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Metallic Electroconductive Transmission Lines Obtained on Textile Substrates by Magnetron Sputtering

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Abstract
The paper discusses the results of research concerning the formation of electroconductive transmission lines on textile substrates using the magnetron sputtering technique. The transmission lines developed can potentially be applied in clothing for emergency and security services to affect electrical connections between electronic elements incorporated in the garments. The time of metallic layer deposition and the type of substrate used was optimised in the study. The surface resistivity, resistance to bending and abrasion of the transmission lines obtained were tested. The tests demonstrated that it is possible to obtain electroconductive copper layers with a surface resistivity approximating 0.2 Ω by direct deposition on spun-bonded type polypropylene nonwoven.

Key words: *magnetron sputtering, electroconductive paths, e-textile, vacuum deposition.*

Introduction

The dynamic development of research concerning textronic solutions involving the placement of electronic elements directly on textile substrates has been observed during the last decade. The authors of such research papers take advantage of both classic techniques and innovative ones which have not been used to date. The classic techniques undoubtedly include the use of electroconductive yarns to form transmission lines on the surface of textiles by weaving, knitting or embroidering [1-6]. Most of such solutions involve obtaining electroconductive yarns by galvanic deposition of metallic layers on a fibre surface [7-11], the introduction of electroconductive particles into the polymer used for fibre production, or manufacturing yarns with the use of metallic fibres [7, 8, 11-13]. Printing electronic elements on textile substrates is another classic method. Screen printing with electroconductive pastes obtained by the introduction of electroconductive particles such as metals, carbon soot and, recently, carbon nanotubules into the binder is the method most commonly used [14-16]. The relevant literature includes papers describing a jet printing method using electroconductive inks [17-20]; but that technique is still problematic in industrial practice because it is difficult to obtain print continuity. Other solutions, innovative in character, include techniques involving the deposition of thin electroconductive layers of textile substrates. There are various methods of thin layer deposition. According to the character of the deposition process, they can be divided into physical – PVD (Physical Vapor Deposition) and chemical – CVD (Chemical Vapor Deposition) [21]. Physical vacuum deposition (PVD) processes are more versatile than CVD because they allow the deposition of a differentiated group of materials (dielectrics, metals, alloys) at lower temperatures. The thickness of the deposited layers ranges from a few kiloangstrems to a few micrometers, and a specially designed vacuum chamber enables the deposition of several layers at the same time. The film deposition process in PVD is divided into three stages: bringing a refractory material to a vapour state, material vapour transport and vapour deposition on the substrate [22].

The PVD methods can be divided into three basic groups: sputtering, evaporation and. ion plating. Sputtering involves the deposition of ionised metal vapours obtained by pulverisation of a metal disc-shaped target with working gas ions (e.g. magnetron sputtering) [23, 24].

Magnetron sputtering is a process frequently applied to obtain thin and durable films in many industrial sectors: construction, automotive (dark tinted windows, mirrors), the production of optical filters, material engineering (anticorrosive coating and hardening tools) and electronics (electroconductive transmission lines and layers, sensors) [23, 25, 26]. The magnetron sputtering process can be conducted in plasma in the presence of a neutral gas (argon in the case of the deposition of metallic materials) or a reactive gas (oxygen, nitrogen). Magnetrons are crossed-field electron lamps (with perpendicular electric and magnetic fields) and a cold discharge cathode. In a magnetron sput-

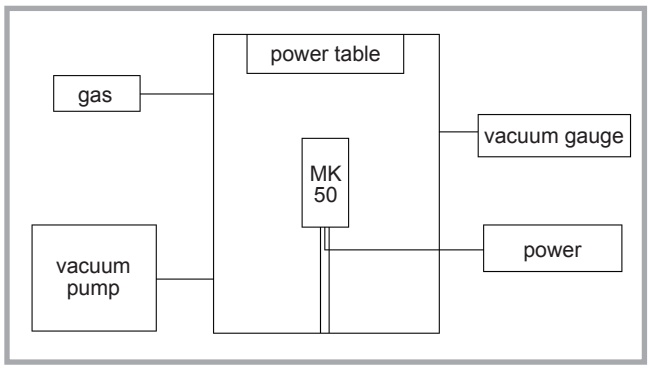


Figure 1. *Vacuum chamber of a Classic 500 sputter equipped with an MK-50 magnetron.*

