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Using Vacuum Deposition Technology for the Manufacturing of Electro-Conductive Layers on the Surface of Textiles

Abstract
Work associated with obtaining an electroconductive layer on the surface of selected textile structures using vacuum deposition technology is described in the paper. The vacuum deposition method gives the opportunity to receive products if the metal layers obtained have good electrical conductivity with just the adhesion of particles to the textile substrate. The experimental work on the application of metallic layers on fabric samples were divided into three stages. At each stage, the conductivity of the layers obtained was investigated. A microscopic examination was carried out using optical microscopy and scanning electron microscopy in order to evaluate the continuity of the electroconductive layers deposited. Based on the work, it was found that the sputtered layers are characterised by an electrical conductivity depending on the process parameters.

Key words: *electroconductive layers, conductive textiles, vacuum deposition, surface resistance.*

Introduction

The problem of obtaining electrical conductivity in the layers applied to a flat textile structure appears more often in connection with the development of specialised textiles, among others, for the purpose of protecting against the influence of electric and electromagnetic fields, as well as for the development of textronics. This is a rapidly developing area, which is a synergistic combination of electronics, computer science and textiles. It is related to the design of devices in which a textile product can play a different role: a part of it may be an element of an electronic circuit or flexible substrate for integrated active and passive components. The basis for such applications is the production of an electrocon-ductive surface layer on textile products (electroconductive base) with a very low surface resistance, similar to the traditional parameters of metallic conductors.

The preparation of a low-resistance electroconductive layer on a textile structure using vacuum methods is difficult for at least three reasons: Firstly, in the vacuum process the substrate should be flat, whereas in the case of fabric the substrate has a three-dimensional surface structure as a result of warp and weft yarns interlacing. Secondly, the textile structure is a system made complex in material and is geometrical – spatial respect. In the majority of textile yarn it is a porous structure, formed from elementary fibres with specific physical properties and limited length (about a few cm). In the case of multifilament threads, they are formed from individual filaments of the same length as that of the thread. Thirdly, the layer deposited from the outside, seemingly on the surface of the textile product, penetrates, to a lesser or greater extent, into the structure.

The basic methods of obtaining electrical conductivity on the surface of textiles depends on manufacturing fibres made of electroconductive materials and spinning yarns from them [1]. Electroconductive fibres are obtained mainly through the modification of non-electrical-conductive polymers. The modification is associated with the addition of conductive materials (soot, metal powder) to these polymers. There is also the possibility of using metal fibres, with a very low resist-ance per unit length, less than 0.1 Ω/m. A characteristic feature of these fibres is the presence of metals in their structure. The preparation of conductive metal yarns involves the twisting of single metal fibres (steel, copper, silver, gold, among others) in the process of spinning or surface metallisation of non-conductive yarns and continous filaments. Woven and knitted fabrics, nonwovens and other fibrous products, such as multilayered structures and clothing packages are manufactured in traditional textile processes with electroconductive fibres and yarns. Using the electroconductive structures obtained in these ways is quite limited due to the unstable nature of the conductivity resulting from the mutual intertwining threads.

Extremely promising technologies associated with obtaining homogenous conductive layers on the surface of textiles are associated with the modification of the flat surface of textile products through obtaining conductive layers. One such technology is widely used in various application methods of thin films produced by vacuum deposition. It was used in the case of fibrous substrates to obtain layers which are a barrier against electromagnetic radiation [2, 3]. Through the vacuum deposition of materials such as Zn, Ti, Cu, Ag & Al on the surface of textile materials, electromagnetic shields are obtained, characterised by good adhesion to the substrate layers and by a shielding effectiveness (SE) over 35 dB at frequencies ranging from 10 MHz to 1.3 GHz,. Vacuum deposition technology

