Research regarding the cover factor of magnetron sputtering plasma coated fabrics

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LILIOARA SURDU
EMILIA VISILEANU
ALINA ARDELEANU
CORNELIA MITRAN
ION RĂZVAN RĂDULESCU
CRISTIAN STANCU
IRINA SANDULACHE
BOGDANA MITU

REZUMAT – ABSTRACT

Cercetari privind factorul de acoperire al țesăturilor pulverizare în plasma tip magnetron

Domeniul textilelor tehnice Buildtech necesită noi tehnologii pentru fabricarea materialelor flexibile ecologice. Pulverizarea prin magnetron permite acoperirea la scară nanometrică a țesăturilor cu diferite funcționalități și beneficii. Funcțiile noi ale țăsăturilor constau în ecranarea electromagnetică și ignifugare. Studiul urmărește să analizeze factorul de acoperire al țesăturilor prin pulverizare în plasmă și a țesăturilor crude, destinate funcționalităților menționate mai sus. Țesăturile cu compoziție fibroasă de 100% bumbac și 100% poliester au fost acoperite cu pelicule de Cu prin pulverizare tip magnetron, pentru a conferi conductivitate electrică de suprafață și caracter ignifug. Densitatea țesăturii și diametrul firelor au fost determinate pentru țesăturile acoperite în plasmă și pentru cele crude, cu scopul de a calcula factorul de acoperire, în timp ce testele de absorbție atomică au fost efectuate pentru a determina conținutul de cupru depus. Factorul de acoperire al țesăturii acoperite în plasmă scade ușor în funcție de timpul de procesare pentru cele patru probe analizate în cadrul studiului. Acest comportament fizic este subliniat de imaginile de microscopie optică ale țesăturilor crude și a celor acoperite și poate fi explicat prin scăderea ușoară a diametrului firului după acoperire în plasmă. Mai mult, țesăturile acoperite obținute au prezentat o bună performanță în ceea ce privește ecranarea electromagnetică, măsurată cu celulele TEM și un caracter ignifug, evidențiat prin determinarea punctului de topire.

Cuvinte-cheie: ignifugare, ecranare electromagnetică, elemente de construcție, bumbac, probe PES

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Buildtech technical textiles domain requires new technologies for manufacturing flexible eco-friendly materials. Magnetron sputtering allows nanometer scale coatings on fabrics with various functionalities and benefits. Novel fabric functionalities consist in electromagnetic shielding and fireproofing. This paper aims to analyse the cover factor of raw and plasma coated fabrics, meant for the above mentioned functionalities. Fabrics with fibrous composition of 100% cotton and 100% polyester (PES) were coated with Cu films by magnetron sputtering in order to render surface electrical conductivity and fireproof character. Fabric density and yarn diameter were determined for raw and plasma coated fabrics in order to compute the cover factor, while atomic absorption tests were performed in order to determine the content of deposited copper. The cover factor of plasma coated fabric slightly decreases as a function of the process time for the four analysed samples within the study. This physical behaviour is underlined by optical microscope images of raw and coated fabrics and can be explained through slight decrease of yarn diameter after plasma magnetron coating. Moreover, obtained coated fabrics presented good performance regarding electromagnetic shielding, measured with TEM cell and fire-proofing character, evidenced by melting point determination.

Keywords: fireproofing, electromagnetic shielding, building elements, cotton, PES samples

INTRODUCTION

Safety and protection of human's health against various external factors has been always a priority. Electromagnetic interference is nowadays increasing due to the accelerated development of telecommunication. The shielding of electromagnetic radiation is especially important for the preservation of human's health and proper functioning of electronic equipment. The specifications imposed for the materials used for electromagnetic shielding depend on the shielding application requirements. In the case of Buildtech technical textiles, metallic braids or metallic coated fabrics are considered in order to improve shielding effectiveness [1–2].