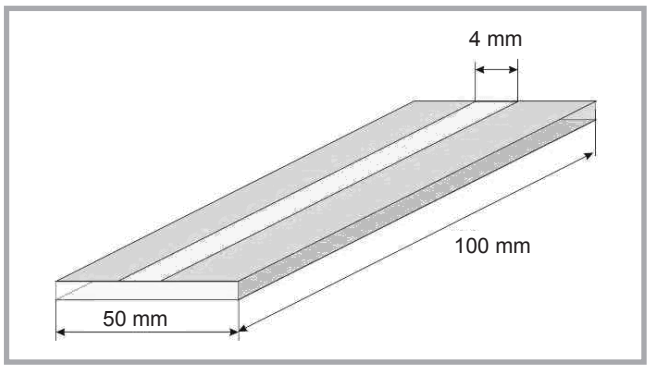


Figure 2. *Transmission line geometry.*

tering reactor, the plasma mode is initiated between the cathode and anode of the magnetron under ca. 10^{-3} Torr pressure [30-32]. The interaction of plasma with the magnetic field modifying the movement of electrons, and the consequent transport of the deposited substance, is very important. Due to the fact that the magnetic field lines run in parallel to the cathode surface, the pathways of the electrons emitted form a closed loop. Sputtering is affected by bombarding the material to be deposited (a disc-shaped target) with working gas ions. As a result of that phenomenon, after initiation of glow discharge, a closed plasma (ionised gas) ring is obtained above the target. Its shape depends on that of the target – e.g. it will be torus-shaped if the target is circular. The positive ions of the working gas are attracted by the negative potential of the target, bombarding the target and striking off atoms from it. The intensity of the sputtering process and the etching area depends on the voltage and thickness of the target, the pressure in the vacuum chamber, as well as on the type and composition of the working gas [27-35].

The method of film deposition by magnetron sputtering has been implemented in the textile industry quite recently. The literature [36-40] described the application of these methods for conferring electroconductive properties to nonwovens. Magnetron sputtering is also used for the deposition of ceramic layers on woven fabrics to reduce heat transmission through the external layer of firemen's clothing and to protect the users from exposure to radiant heat [41]. Besides magnetron sputtering, a method employing low-temperature plasma glow discharge – PAPVD (Plasma Assisted Physical Vapour Deposition) is also used to obtain electroconductive layers on textiles [42, 43].

The aim of the paper is to present the results of research concerning the deposition of electroconductive transmission lines with precisely controlled geometry on the surface of a textile material using the magnetron sputtering method. The transmission lines can be used to effect electrical connections between electronic elements incorporated in garments.

Substrate characteristics

Nonwoven substrates potentially applicable in firemen's clothing were selected for electroconductive transmission line deposition. The substrates selected are characterised in ***Table 1***.

The first two substrates are spun-bonded type polypropylene nonwovens, whereas the latter two ones are needled polyimidamide nonwovens. The KF2 nonwoven was additionally smoothed on one side by the scorching of protruding fibre ends to eliminate the fluffiness effect. In the case of film deposition onto textile substrates, the topography of the substrate surface is of utmost importance for the properties of the newly formed layer [33, 34]. The polyamidoimide nonwovens were fused by pressing with polyolefin foil of 20 g/m^2 surface mass and 24 μm thickness at 120 °C for 120 seconds to obtain a smooth and continuous surface. The polypropylene nonwoven manufactured by Lentex was also subjected to press fusion at 170 °C – four layers of the substrate were fused together for a period of 120 seconds.

Within the framework of substrate characterisation before commencing the deposition of metallic layers, the coarseness of the external layer of the textile substrates was determined using Kawabata Evaluation System (KES) module 4 apparatus (Japan). The sample mean deviation (SMD) of thickness was read, the results of which are summarised in ***Table 2***.

