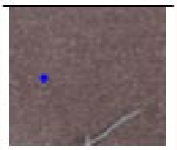
SEM and EDX- EB-PVD-Itobinder AG – Cu images		
		
		
After 5 washing cycles	After 3000 rubbing cycles	

Table 2

Surface appearance - Sample after washing					
1 cycle		5 cycles		1 cycle	5 cycles
ITOBINDER	Permutex	Itobinder	Permutex	Non-treated	
					

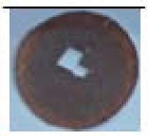
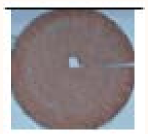
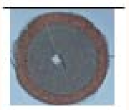
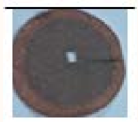
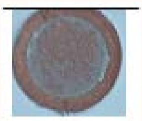
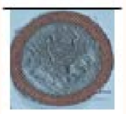
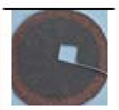
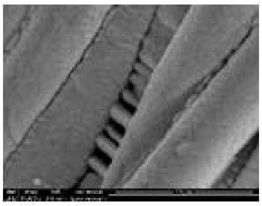
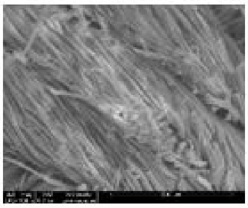
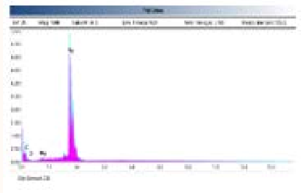
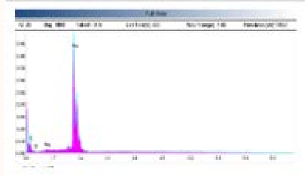
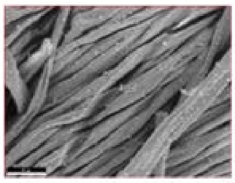
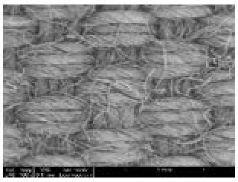
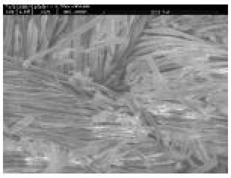
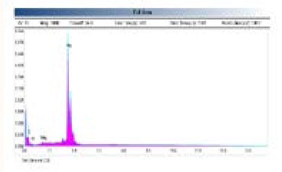
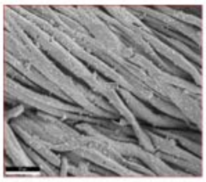
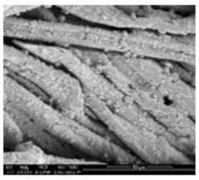
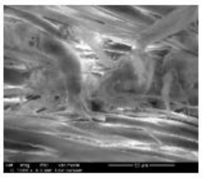
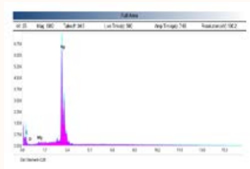
The sample after the abrasion test								
1000 cycles			3000 cycles			5000 cicli		
N	Itobinder	Permutex	N	Itobinder	Permutex	N	Itobinder	Permutex
							Broken	Broken

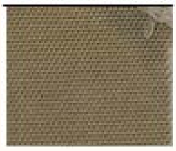
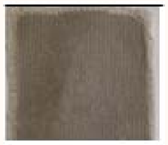
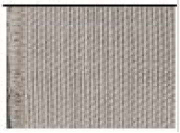
Table 3 show the SEM images of the non-treated textile structure, coated with Ag by the EB-PVD and Sputtering method, and Table 4 shows the surface aspects of the textile structures treated with ITOBINDER-AG-Acrylate or PERMUTEX-EX-RU-Urethane and coated with thin layer film of Ag by the EB-PVD and Sputtering methods after washing and rubbing tests (organoleptic evaluation).