was also used to obtain a electroconductive layer on the surface of textile materials. By Ag deposition on textiles using the plasma sputtering method, excellent adhesion on polyester fibres could be obtained with smooth coatings [4]. Therefore, electrical conductivity is obtained with low amounts of deposited Ag maintaining their textile properties, while showing a metallic appearance and antimicrobial activity. Copper (Cu) nanocomposite textiles are prepared by magnetron sputter coating [5]. The surface conductivity of textiles coated with Cu nanostructures shows a significant increase compared to uncoated ones. Increased coating thickness leads to better electrical conductivity. Sequential vacuum deposition technology of metals, such as Ti/T Au and Pt on the surface of aramid fibre Kevlar 49 was used to obtain leadwires and intrafascicular microelectrodes [6]. Based on research on the electroconductive and mechanical properties of the metallised fibres obtained, the possibility of using them as intrafascicular electrodes for recording multi-unit neural activity in feline peripheral nerves is confirmed. To obtain homogenous layers on the surface of textile materials, metals such as Ag, Cu, Au, Pt & Pt/Rh were deposited using the technology of vacuum deposition. Next their antibacterial and antifungal properties were tested [7], and it was found that copper was effective against a bacteria bridge and fungi. Silver was also effective against bacteria, but against fungi its effectiveness proved to be limited. The other metals tested did not achieve effectiveness.

The main aim of the research was to determine the possibilities and conditions for the manufacturing of a metallic conductive layer on samples of flat textile products. The investigation was conducted from the viewpoint of the application of the electroconductive fabric obtained as electrodes for muscle electrostimulation.

■ Materials

Four fabrics were chosen for surface modification. Selected parameters of the textiles used are collected in **Table 1**.

A select group of fabrics allows the preliminary assessment of the impact of media on the possibility of obtaining of a conductive metallic layer by the vacuum sputtering method.

Table 1. Parameters of woven fabrics.

Woven fabric symbol	Raw material	Weave type	Thick-ness, mm	Areal surface g/m²	Warp density, yarns/cm	Weft density, yarns/cm	Yarn mass, tex		Number of twists, twists/m	
							Warp	Weft	Warp	Weft
M3	Cotton	Twill	0,36	185	38	35	29,3	20,3	824	780
M19	Cotton	Twill	0,33	207	45	33	19,4	35,1	976	686
TT4	Polyester	Plain	0,35	161	25	22	32,8	31,7	344	124
TN	Polyester + Cotton	Twill	0,41	170	43	24	25,1	25,7	800	1160

■ Methods

Manufacture of electroconductive layers on textiles

The research involved the production of metallic layers on selected samples of textile products in varying process conditions and microscopic analysis of the layers obtained using optical microscopy and scanning electron microscopy. Metal layers were applied using the Classic 250 Pfeiffer Vacuum system. The resistance vapour source (tungsten boats) with evaporator current control was used. The deposition process was carried out after obtaining a vacuum of 0.01 Pa (1.0×10⁻⁴ mbar. The pressure in the chamber changed to a value of 0.1 Pa (1.0×10⁻³ mbar) during the deposition. The application of the layer lasted 5 minutes, and the samples were conditioned for 3 hours in an environment with a temperature of 22 ± 0.5 °C and relative humidity of 55 ± 5% prior to layer application. Metals with high purity: Ag - and Au 99.99% - 99.99% were used in the process of layer application.

The experimental research on the application of metallic layers on samples of selected fabrics was divided into three stages:

I - initial tests on the possibility of obtaining the surface electrical conductivity of metallic layers sputtered on the fabric;

II - deposition and research on the conductive layers on fabrics selected at stage I;

III - preliminary optimisation of the process – deposition of layers under conditions specified on the basis of the results obtained in stage II.

A preliminary microscopic examination was performed using the following optical microscopes: a stereomicroscope with a magnification of 16× to 100× and a metallographic microscope - Neophot 21 with a magnification of 100× to 800×. The scanning electron microscope Hitachi S-4200 with the Voyager 3500 microanalysis system was also used in the research on metallic layers. SEM ob-

servations were carried out at magnifications from 30× to 2000×.

