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SPUTTERING METHOD FOR CONDUCTIVE TEXTILES

DONDEA Felicia-Maria[1], GROSU, Marian-Cătălin[2], VISILEANU Emilia[3], VLADU Alina-Florentina[4] , SCARLAT Răzvan-Victor[5]

[1-5] The National Research & Development Institute for Textiles and Leather, 030508, Bucharest, office@incdtp.ro

Corresponding author: Grosu Marian-Cătălin, E-mail: catalin.grosu@incdtp.ro

Abstract: *PVD Sputtering is a very popular, and versatile coating method to create multi-functionalities on various kinds of substrates including textile surfaces. The paper presents the obtaining of the two conductive textile structures via the hybrid PVD by DC sputtering method with copper target and analysis of them from the structural and electrical properties point of view. The two textile substrates are woven, made of 100% cotton yarns, with a specific mass of 207 g/m^2 that was preliminary prepared by chemical finishing (padding) with two different substances: ITOBINDER-Acrilat (TA1) si PERMUTEX-EX-RU-Urethan (TA2). After the sputtering process a Cu thin layer with a specific mass of about 5 mg/cm^{2}were obtained. The SEM analyses revealed a fibrous structure, on the surface of which a thin coating has been deposited. It is found that the coating has a columnar appearance and is uniform and continuous on the surface of the sample. The electric measurements were made with BX PRECISION 889B Bench LRC/ESR METER. on 1 inch and 10 cm show that the electrical conductivity values are up to 83% higher for the textile samples preliminary finished with TA1 than those with TA2 and decrease with increasing distance between electrodes.*

Keywords: *Hybrid PVD, DC magnetron sputtering, woven substrate, conductive, copper layer*

1. INTRODUCTION

New generations of functionalized textiles will serve new types of performance requirements in military, biomedical/healthcare, industrial, environmental protection, etc., under sustainable conditions [1]. Advances in the areas of manufacturing and increasing the service life of structural elements and tools applicable to many areas of life are made possible by the frequent use of coating techniques. The extensive selection of currently available coating types and their application technology are the result of the increased demand in the field of innovative methods for functionalization and surface protection of materials, including textiles, in recent years [2].

As a physical vapor deposition (PVD) method, sputtering deposition is a dominant technique to grow thin films onto various kinds of substrates that enhance specific properties like scratch resistance, conductivity, and durability [3]. 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. Magnetron Sputtering is a remarkable environment-friendly process that can be used to prepare various kinds of materials: conductive semiconductive and insulator materials [4]. More, this technique has many advantages such as simple and low-cost handling and control of the equipment, large and high-density coating area with high-purity thin film, strong adhesion onto the surface, low operation temperature, etc. The sputtering deposition technique, although not new, can also be used for the deposition of conductive particles on textile surfaces. Multifunctional textiles can be fabricated by deposition of metal, which induces multifunction simultaneously (e.g. antibacterial, hydrophobic, and conductive flexible surface) [5]. Sputtered thin films have excellent uniformity, density, and adhesion making them ideal for multiple applications (UV protection, antistatic, self-cleaning, RF interference shielding materials for industrial, space, military, and medical fields, etc.) [6]. The textile substrates for sputtering can contain both natural and synthetic fibers such as cotton, silk, nylon, and polyester. The quality/uniformity/morphology of the sputtered coating is highly influenced by the structure parameters of the textile substrate (weave pattern, yarn thickness, and density, obtaining technology of the yarn (e.g. carded, combed), degree of compactness of the yarns, free fibers content, etc.) [7]. Moreover, the sputtering technology and structure of the textile substrate and preliminary treatments/finishings of the textile substrate influence the electroconductive properties of the textile.

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

2. METHODOLOGY

2.1 Materials

For experimental, 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/m^2 has been prepared by chemical finishing (padding) with tho different substances: ITOBINDER-Acrilat si PERMUTEX-EX-RU-Uretan. Previous research has revealed the tendency of metal ions to penetrate between the fibers of the textile substrate so that their deposition does not form a continuous film. So finishing with the two chemical auxiliaries results in the deposition of metal ions in a film with continuity in terms of electrical properties.

The 2 samples of 100 x 100 mm each, cut from the finished textile structures have been used as substrates in the process of DC sputtering. The textile fabrics used as substrate were fixed to the support of the deposition installation. Thin film deposition was performed on a hybrid PVD (Physical Vapor Deposition) system (fig. 2.9) 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 300W. A commercial Cu target (99.999%) with a diameter of 2 inches and a thickness of 3 mm was used for deposition, was mounted on the magnetron sputtering cathode.