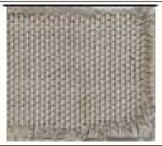
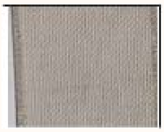
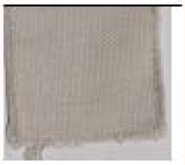
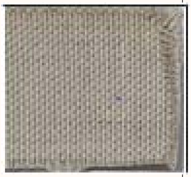
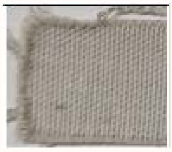
Table 3

			
SEM-Itobinder AG -Sputtering; a)initial,b)after washing c)after rubbing)EDAX initial			
			
SEM-Permutex -Sputtering; a)initial,b)after washing c)after rubbing)EDAX initial			
			
SEM-ITOBINDER AG -EB-PVD; a)initial,b)after washing c)after rubbing)EDAX initial			
			
SEM-Permutex -EB-PVD; a)initial,b)after washing c)after rubbing)EDAX initial			

The SEM images revealed a fibrous structure, on the surface of which a fine coating layer was deposited. The coatings have a columnar appearance and are uniform and continuous on the surface of the textile substrates. The damage to the surfaces after washing and rubbing is highlighted. EDS point semi-quantitative chemical analyzes highlighted the presence of elements with the atomic and mass percentages C, O, Mg and also Cu and Ag in a percentage of 82.62-82.88% for the initial variant treated by Sputtering and 81.94-82.91% for the initial variant treated by the EB-PVD process. After a Sputtering washing cycle, the limits are 77.94-80.88% and for EB-PVD: 64.83-77.72%. After rubbing at 2000-3000 cycles: for Sputtering the limits are 10.0-19.96% and for EB-PVD: 4.16-12.25%.

Table 4

No	**Sample**	**Washing, cycles**		**Rubbing, cycles**		
		1 cycle	**5 cycles**	**1000 cycles**	**3000 cycles**	**5000 cycles**
1	Non-treated sample with deposition through EB-PVD					
2	The sample treated with ITOBINDER AG and deposition through EB-PVD					

3	The sample treated with PERMUTEX and deposition through EB-PVD					Broken
4	Non-treated sample with Sputtering deposition				Broken	
5	The sample treated with ITOBINDER AG with Sputtering deposition					Broken
	The sample treated with PERMUTEX and Sputtering deposition					Broken

The organoleptic evaluation of the textile structures coated with thin films by the EB-PVD method (Cu and Ag) and the Sputtering method (Ag) after 1-5 washing cycles highlights surface aspects that also showed mechanical changes compared to the variants of untested textile structures.

The evaluation of the textile structures coated with thin films by the EB-PVD method (Cu and Ag) and the Sputtering method (Ag) after the rubbing tests highlights significant mechanical changes in the surfaces compared to the variants of untested textile structures, showing napping and thread breaks. After 3000 cycles, the variant of the non-treated textile structure coated with Cu by the Sputtering method showed yarn breaks and the formation of holes on the surface. The same phenomena were observed for the variant of the textile structure treated with PERMUTEX and coated with Ag by EB-PVD, the variants of the treated textile structures with ITOBINDER AG and Permutex EX-RU with Sputtering coating with Ag, after 5000 cycles.

Electric Properties

Table 5 presents the results of resistivity measurements both for the Cu and Ag thin films deposited on the surface of textile substrates treated with ITOBINDER AG-Acrilat and PERMUTEX-EX-RU-Uretan.

From the analysis of the data presented in Table 5 and Figure 4, the following aspects result:

Table 5

Linear electrical resistance measurements (05.02.2024)									
No.	**Variant**	**Resistance, Ω**				**Average electrical resistance, Ω**	**Thick-ness, mm**	**Resistivity, Ωm**	**Conducti-vity, S/m**
1	N-SPT-Ag	1.17	1.6	1.7	1.3	1.44	0.55	0.000793375	1260.44
2	TI-SPT-Ag	4.5	4.7	4.7	4.8	4.68	0.56	0.002618	381.97
3	TP-SPT-Ag	0.22	0.37	0.12	0.4	0.28	0.59	0.000163725	6107.80
4	N-EB-PVD-Ag	7.13	20.4	10.8	10.4	12.18	0.7	0.00852775	117.26
5	TI-EB-PVD-Ag	0.15	0.17	0.2	0.17	0.17	0.52	0.0000897	11148.27
6	TP-EB-PVD-Ag	0.02	0.03	0.038	0.055	0.04	0.78	0.000027885	35861.57
7	TI-EB-PVD-Cu	0,117	0,210	0.119	0,200	0,119	0,45	0,000029	34426,67