Measurement of the electrical conductivity of layers produced on textiles

The estimation of the electrical conductivity of metallic layers obtained on the fibre substrate was based on resistance measurements of the samples made. Two methods of resistance measurement were used: The first one was developed on the basis of the idea of two-point resistance measurement, the second - four-point.

The two-point method developed is based on the direct measurement of local resistance on the surface of the sample using a two-point probe connected to a multimeter - Agilent 34410A, working as an ohmmeter [8, 9]. Both electrodes of the probe, with a diameter of 8 mm each, are 10 mm from each other. The probe is placed on the sample, divided into segments, and the resistance is measured in all pairs of segments. In this way, the values of local resistance on each textile sputter sample are obtained, enabling to draw up a local resistance map according to the measurement results obtained using the test method presented.

The four-point method developed is based on indirect measurement of the resistance of the samples using electrodes, which are instructed in four corners of the sample and marked, respectively, as measuring electrode 1, 2, 3 and 4 [10, 11]. This method is based on the following measurement model:

$$R = \frac{1}{2}\left(\frac{U_{1-3}}{I_{2-4}} + \frac{U_{2-4}}{I_{1-3}}\right), \qquad (1)$$

where:

I_{1-3}, I_{2-4} – forced current between opposite measuring electrodes, i.e. 1 and 3 or 2 and 4, respectively;

U_{1-3}, U_{2-4} – measured voltage drop between the second pair of opposing electrodes, i.e. 1 and 3 or 2 and 4, respectively.
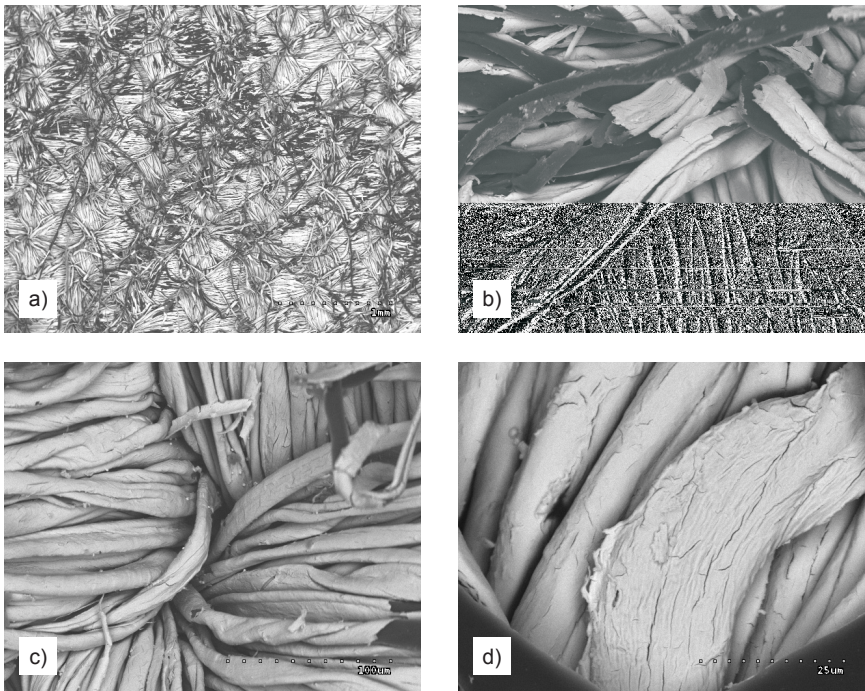
Figure 1. *SEM images of the silver layer on a cotton fabric - M3 in the first stage of research; a - 35× magnification, b, c - 180× magnification, d - 500× magnification.*
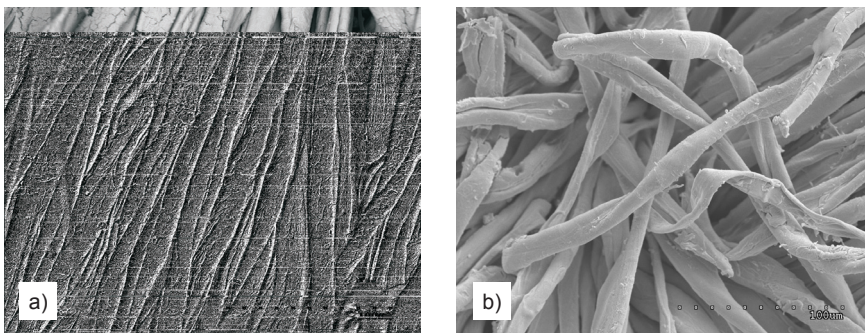