3. EXPERIMENTAL

The magnetron sputtering deposition technology flow for Cu coating on textile substrate comprises the following steps:

1. ***Substrate preparation step (a.*** cleaning and degreasing of the textile surface; ***b.*** loading the substrate and deposition materials into the vacuum chamber.)
2. ***Setting evaporation parameters for the copper target c.*** vacuuming of the enclosure (high vacuum up to 10^{-7} Torr); ***d.*** introduction of Argon (Ar) into the enclosure to a partial vacuum of 10^{-3} Torr; ***e.*** ignition of the plasma and sputtering of the Cu target; ***f.*** deposition itself.

The experimental matrix is shown in Table 1.

Table 1: *Experimental matrix*

Charge no.	*Substrate*	*Substrate dimensions*	*Material deposited*	*Operating parameters*
1	Impregnated textile material (ITOBINDER-AG-Acrylate) – Textile auxiliary 1 – TA1	100 x 100 mm	2-inch Cu target of 99.999% purity	Starting vacuum: 9*10 e^{-6} mbar working vacuum: 1.9*10^{e-3} mbar Flow Ar: 50 ml/min Power source: DC: 100 W Deposition time: 120 min
2	Impregnated textile material (PERMUTEX-EX-RU-Urethane) - Textile auxiliary 2 – TA2			

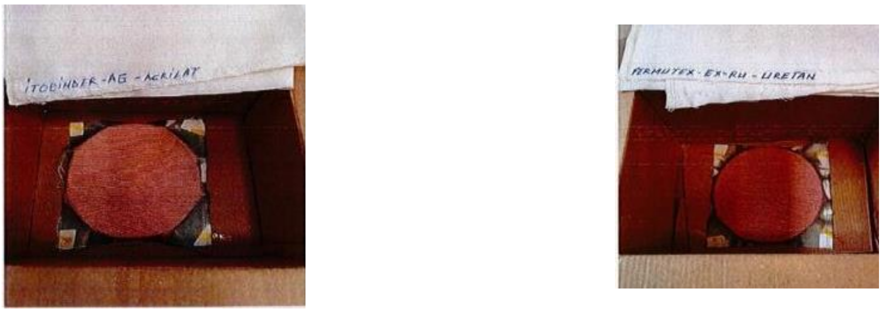


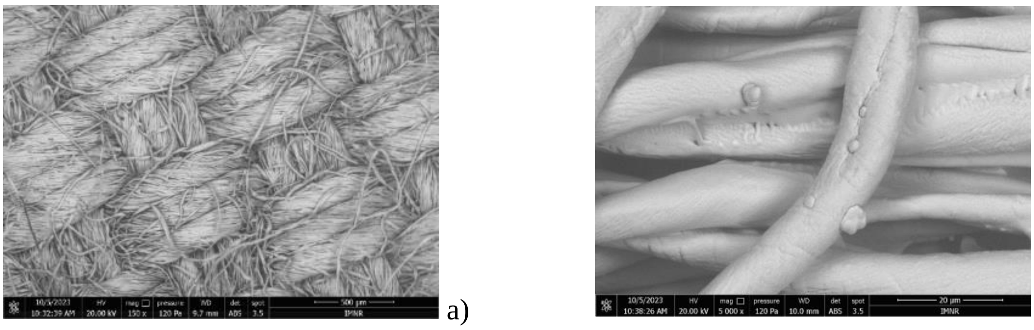
Fig. 1*: Textile samples with Cu deposition by DC/RF Magnetron Sputtering*
Impregnated with ITOBINDER-AG-Acrylate *Impregnated with PERMUTEX-EX-RU-Urethane*

The specific mass of the layer deposited on the samples is about 5 µg/cm^2

4. RESULTS AND DISCUSSIONS

4.1 Scanning Electron Microscopy – SEM

The sputtered textile samples were analyzed on the surface and in cross-section by scanning electron microscopy in Low Vacuum mode, using the secondary electron backscatter detector (ABS) and the energy dispersive spectroscopy (EDS) detector.



a) b)

Fig. 2: *SEM with the surface of the analyzed sample (a. Overview- this image was analyzed at 150X magnification and 500 µm scale., b. Detail- this image was analyzed at 5000X magnification and 20 µm scale.)*