Table 1. *Characteristics of substrates for magnetron sputtering process.*

Code	Product type	Manufacturing method	Material	Surface mass, g/m^2	Thickness, mm	Manufacturer
PP1	Nonwoven	Spun-bonded	Polypropylene	200	1.00	Lentex
PP2	Nonwoven	Spun-bonded	Polypropylene	196	0.70	Wigolen
KF1	Nonwoven/foil	Needling/fusion	Polyamidimide + polyolefin foil	280	1.0	K-48
KF2	Nonwoven/foil	Needling/ scorching/fusion	Polyamidimide + polyolefin foil	450	3.4	Moratex, K-48

Table 2. *Sample mean deviation (SMD) of thickness.*

Substrate type	PP1	PP2	KF1	KF2
SMD Longitudinal direction	1.44	1.82	1.58	1.415
SMD Transverse direction	1.41	4.70	1.01	1.61

Layer deposition

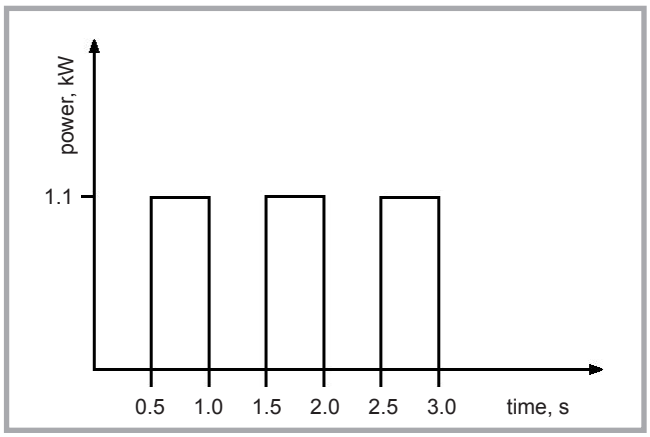
A Classic 500 Pfeiffer Vacuum sputter equipped with an MK-50 magnetron was used to obtain electroconductive layers by magnetron sputtering. A diagram of the device is presented in ***Figure 1***.

Prior to the thin film deposition process, all the samples underwent preliminary cleaning by extraction in ethanol and by using an ultrasound washer for 10 min.

The sample surface was then covered with previously prepared adhesive foil masks with contoured openings, reflecting the course of the electroconductive transmission lines designed. The distance between the substrates and magnetron was approximately 13 cm. The geometry of the transmission lines is presented in ***Figure 2***.

The samples prepared together with the masks were mounted on a power plate in the sputter chamber above the target, i.e. the source of the sputtered metal. Copper of 99.99% purity was selected for deposition onto the textile substrate. The films were deposited under 3.2-3.4 × 10^{-3} Torr vacuum conditions at room temperature. The deposition process was conducted with a variable power supply and constant frequency. The work of the magnetron was cyclic, and it was turned off at 0.5 s intervals. Characteristics of the process are presented in ***Figure 3***.

Figure 3. *Characteristics of magnetron sputtering process.*



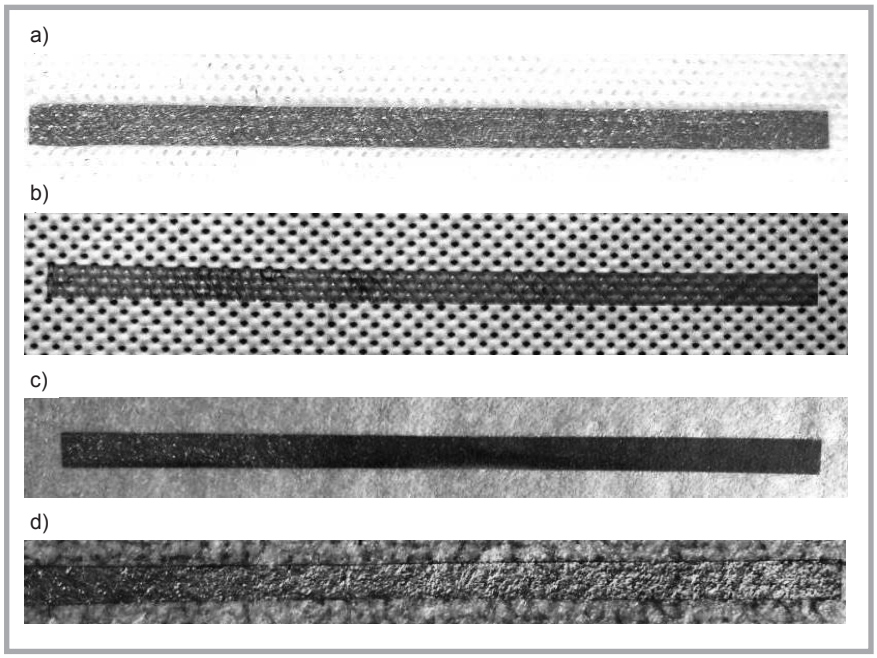


Figure 4. *Photographs of electroconductive transmission lines obtained on substrates: a) PP1, b) PP2, c) KF1 & d) KF2 by magnetron sputtering.*