Figure 2. *SEM images of the silver layer obtained in the second stage of research; a – on fabric M19, b - on fabric M3.*
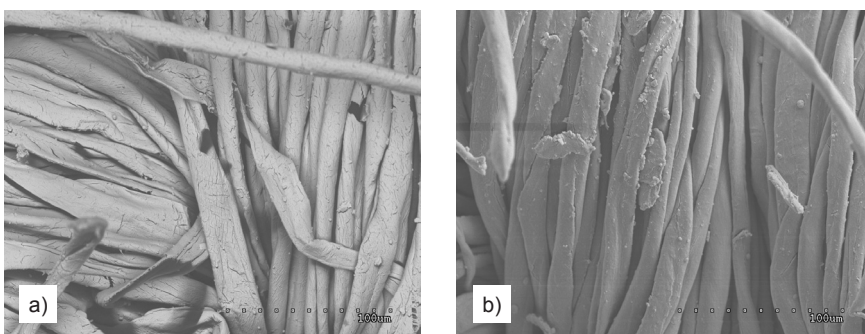

Figure 3. *SEM images of the metallisation layer on fabric M19 obtained at the third stage of research; a - gold layer, 500× magnification, b - silver layer deposited on the gold layer, magnification 500×.*

Table 2. *Measurement results of the average surface resistance.*

Variant	Temperature, °C	Relative humidity, %	Average surface resistance, Ω	Coefficient of variation, %
I	31.7	25	112.4	2.5
II	32.3	35	101.4	2.1
III	35.4	60	71.3	4.5

This method allows to determine changes in the sample resistance with relatively small dimensions of the surface. In particular, it allows the assessment of the impact of test conditions on the resistance measurements.

■ Results and discussion

Influence of vacuum process parameters on electroconductive properties of the layers

Metal layers were applied on selected flat textile products in a multistage process in order to give electroconductive properties. Fabric samples with dimensions of 60 × 60 mm were prepared and placed casually on special substrates in a vacuum chamber. In the first step a layer of silver was applied. Initially the ability of the current conduction in the layers obtained was tested and their microscopic examination carried out.

In the first stage, the silver layers deposited on the textile substrate did not generally show continuous electrical conductivity. In the case of textiles TN and TT4, a complete lack of conductivity was found. Neither the low adhesion of the layer to the fibre nor the low volume concentration of fibres created even the possibility of the appearance of percolation-type conduction.

In the case of fabric M3 , due to the fact that the measurements were performed on different parts of the surface, in some samples sporadic and unstable current conduction was revealed. Sample M3 was subjected to SEM observation (*Figure 1*). The silver layer covers the apparent ("observed" from the vapour source) surface of the fabric, but it is not sustainable and can be easily destroyed, for example as a result of contact with current and voltage electrodes (*Figures 1.a* and *1.b*). The individual fibres are coated with a layer of numerous longitudinal and transverse cracks (*Figures 1.c* and *1.d*). Sporadic electrical conductivity occurred due to the formation of conductive paths created by contact between the individual weaves of fibres coated with a silver layer.

A similar situation occurred in the case of fabric M19. Therefore fabrics M3 and M19 were selected for stage II. At this stage, the layer of silver was re-applied with variable setting parameters and sample preparation. Observations were

carried out with optical microscopes. The electrical conductivity of the metallisation layers were investigated, depending on process conditions (type of fabric, sample preparation and sample location in the chamber).