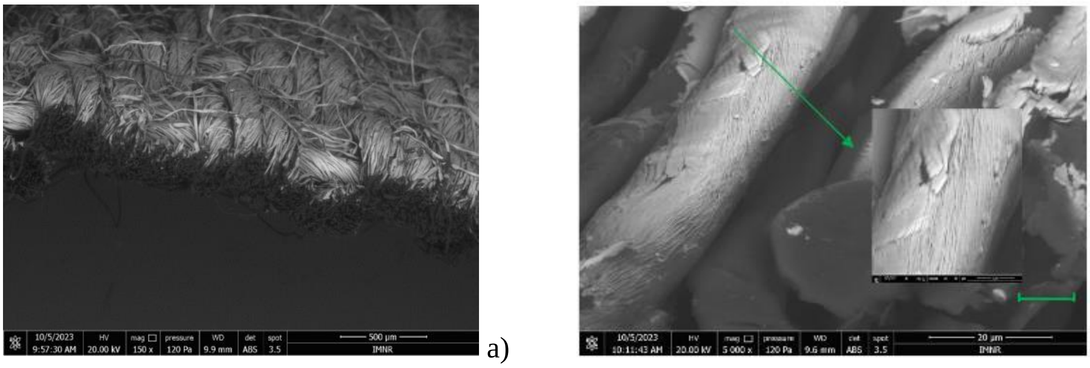


Fig. 3: *SEM with a cross-section of the analyzed sample (a. Overview- this image was analyzed at 150X magnification and 500 µm scale., b. Detail- this image was analyzed at 5000X magnification and 20 µm scale)*

The study of the samples revealed a fibrous structure, on the surface of which a thin coating has been deposited (Fig. 2a, b and 3a, b) It is found that the coating has a columnar appearance and is uniform and continuous on the surface of the sample. Semi-quantitative EDS chemical spot analysis revealed the presence of the main element of interest (Cu) (fig. 4)

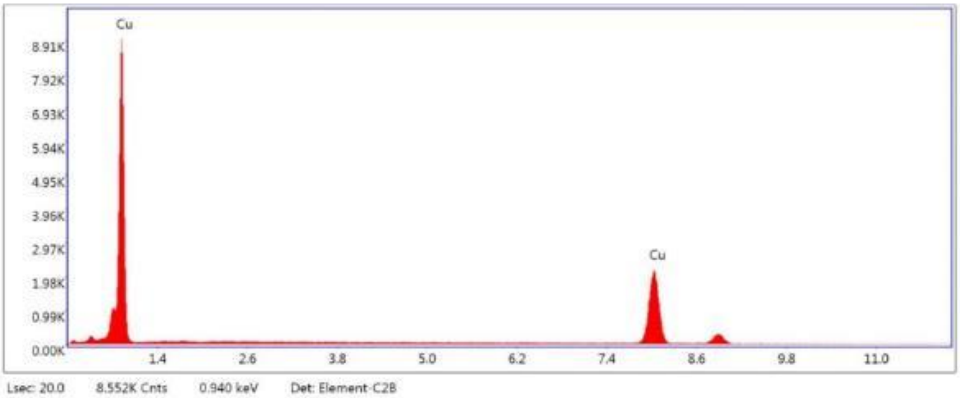


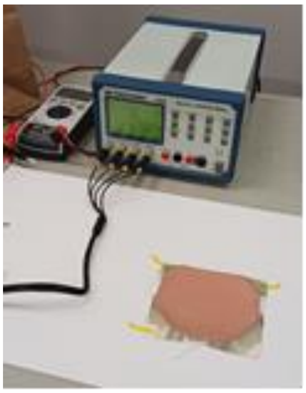
Fig. 4. *ESD analysis with identification of the main target element Cu*

4.2. Electrical characteristics of the sputtered textile structures

The electrical characteristics of textile structures obtained by the sputtering method were determined using the BX PRECISION 889B Bench LRC/ESR METER. The 2 measuring electrodes of the device were placed at a distance of 1 Inch and 10 cm. The calculation formula for electrical conductivity is expressed by relation 1.

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

Where:
D - Electrode width (0.01 m); distance between electrodes (L1 - 0.025 m and L2 - 0.1 m);
g -thickness of textile material (0.62 mm)


a)


b)

Fig. 5: *Linear electrical resistance measurement*

Table 2 presents the values obtained for the electrical resistivity of structures treated with TA1 and TA2 coated with Cu by the sputtering method (Fig. 5a. measurement equipment, Fig. 5b. actual measurement).

Table 2: *Coated fabrics properties*

values	***ITOBINDER AG-Acrilate***		***PERMUTEX***	
	1 INCH	10 CM	1 INCH	10 CM
Average, OHM	0.12	0.19	0.66	2.05
Thickness, mm	0.64	0.64	0.7	0.7
Thickness, m	0.00064	0.00064	0.0007	0.0007
Resistivity, OHM*m	7.616E-05	3.12E-05	0.000464	0.000365
Conductivity, S/m	13130.25	32039.45	2154.71	2742.07

The average values of resistivity (ohm/m) and electrical conductivity (siemens/m) show that there is continuity over the entire textile-coated surface, with higher values for textile samples pretreated with TA1. It is thus found that treatment with this chemical auxiliary causes a decrease in the permeability and porosity of the textile surface, higher than in the case of using TA2, with a more pronounced effect on the uniformity of the Cu layer deposited by the sputtering method.
The electrical conductivity values are thus up to 83% higher for the textile samples preliminary finished with TA1 (32039 S/m) than those with TA2 (2742.07 S/m) and decrease with increasing distance between electrodes.