8	TP-EB-PVD-Cu	0,559	0,661	0,662	0,559	0,663	0,43	0,000162	6179,15
7	N-EB-PVD-3000 cycles-Cu	32.6	21.6	17.6	17.1	22.23	0.52	0.011557	86.53
10	TP-EB-PVD -Cu -1 s	11.7	9.4	13.8	14.7	12.40	0.68	0.008432	118.60
11	TP-EB-PVD-Cu-_5 s	21.3	93.5	50.9	34.9	50.15	0.73	0.0366095	27.32
12	TP-EB-PVD -Ag	0.15	0.16	0.08	0.08	0.12	0.58	0.00006815	14673.51
13	TI-EB-PVD- Ag	0.2	0.15	0.26	0.27	0.22	0.56	0.0001232	8116.88
14	N-TI-RB_PVD_Cu	0.7	5.3	3.5	4.9	3.60	0.55	0.00198	505.05
15	N-TP-EB_PVD-Cu	12.2	8.4	11.9	11.8	11.08	0.88	0.009746	102.61

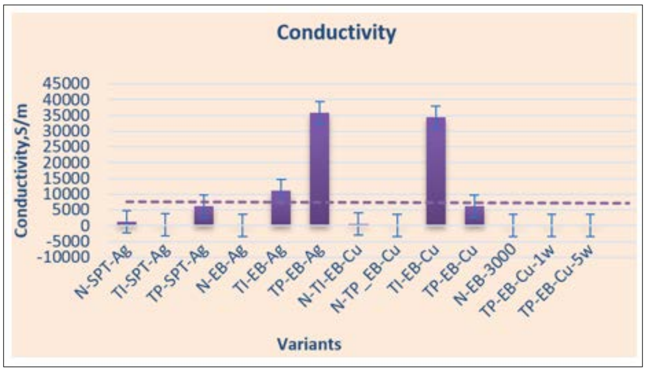


Figure 4: Conductivity evolution

- The highest values of electrical conductivity (S/m) are recorded for the variant of the textile structure treated with Permutex EX-RU and coated with Ag by the EB-PVD method (35,861.57 S/m) and the variant treated with ITOBINDER AG coated with Cu by the EB-PVD method (34,426,679 S/m);
- Good conductivity value was also obtained for the textile structure variant treated with ITOBINDER AG and coated with Ag by the EB-PVD method (11.148(S/m);
- The textile structures coated with Ag or Cu through the 2 applied methods, were tested for washing. (1 or 5 cycles) and rubbing (1000-5000 cycles) recorded changes in the metal film deposited on the surface, which led to discontinuities in the electric circuit;
- The variants that maintained the continuity of the film recorded reductions in the electrical conductivity value through:

o **Washing:** the variant of the textile structure treated with PERMUTEX EX-RU and coated with Cu by the EB-PVD method after 1 washing cycle, 118.6 S/m and 5 washing cycles: 27.32 S/m compared to the initial variant of 6179.15 S/m.

o **Friction:** the variant untreated with chemical auxiliaries and coated with metallic Cu particles by the EB-PVD method which after 3000 cycles recorded a conductivity level of 86.53 S/m compared to 6179.15 S/m.

Conclusions

- Variants of 100% cotton textile structures treated with ITOBINDER AG and PERMUTEX EX-RU coated with Cu and Ag through EB-PVD and Sputtering technologies were tested from the point of view of electrical properties after washing tests (1 and 5 cycles) and abrasion (1000, 3000 and 5000 cycles) compared to samples not coated with metal particles. SEM and EDAX analyzes highlighted the surface morphological changes after the performed tests. The highest values of electrical conductivity (S/m) are recorded for the variant of the textile structure treated with Permutex EX-RU and coated with Ag by the EB-PVD method (35861.57 S/m) and the variant treated with ITOBINDER AG coated with Cu by the EB-PVD method (34426.679 S/m);
- Washing tests (1 cycle and 5 cycles) and abrasion tests (1000-5000 cycles) produce surface morphological changes that lead to discontinuities of thin films.
- The variants that maintained the continuity of the film recorded reductions in the electrical conductivity value through:

o Washing: the variant of the textile structure treated with PERMUTEX EX-RU and coatedwith Cu by the EB-PVD method after 1 washing cycle, 118.6 S/m and 5 washing cycles: 27.32 S/m compared to the initial variant of 6179.15 S/m.

o Rubbing: the variant not treated with chemical auxiliaries and coated with metallic Cu particles by the EB-PVD method, which after 3000 cycles recorded a conductivity level of 86.53 S/m compared to 7179.15 S/m.

- In the next phase of the research, methods will be applied to increase the resistance of the metallic layers deposited on the textile supports to the washing and rubbing tests by coating with protective films/ membranes.

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