Fire may be an enemy for humans, if it takes one unprepared. In combination with highly flammable materials, flames may generate real disasters. Classical textile materials for home textiles, upholster etc., have a great disadvantage: they are highly flammable due the fibers they are made of, either consisting of natural fibers (linen, hemp, cotton, wool and blends thereof) or of synthetic fibers. In order to prevent undesired events and to avoid disasters, textile materials may be functionalized by specific substances and become thus not only a solution for design and comfort, but a solution for safety as well. Fireproofing of textile materials [3] represents nowadays the most advanced procedure of fighting against accidents caused by flames.

Plasma coating may be used to impart various other functionalities to textile materials: anti-static, anti-microbial, fireproof, anti-felting (for wool), hydrophilic, hydro-/oleo phobic etc. [4]. Magnetron sputtering technique for rendering electrical conductivity to textile fabrics has been reported in the scientific literature. PP nonwoven fabrics were coated with Zn-Bi metal particles for achieving electromagnetic shielding of about 40 dB [5–6]. In order to render electrical surface conductivity for textile fabrics, carbon nanotubes were attached on PET fabrics by means of oxygen plasma pre-treatment and subsequent acrylic acid grafting [7], while shielding properties were assessed by absorption loss measurement via vector network analyzer [8]. Electromagnetic shielding was achieved on fabrics as well with other modern techniques, such as coating with carbon or PEDOT-PSS conductive polymers [9], or printing technique [10]. Measurement of shielding effectiveness was usually performed via coaxial TEM cell, according to ASTM D-4935 standard for plane materials [11, 12]. On the other hand, plasma coating for fireproofing textiles is a relative recent technique. Plasma induced graft-polymerization was applied in order to impart fireproof properties on cotton textiles [13], PAN fabrics [14] and silk [15]. Other research studies reported activation of wool fabrics via ultrasonic waves and treatment by organic di-carboxylic acids in order to increase uptake of zirconium and titanium salts, which improve their fireproofing [16]. Good results of fire proofing silk fabrics by means of flavonoids-metal salts combination was reported in [17]. An optimization study on fire proofing of cotton woven fabrics was performed by means of classical finishing agents, having as independent parameters solution concentration, temperature and process time [18].
Industrial upscale of plasma technologies for textiles is nevertheless still limited, for various reasons such as: different deposition on warp and weft, irregular deposition compared to padding and the large dimensions of fabrics [19].

The present research study tackles the optimal woven fabric structure with EM shielding and fireproof properties destined to the Buildtech domain. This paper focuses on the analysis of cover factor [20–21] for raw and plasma coated fabrics used within the research study.

MATERIALS AND METHODS

In order to obtain adequate flexible eco-friendly materials for BUILDTECH domain with fireproof and electromagnetic shielding character, various samples of woven fabrics were manufactured, coated with magnetron plasma and tested.

Materials

Woven fabrics from 100% cotton (BBC) and 100% polyester (PES) were used for this purpose. Several samples with various yarn count, density and specific mass were designed and manufactured. Raw materials and fabric structure were selected in order to meet requirements of Buildtech technical textiles: good mechanical resistance, light weight and cost-effectiveness. For studying the cover factor, two samples from 100% cotton and two samples from 100% PES were selected and coated by magnetron plasma with copper films of 400 and 1200 nm. Raw and coated fabric samples were comparatively assessed. Table 1 presents the physical-mechanical properties of yarns used for the four fabric samples.
Table 2 presents physical-mechanical properties of the four raw fabric samples.