Resistance measurements of the layers of the samples tested confirmed the decisively positive impact of the assumed treatments of the layers applied, as observed in the second stage. These observations are essentially confirmed by SEM microscopic image analysis (*Figure 2*). A definitely better quality of the metallisation layer on fabrics M3 and M19 was obtained, including a lack of discontinuities. Changes in the process conditions improved the conductivity of the layers made on these fabrics.

In stage III, layers of gold and silver were applied on fabric samples M3 and M19. Good conductivity of the layers applied was achieved for both fabrics. In stage III, the best conductivity was obtained for fabric M3. Fabric M19 has good, but slightly lower electrical conductivity. Observations using scanning electron microscopy were carried out. Images of sample M19 obtained at stage II (*Figure 2.a*) and stage III (*Figure 3*) are compared.

Improvement in the adhesion of these layers to the substrate fibre was observed as a result of the deposition of metallic layers at stage III.

Assessment of the electrical conductivity of the metallic layers obtained

Fabric M19 was selected for estimation of the conductivity of the electroconductive layers obtained. Research of the temperature and humidity effects on the properties of the electroconductive layers applied on fabric was conducted in the first part of the work. These investigations were carried out in an Angelatoni UD150C Industrie climatic chamber. Changes in the temperature and humidity were possible. Three variants of the temperature and relative humidity were selected, chosen on the basis of medical literature in order to simulate human physiological conditions [12]. The values for each variant are collected in *Table 2.*

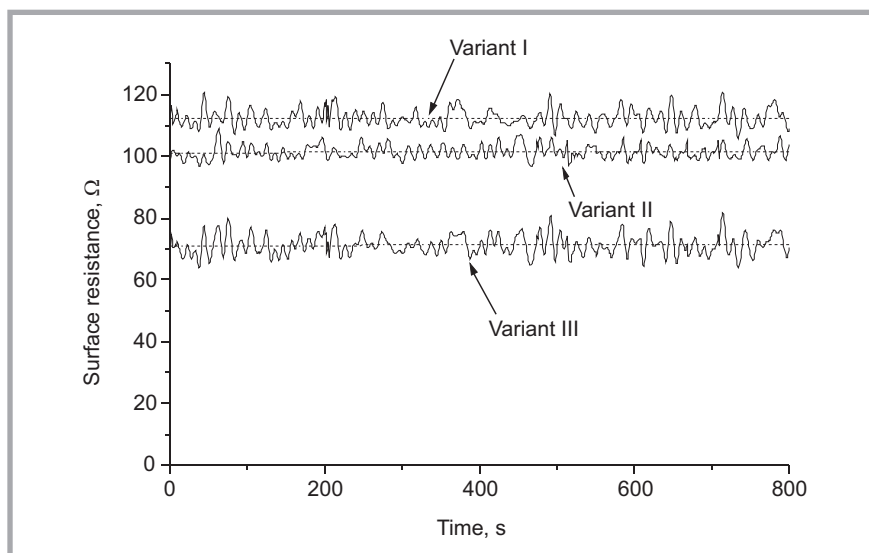Resistance measurements were carried out by the four-point method using Keithley's multimeter. The wires were tight-

Figure 4. *Changes in the electroconductive layer surface resistance for particular variants of the research.*

ened with electroconductive glue at the four corners of the conductive surface, which was a square with dimensions of 6 × 6 cm. Then the wires were connected to the measuring instrument. The resistance was determined according to model (1). Results of the measurements are shown in *Figure 4*, carried out over periods of 800 s , which is comparable to the time of a single electrical stimulation treatment. The surface resistance fluctuations shown in *Figure 4* are the result of electromagnetic disturbances generated by climatic chamber devices. The average resistance values and standard deviations calculated from 800 samples for different variants are shown in *Table 2*.

The surface resistance value should not exceed 300 Ω in the case of electrodes used in electrostimulation. This value is recommended by manufacturers of medical generators used for electrostimulation. It is experimentally found that the higher resistance values can cause local patient burns during electrotherapy. All the average values of surface resistance in the individual variants are less than 300 Ω. A significant decrease in the average surface resistance can be observed in the third stage of research, due to a much higher humidity of 60% compared to the one adopted at stages I and II .