Testing methodology

Electroconductive properties of the transmission lines obtained were assessed on the basis of electric surface resistivity measurements according to the PN-EN 1149 standard. Samples for the tests were conditioned for 24 hours and tested at 23 °C temperature and 25%. relative air humidity. The tests were performed using a 2-electrode system, an Extech EX570 multimeter, a Rigol DM3052 electrometer and stabilised voltage power supply. A surface resistivity value lower than $1 \cdot 10^7$ Ω, was adopted as the material electrical resistivity threshold according to the PN-92/E-5200 standard. The durability of the deposited layers was assessed by means of two utility tests: resistance to multiple bending and abrasion. The multiple bending test was performed using a DP5/3 type apparatus, in which the samples were bent in 10 cycles at a 120° angle and 100 g load. Resistance to abrasion was tested on a Geiger-Shopper apparatus, in which the samples were abraded in a cycle consisting of 10 rotations. After completion of the utility tests, changes in the electrical resistivity of the electroconductive layers obtained on the samples were assessed.

Values of the surface resistivity parameter were compared for dependent and independent variables. Measurements of the surface resistivity parameter for the same sample variants before and after the bending test were recorded. First the mean values and standard deviations for each of the variants analysed were determined, and then the confidence intervals were established for each deposition time and substrate variant. To check if the specific distributions were normal, an analysis utilising the Shapiro-Wilk test was carried out, assuming the consistency of the variable analysed with the theoretical normal distribution. The zero hypothesis H_0 was adopted presuming that the distribution of the parameter analysed is normal, as well as the alternative hypothesis, assuming that the parameter analysed has a different distribution. At a $\alpha = 0.05$ significance level and for a given sample size n, critical values Wα for the Shapiro-Wilk test were read from the tables. In the case that $W \geq W\alpha$ at the significance level α, there are no grounds to reject the H_0 hypothesis.

Table 3. *Copper layer thickness on the substrates.*

Sputtering time, min	10	30	60	90	180
Layer thickness, µm	0.3	0.64	0.85	1.56	2.2

To verify the significance of differences between variances and mean values for the samples before and after bending tests, two statistical tests were performed: the Fisher-Snedecor (F test) and Student-t test. The F test was conducted to find out whether there were statistically significant differences between variances at a $\alpha = 0.05$ significance level. Critical values vere read from the tables. If $F_{calculated} < F_{tabulated.}$ there are no grounds to reject the zero hypothesis, assuming the equality of variances. Then it was checked by means of S the tudent-t test whether there were significant differences between

Table 4. Surface resistivity results for PP1 nonwoven sample.

Sputtering time, min	After deposition				After bending				Comparison	
	r_S, Ω	s, Ω	Uc,Ω	W	r_S, Ω	s, Ω	Uc, Ω	W	F	t
10	27	7.48	3.74	1.49	34	1.66	1.17	1.52	0.049	1.345
30	7.8	2.15	1.08	1.53	8.87	0.67	0.47	0.87	0.097	0.7045
60	2.9	0.54	0.27	1.41	5.17	0.95	0.67	0.86	3.116	**3.398**
90	0.7	0.06	0.03	1.39	0.73	0.04	0.03	0.98	0.606	0.651
180	0.2	0.02	0.01	5.64	0.24	0.04	0.01	2.00	2.167	0.738

Table 5. Surface resistivity results for PP2 nonwoven sample.

Sputtering time, min	After deposition				After bending				Comparison	
	r_S, Ω	s, Ω	Uc,Ω	W	r_S, Ω	s, Ω	Uc, Ω	W	F	t
10	155.2	4.71	2.36	1.49	320	8.46	5.98	1.52	3.224	**27.765**
30	94	5.84	2.91	1.53	151	24.81	17.54	1.34	18.057	**3.749**
60	30.4	2.73	1.37	1.41	32.73	3.63	2.57	1.36	1.767	0.819
90	13	2.61	1.3	1.39	15.53	2.28	1.61	1.16	0.767	1.133
180	1.8	0.32	0.16	5.64	1.83	0.74	0.52	1.42	5.433	0.068

Table 6. Surface resistivity results for KF1 nonwoven sample.