Methods

The Cu coating of the textile fabrics was performed into a dedicated spherical stainless steel vacuum chamber (K.J. Lesker), pumped out by an assembly of a fore pump and turbomolecular pump (Pfeiffer), which allowed the obtaining of a base pressure prior deposition down to $3x10^{-5}$ mbar. The chamber is provisioned with a Magnetron Sputtering Source from K.J. Lesker, accommodating 2″ diameter Cu target of purity 99.999%. The fire-resistant pre-treated cotton

Table 1

PHYSICAL-MECHANICAL PROPERTIES OF YARNS INCLUDED IN THE RAW FABRICS

Property	Unit		Sample A. 100% cotton	Sample B. 100% PES	Sample C. 100% cotton	Sample D. 100% PES
Yarn fineness	tex (Nm)	Warp	53.51×1 (18.69/1)	-	91.68×1 (10.9/1)	-
		Weft	51.94×1 (19.25/1)	-	93.15×1 (10.7/1)	-
	dtex (den)	Warp	-	122×1 (109.8×1)	-	352.4×1 (317.2×1)
		Weft	-	121×1 (108.9×1)	-	339.1×1 (305.2×1)
Tensile strength	N		4.97	11.40	6.50	13.69
	Cv%		12.72	1.56	14,6	2.06
Relative elongation	%		6.03	17.59	5.5	34.53
	Cv%		11.01	3.89	12.3	7.37
Yarn diameter	µm	Warp	296.4	152	458.2	326
		Weft	311.2	134	397	310

Table 2

Property	Unit	Sample A.	Sample B.	Sample C.	Sample D.
Fiber composition	**Raw material**	**100% cotton**	**100% PES**	**100% cotton**	**100% PES**
Specific mass	g/m^2	231	88	416	207
Density Warp	No. yarns/1 cm	30	41	26.4	36.4
Density Weft	No. yarns/1 cm	16	28	16.6	21.4
Tensile strenght Warp	N	910	937	1318	2345
Tensile strength Weft	N	363	644	891	1331
Rel. Elong. Warp	%	10.61	27.2	23.7	40.0
Rel. Elong. Weft	%	8.63	28.5	12.85	38.2

fabrics of 20 cm diameter were mounted inside the chamber, on a substrate holder placed at 14 cm inclined with respect to the magnetron. In order to enhance the deposition uniformity, the samples were rotating during the process with a constant speed of 200 rotation/min. An Ar flow (99.99%) of 50 sccm was continuously introduced in the chamber during the process by means of a Bronkhorst mass flow controller, so that the pressure established during the process was 5.3×10^{-3} mbar. The discharge was ignited at 100 W by using an Advanced Energy RF generator (13.56 MHz) – model CesarR provisioned with an automatic matching box for adapting the impedance; under the described experimental conditions, the measured deposition rate of Cu at the substrate level was around 9 nm/min. The deposition time was set such as to insure a coating thickness of 400 nm (45 min) and 1200 nm (135 min) onto the textile.

The cover factor relation

The cover factor indicates the degree in which the surface of a fabric is covered by a set of yarns. One method of calculating the cover factor of fabrics is function of yarn diameter and fabric density, according to relation (1) [20].

$$e_t = d_u \cdot Pu + d_b \cdot Pb - d_u \cdot Pu \cdot d_b \cdot Pb \qquad (1)$$

Where following notations apply:
e_t is Cover factor;
d_u – diameter warp yarn (μm);
Pu – fabric density warp (number of yarns/10 cm);
d_b – diameter weft yarn (μm);
Pb – fabric density weft (number of yarns/10 cm).

RESULTS

In order to be able to compute the cover factor, yarn diameter and fabric density were measured for the raw and plasma coated fabric samples. Yarn diameter was measured via the optical microscope Projectina, by assessing width of yarn to microscope scale in micrometer, while fabric density was determined according to standard EN 1049-2/2000 – method A and B.
Moreover, optical microscope images were taken for raw and plasma coated fabrics, in order to assess the structure of yarns surface and to verify the influence of plasma coating on the diameter of yarns. Table 3 presents values for density and diameter of raw and plasma coated fabrics, destined for computing the cover factor, according to relation (1).
Figure 1 shows optical microscope images of the fabric surface of 100% cotton sample A, in raw (figure 1, *a*) and plasma coated condition (figure 1, *b*), with a magnification of ×5.