The surface resistance stability over time for particular options was analysed on the basis of the measurement results presented in *Figure 4*. For this purpose, linear regression analysis was used. The model in the form $R = R_0 + \beta t$ was verified, where R_0 is the surface resistance

at t = 0, and β is the coefficient of the function code. The results of the analysis were obtained using STATISTICA and are presented in *Table 3*. A level of significance equal to 0.05 was accepted during the statistical inference about the relevance of the model coefficients.

In each of the three options, it was found that the intercept is an important factor of the model verified, showing the stability of the surface resistance in cases I, II and III. The results of the analysis allow to conclude that the electrically conductive surface obtained on the textile substrate by vacuum deposition can be used as an electrode in the process of electrostimulation.

The distribution of surface resistance was analysed in the second part of the research. In fact, local changes in the resistance may be greater than the limit value of 300 Ω. For this reason, a sample of fabric with an electrical conductive surface was divided into segments. Then resistance measurements were performed using the two-point method at an ambient temperature of 24 °C and relative humidity of 35%. Based on the results,

Table 3. *Results of regression for model* $R=R_0+\beta t$

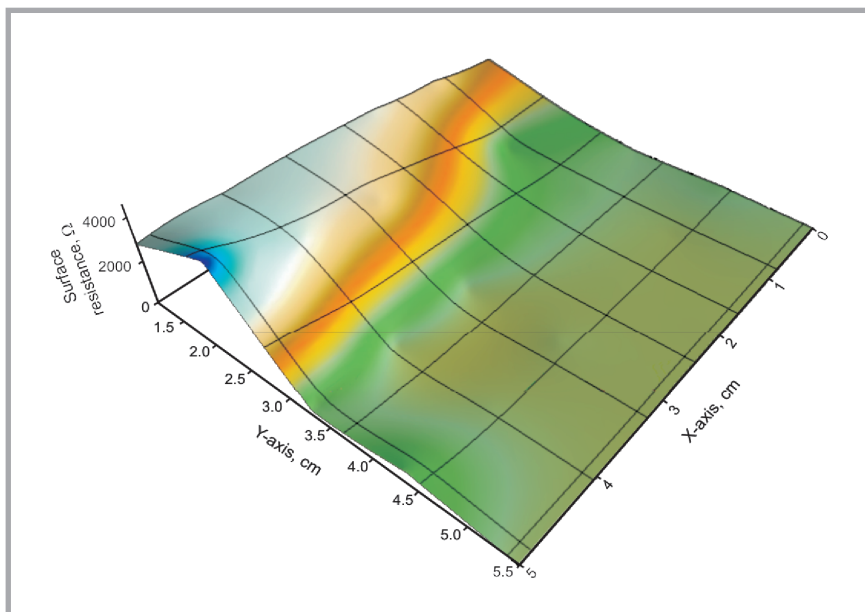| Variant	Regression coefficients		Decision
I	R_0	112.33	important
	β	0.0002	negligible
II	R_0	101.15	important
	β	0.0003	negligible
III	R_0	71.01	important
	β	0.0007	negligible

Figure 5. *Map of the local distribution of surface resistance.*

a map of the distribution of local resistance changes on the sample surface was drawn (***Figure 5***).

The analysis of the distribution of local resistance shows that these values vary from 19.6 Ω to 4152.2 Ω. Therefore, the sample obtained may not be used as an electrode in the process of electrostimulation. Local changes in resistance on the sample surface are the result of the parameters that were accepted during the sputtering process. The alignment of the sample in the evaporator chamber has a significant impact.

■ Conclusions

The use of vacuum deposition technology makes it possible to produce flexible and lightweight electroconductive textiles.