Sputtering time, min	After deposition				After bending				Comparison	
	r_S, Ω	s, Ω	Uc,Ω	W	r_S, Ω	s, Ω	Uc,Ω	W	F	t
10	136	9.5	5.23	1.00	150.3	24.19	27.34	1.25	6.485	0.891
30	24	3.61	2.25	1.19	30.1	6.13	6.93	2.00	2.889	1.384
60	0.7	0.1	0.24	1.77	2.3	0.32	0.36	1.12	10.264	**7.768**
90	0.6	0.19	0.32	2.07	3.2	0.53	0.6	1.44	7.821	**7.738**
180	0.2	0.06	0.17	2.12	0.3	0.08	0.09	1.19	1.929	0.655

Table 7. Surface resistivity results for KF2 nonwoven sample. ***Note:*** *The critical value for A test$_{(\infty = 0,05,\ c = 0,03,\ V = 2,21)}$ was 1,94.*

Sputtering time, min	After deposition				After bending				Comparison	
	r_S, Ω	s, Ω	Uc,Ω	W	r_S, Ω	s, Ω	Uc,Ω	W	F	t/A
10	206	7.17	4.05	1.69	249	33.04	37.34	1.25	**21.25**	**2.114**
30	75.1	4.08	2.49	2.20	83	10	11.3	2.00	6.007	1.208
60	17.5	2.64	1.75	1.71	20.8	4.04	4.57	1.12	2.349	1.093
90	1.03	0.08	0.21	11.61	1.1	0.09	0.1	1.44	1.075	1.29
180	0.5	0.13	0.26	03.76	0.9	0.1	0.11	1.19	0.566	**3.581**

Table 8. Surface resistivity results obtained according to the methodology presented in ***Figure 5*** *for electroconductive transmission lines coated with a acrylic enamel layer and subjected to abrasion.*

Substrate type	After deposition			After abrasion			Comparison	
	r_S, Ω	s, Ω	Uc, Ω	r_S, Ω	s, Ω	Uc, Ω	F	t
PP1	28.7	1.53	1.08	30.2	0.61	0.44	0.159	0.335
PP2	165.1	8.6	6.08	156.5	1.57	1.11	0.0333	0.062
KF1	133.8	7.89	5.58	138.8	4.62	3.27	0.343	0.061
KF2	206.2	6.15	4.35	213.4	4.43	3.13	0.519	0.074

the mean values at $\alpha = 0.05$ and the number of degrees of freedom equal to 5 (n-1).

The character, continuity and thickness of the deposited layers were observed studying images of surfaces and cross-sections of the substrates under a scanning electron microscope (SEM). The cross-sections were obtained in a liquid nitrogen environment. A JSM-5200LV(JEOL, the Netherlands) scanning microscope was used. The observations were conducted under 5×10^{-3} Pa vacuum conditions using an acceleration voltage of 25 kV.

Results

The samples produced as a result of the magnetic sputtering process are presented in the photos in ***Figure 4***.

The copper layer thickness on the substrates was determined, the results of which are listed in ***Table 3***.

Tables 4-7 present results of the tests: mean surface resistivity values for the copper layers deposited by sputtering r_S, standard deviation for the sample (s) and complex measurement uncertainty Uc, constituting a sum of measurement uncertainty U_A (type A) calculated on the basis of statistical analysis of a series of measurements, and U_B (type B), taking into account the error of the measuring instrument. Additionally values of statistics calculated for the Shapiro-Wilk (W), Fisher-Snedecor (F), Student-t (t) and Aspina-Welcha tests (test A – sample KF2, sputtering 10 minutes) are presented. The results are presented in a separate table for each substrate type.

The critical value for Shapiro-Wilk test was: for samples after film deposition $W_{(0,05;\ 3)} = 0.767$ and for samples after bending $W_{(0,05;\ 2)} = 0.748$, whereas the critical value for F ($_{v1 = 3\ i\ v2 = 2}$) test at $\alpha = 0.05$ amounted to 19.17. The critical value for t test $_{(\infty = 0,05,\ k = 5)}$ was 2.571.

For PP1 substrate, in most cases there are no significant statistical differences between the mean surface resistivity values determined for the samples before and after bending, although for samples obtained with 60 min sputtering time, the differences between the mean r_S values before and after bending reached statistical significance.

For the PP1 substrate, statistically significant differences between the mean copper layer surface resistivity values were observed only in cases of transmission line depositing times of 10 and 30 minutes, respectively.