Table 3

Sample	Treatment	Experimental data				Result
		Density Warp	***Density Weft***	***Diameter Warp***	***Diameter Weft***	***Cover factor***
		No. yarns/1 cm		μm		
Sample A.	RAW	30	16	308.4	331.2	0.96483802
	P1-Plasma 45 min	30.2	16.4	310	318	0.96947298
	P2-Plasma 135 min	30	16.2	306	310	0.9591804
Sample B.	RAW	41	28	152	134	0.76457536
	P3-Plasma 45 min	41	28.4	120	126	0.67378272
	P4-Plasma 135 min	40.4	28.4	118	120	0.65505382
Sample C.	RAW	26.4	16.6	458.2	397	1.07148578
	P5-Plasma 45 min	26	16	376	424	0.99279616
	P6-Plasma 135 min	26.4	16.4	388	396	1.00852562
Sample D.	RAW	36.4	21.4	326	310	1.06282302
	P7-Plasma 45 min	37	22	258	276	0.98216688
	P8-Plasma 135 min	37	22	222	244	0.91727248

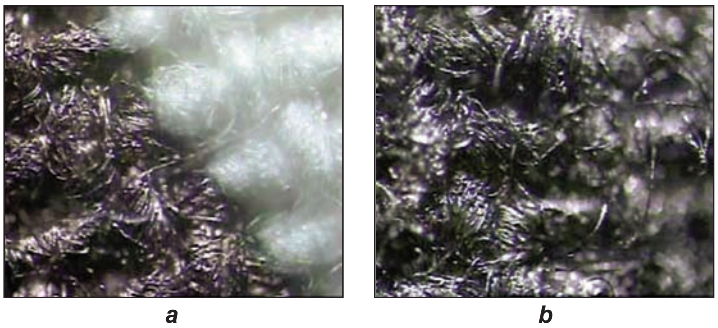

Fig. 1. *a* – Surface structure of raw/plasma coated cotton fabric; *b* – surface structure of plasma coated cotton fabric

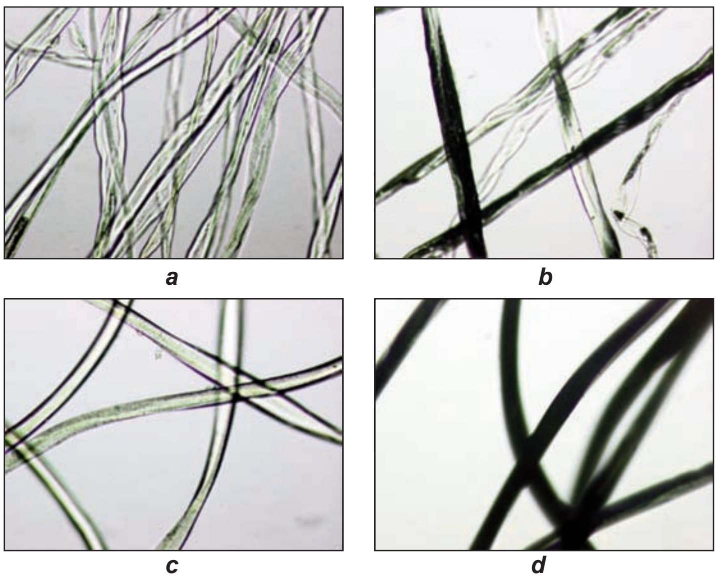

Fig. 2. Optical microscopy images (magnification ×40) of fibers from raw and plasma coated cotton (*a*, *b*) and respectively PES (*c*, *d*) fabrics