In this study, the experimental work carried out to produce metallic layers on textile substrates in varying conditions of the deposition showed the impact of the substrate on the quality of the metallic layers obtained. An iterative process for the preparation of layers has a beneficial effect on the electrical conductivity of sputtered layers. The best results were obtained in the third stage of research for fabrics M3 and M19 . In stage III, an improvement in the adhesion of the metallic layers to the fibre substrate was observed.

The sputtered conductive layer is characterised by a constant surface resistance, measured by the four-point method, for different variants of temperature and humidity. A significant decrease in resistance was observed in the event of a significant increase in humidity for option III.

Researches on local resistance show that the distribution of the resistance of the electrical surface can be irregular. For applications of such structures as electrodes for electrostimulation, a map of the distribution of local resistance should be analysed by either the two-point method of resistance measurement or by the four-point one. There is a possibility of the existence of local regions with a higher resistance value than the acceptable value of 300 Ω. Local changes in resistance on the sample surface result from the parameters that were accepted during the sputtering process. Therefore, further research will be continued to achieve optimal sputtering process parameters from the viewpoint of textile applications. The resulting product should have good conductivity and uniform distribution of the electrical resistance of the metallic layer

References

1. Taya M. *Electronic composites, Modeling Characterization Processing, and MEMS application*, Cambridge University Press, Cambridge 2005.
2. Ziaja J, Koprowska J, Janukiewicz J. Using Plasma Metallisation for Manufacture of Textile Screens Agains Electromagnetic Fields, *Fibers & Textiles in Eastern Europe* 2008; 16, 5(70): 64-66.
3. Kan Lai, Run-Jun Sun, Mei-Yu Chen, Hui Wu and An-Xia Zha. Electromagnetic Shielding Effectiveness of Fabrics with Metallized Polyester Filaments. *Textile Research Journal* 2007; 77: 242.
4. Hegemann D, Amberg M, Ritter A, and Heuberger M. Recent developments in Ag metallised textiles using plasma sputtering, Materials Technology. *Advanced Performance Materials* March 2009; 24, 1: 41-45(5).
5. Wei Q., Yu L., Wu N., Hong S., Preparation and characterization of copper nanocomposite textiles, *Journal of Industrial Textiles* 2008; 37: 275.
6. McNaughton TG, Horch KW. Metallized polymer fibres as lead-wires and intrafascicular microelectrodes, *Journal of Neuroscience Methods* 1996; 70: 103-110.
7. Scholz J, Nocke G, Hollstein F and Weissbach A. Investigations on fabrics coated with precious metals using the magnetron sputter technique with regard to their anti-microbial properties. *Surface & Coatings Technology* 2005; 192: 252-256.
8. Tokarska M, Frydrysiak M, Zięba J and Tęsiorowski Ł. Określenie właściwości elektroprzewodzących tekstylnych elektrod w oparciu o wybrane metody pomiaru rezystancji, materiały In: VIII *Konferencji Naukowo-Technicznej PPM,* 2011, Prace Komisji Metrologii Oddziału PAN w Katowicach, Seria: Konferencje Nr 15, Krynica Zdrój, pp. 194-198 (in Polish).
9. Zięba J, Frydrysiak M, Tęsiorowski Ł, Tokarska M, Gniotek K., Stempień Z., Innovative textile electrodes for muscles electrostimulation, Proceedings of 6th IEEE International Symposium on Medical Measurements and Applications MeMeA, Bari, Italy, May 30-31, 2011.
10. Zięba J, Frydrysiak M, Tokarska M. Research of textile electrodes to electrotheraphy, *Fibers & Textiles in Eastern Europe* 2011; 19, 5(88): 70-74.
11. Stempień Z, Gniotek K, Zięba J, Tokarska M, Frydrysiak M and Tęsiorowski Ł. Textile-based printed electrodes for muscles electrostimulation, Proceedings of The Fiber Society Spring 2011 Conference, May 23-25, 2011, Hong Kong, China, pp. 14-15.
12. Traczyk W., *Human physiology* (in Polish), PZWL, Warsaw 1992.