For the KF1 substrate, statistically significant differences between the mean copper layer surface resistivity values were observed in cases of deposition times of 60 and 90 minutes. For the remaining variants, the differences did not reach statistical significance

For the KF2 substrate, statistically significant differences between the mean copper layer surface resistivity values were observed only in the case of a transmission line depositing time of 180 minutes. All the electroconductive transmission layers obtained by the magnetron sputtering method demonstrated no resistance to abrasion. The abrasion test was performed for all the variants of samples. In view of the above, it was decided to safeguard the transmission lines by appli-

cation of a protective layer onto the sample. Three methods of electroconductive film protection were proposed: fusion of the sample with foil, fusion of the sample with a thin layer of polypropylene nonwoven, and application of a thin coat of acrylic enamel. The first two methods of increasing resistance to abrasion proved ineffective because the surface resistivity value after abrasion of the protected transmission lines exceeded $1 \cdot 10^7$ Ω, whereas the third one allowed to safeguard the electroconductive transmission lines without decreasing electrical conductivity. The results are presented in ***Table 8***. Safeguarding was attempted only for one sputtering time – 10 minutes. ***Figure 5*** presents schematically the way of connecting the multimeter and mounting the sample for testing the resistance to abrasion of the samples coated with the protective layer. The results were analysed analogically to those of the surface resistivity presented in ***Tables 4-7***.

The statistical analysis of samples coated with the protective layer before and after the abrasion test failed to demonstrate statistically significant differences between the mean surface resistivity values obtained for the transmission lines. The result of the experiment indicates that protection of the transmission lines against abrasive forces with acrylic enamel coating is effective.

To visualise the layers obtained, the sample images obtained by scanning electron microscopy (SEM) were analysed, the results of which are illustrated in ***Table 9***.

Conclusions

It can be stated, on the basis of the experiments, that the surface resistivity value for layers deposited on a polyolefin textile substrate by vacuum magnetron sputtering is essentially dependent on the deposition time. A longer film deposition time for all the substrates investigated caused an increase in their thickness and a decrease in surface resistivity values for the transmission lines deposited. At the same time, thicker layers obtained after 180 min on the PP1, PP2 & KF1 substrates proved to be more resistant in the bending tests. For all the variants mentioned, comparative analysis of mean surface resistivities using the Student-t test demonstrated no statistical differences in that physical parameter before and after the bending test. Statistically significant differences between the mean