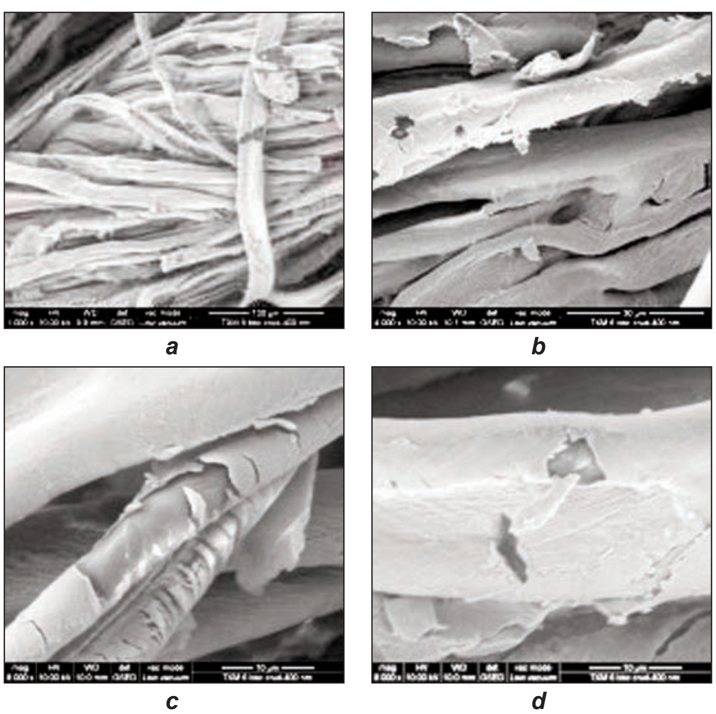

Fig. 3. SEM images of plasma coated cotton fabrics (Cu film of 400 nm), taken at various magnifications: *a* – ×1000; *b* – ×2000; *c* – ×4000; *d* – ×8000

Figure 1 evidences shrinkage of yarn diameter after plasma coating, due to deposition of copper film onto bulk textile material. Figure 2 presents optical microscopy images of fibers taken out of 100% cotton and 100% PES fabric samples in raw and plasma coated condition, in which the blackish regions of the fibers evidence the Cu coated regions. Figure 2 presents optical microscope images of fibers extracted from raw and coated cotton and PES fabrics, with a magnification of x40.

Figure 3 presents SEM images of cotton fibers deposited with 400 nm copper thin films. They evidence on one hand the uniform aspect of the coating onto the fibers, but also the presence of few cracks and small portion of delamination.

Atomic absorption tests were performed on AAS 880 Varian spectrometer in order to determine the content of deposited copper in mg per g of coated textile fabrics. Four different measurements were performed for each type of sample, and table 4 includes the averaged results of the four types of fabric samples coated for 45 min and 135 min.

Results of atomic absorption evidence deposited copper content on textile fabrics. Substantial copper concentrations of 51–172 mg Cu/g of material could be determined. All fabrics coated for 135 min showed larger copper content than fabrics coated for 45 min. Moreover, both fabrics of 100% cotton and 100% PES with lower specific mass (sample A and B) presented a higher amount of Cu into the structure, due to the fact that same Cu thickness was deposited onto all samples, regardless their specific mass. Highest amount of Cu was obtained for Sample B coated for 135 min (1200 nm thin film), which led to 172 mg Cu/g material.

Functional properties for electromagnetic shielding were achieved for plasma coated samples on both sides of the fabric, with results presented for sample D in figure 4.

Special functional properties for fireproofing character were achieved for 100% PES samples coated with silicon oxide. Melting point has increased from 254,4°C of raw materials to 258,2 °C for fabric samples treated with silicon oxide (according to SR 13231:1994).