5. CONCLUSIONS

Thin film deposition was performed on a hybrid PVD (Physical Vapor Deposition) - DC/RF Magnetron Sputtering module, using a commercial Cu target (99.999%). The Cu target was mounted on the magnetron sputtering cathode. SEM analyses revealed the surface morphology of textile structures on whose surface a thin coating of 5μg/cm^2 was deposited. The coatings have a columnar appearance and are uniform and continuous on their surface. Determination of the electrical characteristics of the textile structures reveals that the best values of electrical conductivity were obtained for textile structures pre-treated with ITOBINDER AG-Acrylate. The values obtained are almost the same as those achieved in the first phase of the project, in which the tubular knitted structures with conductive textile yarn content recorded maximum values of ~ 32.808 S/m - a tubular structure made of 100% AgSIS Lib40.

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REFERENCES

[1] M. Asad, N. Saba, A. M. Asiri, M. Jawaid, E. Indarti, W.D. Wanrosli, "*Preparation and characterization of nanocomposite films from oil palm pulp nanocellulose/poly (Vinyl alcohol) by casting method"*, Carbohydrate Polymers, vol. 191, 2018, pp 103-111,

[2] D. Depla, S. Mahieu, J. E. Greene,. "*Chapter 5 - Sputter Deposition Processes*". In: Handbook of Deposition Technologies for Films and Coatings (Third Edition). P. M. Martin (ed.) William Andrew Publishing, Boston, 2010.

[3] J. Stoner, J. (2022, 04 20). What is Magnetron Sputtering and How Does it Work? Retrieved from Korvus Technology: https://korvustech.com/magnetron-sputtering/#behrisch

[4] F. Shi, (2018). Introductory Chapter: Basic Theory of Magnetron Sputtering. IntechOpen. doi: 10.5772/intechopen.80550

[5] A. Kramar, V. Prysiazhnyi, B. Dojčinović, K. Mihajlovski, B.M. Obradović, M.M. Kuraica, M. Kostić, "*Antimicrobial viscose fabric prepared by treatment in DBD and subsequent deposition of silver and copper ions—Investigation of plasma aging effect*", Surface and Coatings Technology, vol. 234, 2013, pp. 92-99, https://doi.org/10.1016/j.surfcoat.2013.03.030.

[6] K. Vinisha Rani, B. Sarma, A. Sarma, "*Plasma sputtering process of copper on polyester/silk blended fabrics for preparation of multifunctional properties*", Vacuum, vol 146, 2017 pp. 206-215, https://doi.org/10.1016/j.vacuum.2017.09.036.

[7] B. Mahltig, D. Darko, K. Günther, H. Haase "*Copper Containing Coatings for Metallized Textile Fabrics*", J Fashion Technol Textile Eng, vol 3:1. 2015, doi:10.4172/2329-9568.1000118

[8] J. Scholz, G. Nocke, F. Hollstein, A. Weissbach, "*Investigations on fabrics coated with precious metals using the magnetron sputter technique with regard to their anti-microbial properties*". Surface and Coatings Technology, 2005, 192, pp 252-256.

[9] E. Visileanu, R. Radulescu, M. C. Grosu, A. Salistean. "*Electrical parameters of conductive structures for smart textiles*". In: Waldemar Karwowski and Tareq Ahram (eds) Artificial Intelligence, Social Computing and Wearable Technologies. AHFE (2023) International Conference. AHFE Open Access, vol 113. AHFE International, USA. http://doi.org/10.54941/ahfe1004207

[10] M. C. Grosu, R. M. Aileni, T. Sarbu, "*Electrical Resistivity Distribution Analysis for Textile Structures Based on Copper Yarns*", International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM, 2023, vol: 23, Issue: 4.1, Page: 51-57, 10.5593/sgem2023/4.1/s17.07

[11] E. Visileanu, C. Mihai, I.R. Rădulescu, C. Grosu, and R. Scarlat, "*Conductive Textiles Obtained By Unconventional Methods*", TexTeh XI International Conference 2023, Bucharest, Romania, SCIENDO, p. 11-16, DOI: 10.2478/9788367405386-001