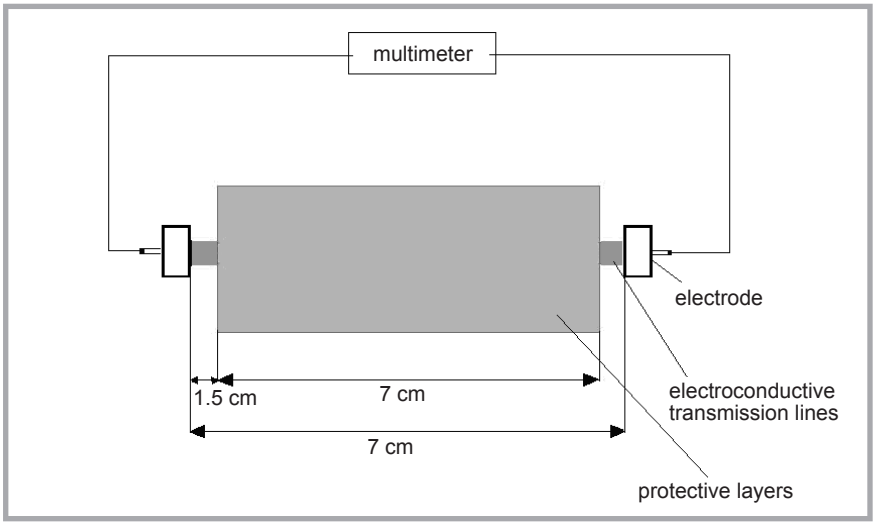
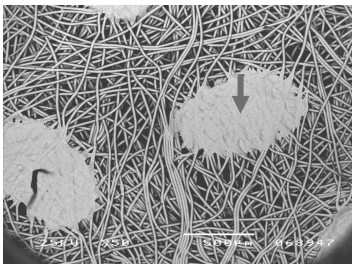
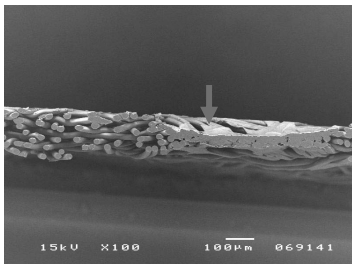
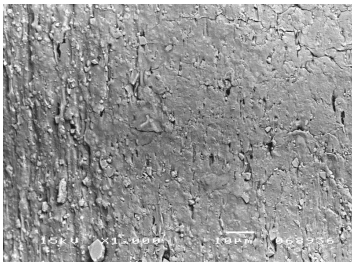
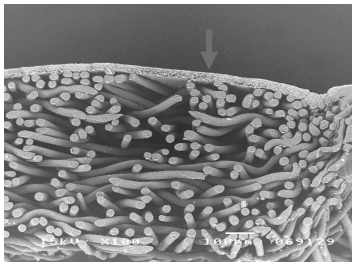
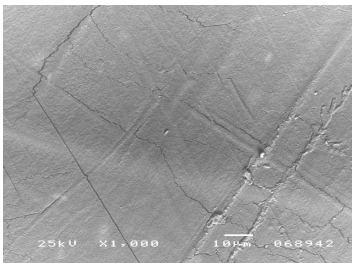
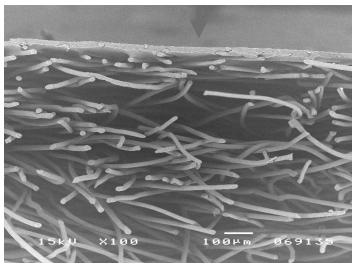
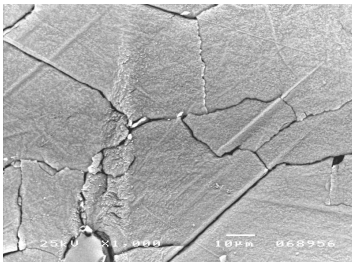
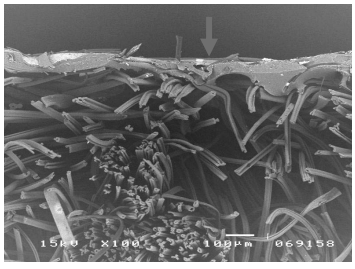


Figure 5. *Connection diagram of a system for resistivity testing before and after the abrasion test.*

Table 9. *Images of samples using SEM.*

Substrate	Longitudinal view	Cross-section
PP1		
PP2		
KF1		
KF2		

surface resistivity values were obtained for the KF2 variant only, in which cracking of the metallic layers was observed. Fusing the textiles with foil in variants KF1 and KF2 allowed to obtain a smooth surface of the polymer substrate; however, there were visible microcracks on it. The occurrence of such was probably the cause of significant differences between the mean surface resistivity values for transmission lines obtained as a result of the longer sputtering time – between 60 and 90 minutes for the KF1 substrate and180 minutes for KF2.

On the PP2 substrate, demonstrating the most coarse surface both in the longitudinal and in transversal directions, metallic layers with the lowest surface resistivity for sputtering times above 10 min, which retained good resistance to the bend tests, were obtained.

None of the variants were resistant to abrasion tests. To safeguard the deposited copper layer and to make it resistant to abrasion, the sample was coated with a layer of acrylic enamel.

With longer sputtering times (exceeding 40 minutes), KF1 and KF2 presented a problem with the foil masks involving the lack of thermal resistance of the masks used (which underwent deformation, thus distorting the transmission line geometry) and required the use of an additional thermoresistant mask applied directly onto the foil one. Too long deposition times led to strong adhesions between the mask and the polyolefin foil layer – when the mask was removed, the metallic lines deposited were partially damaged. Therefore the deposition process was discontinued after 180 minutes. The research has demonstrated that the PVD method using a sputterer equipped with a magnetron makes it possible to obtain electroconductive transmission lines on spun-bonded polypropylene nonwovens without the necessity of fusing them with foil in order to reduce the coarseness of the surface. The transmission lines obtained in this way are characterised by a considerable decrease in surface resistivity values with increased copper sputtering time. Under the optimal sputtering conditions for the PP1 substrate, with 180 min sputtering time, the level of surface resistivity of the deposited copper layer of 2.2 μm thickness was 0,2 Ω. That resistivity value did not change significantly for the sample subjected to 10 cycles of bending at a 120° angle. In order to protect the deposited electroconductive layer against abrasive forces, it should be coated with acrylic enamel.

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