DISCUSSION

The values of cover factor as in table 3 show that this fabric parameter is decreasing with plasma coating. Smaller values are obtained for a longer process time (135 min when compared to 45 min). This is mainly due to shrinkage of yarn diameter, while fabric density only slightly decreases, with values within the margin of error. Optical microscope images show that yarns coated with Cu thin

Table 4

Sample	Sample A (100% cotton)		Sample B (100% PES)		Sample C (100% cotton)		Sample D (100% PES)	
	P1 - Plasma 45 min	*P2 - Plasma 135 min*	*P3 - Plasma 45 min*	*P4 - Plasma 135 min*	*P5 - Plasma 45 min*	*P6 - Plasma 135 min*	*P7 - Plasma 45 min*	*P8 - Plasma 135 min*
Cu quantity reported on 1 g material (mg/kg)	257.5	290.12	6.94	15.69	45.28	43.74	22.76	51.01

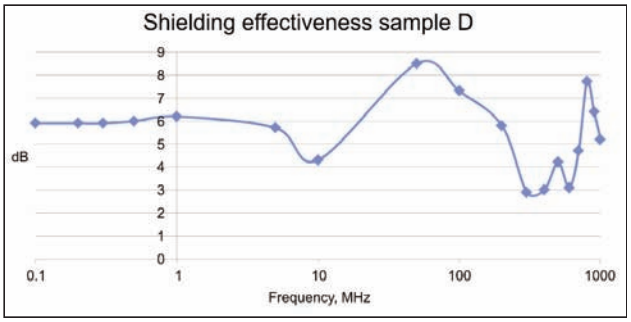

Fig. 4. Shielding effectiveness 100% PES fabric sample D coated on both sides

films by magnetron sputtering plasma have a shrinkage tendency, with effect on yarns diameter. This is mainly to bulk properties of raw cotton fabrics and its contraction under bombardment with Cu atoms provided by the sputtering process, which lead to Cu thin film deposition. PES fabrics undergo a similar physical modification, after sputtering plasma coating. Uniform copper film deposition of fabrics was evidenced by SEM images. The Cu concentration in mg/g of material was determined by atomic absorption, showing an increase of content with the deposition time both for cotton and PES fabrics. Proposed functionalities for building elements, such as electromagnetic shielding and fireproofing character were evidenced by TEM cell measurements and melting point temperature, with good results: 6-8 dB shielding effectiveness for double side coated sample D in the frequency range of 0,1–1000 MHz and an increase of melting point of 4 °C for silicon oxide coated PES fabrics. Both these functionalities are going to be further improved within the research study.

CONCLUSION

The research study envisaged flexible materials for BUILDTECH domain, having fireproof and electromagnetic shielding functionalities. These functionalities were achieved by an innovative manufacturing technology, namely magnetron plasma coating on textile fabrics. This paper focuses on a single aspect of the obtained fabrics: their cover factor. Cover factor of a woven fabric is expressed in relation to yarn diameter and fabric density. These two parameters were investigated for 100% cotton and 100% PES raw and plasma coated fabrics. The four analyzed fabrics presented lower cover factor when coated with plasma sputtering than the raw samples. This is mainly due to shrinkage of yarn diameter upon exposure to energetic Cu atoms originating from the magnetron sputtering plasma, while the fabric density remains unaffected.

For the future, the research study envisages integration within building walls of shielding materials to reduce the electromagnetic interference upon inside electronic equipment and to simultaneous have fireproof character. Such materials have to be cost-effective and eco-friendly, a further aim of our research study. The research study for achieving woven fabrics with electromagnetic shielding and fireproof properties have been performed within the project with title “Manufacturing textiles with electromagnetic shielding and fire-retardant properties by plasma-based methods” (Acronym TexEMFiRe).

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Authors:

LILIOARA SURDU [1], EMILIA VISILEANU[1], ION-RĂZVAN RĂDULESCU[1], IRINA SANDULACHE[1], CORNELIA MITRAN[1], BOGDANA MITU[2], CRISTIAN STANCU[2], ALINA ARDELEANU[2]

[1]INCDTP – Bucharest, Str. L. Patrascanu 16, 030508, Bucharest, Romania
e-mail: certex@certex.ro

[2]INFLPR – Măgurele, Str. Atomistilor No. 409, 077125, Bucharest, Romania
e-mail: mitub@infim.ro

Corresponding author:

LILIOARA SURDU
e-mail: lilioara.surdu@certex.ro