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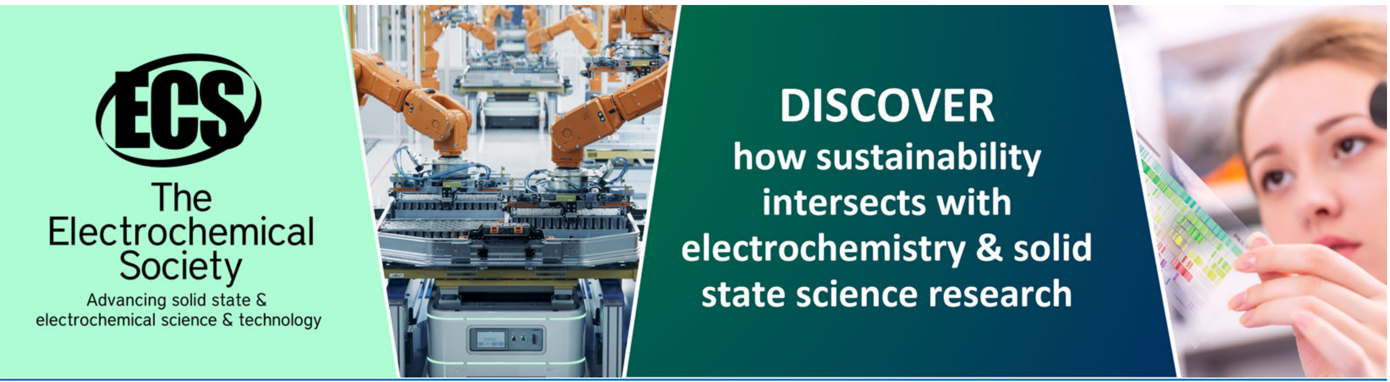

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Nanolevel Functionalization of Natural Fiber Textiles

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Abstract. One of the main tasks of presented research are to impact the additional value on natural fabrics by adding them new properties with a metal nano-level coating, evaluate coating technologies. Having the ability to control the surface of a natural fiber offers great rewards that go far beyond pure economics as natural fibers are renewable and biodegradable. The paper describes the process of vacuum evaporation and magnetron sputtering of copper coatings on cotton textile materials, analysis of the metal coated textile surface by laser laboratory complex and SEM. The investigation results evince that laser laboratory complex measurements of reflected and through covered material transmitted light can be applied to trace the unevenness of deposited metal film on the covered fabric surface and its changes from exploitation impacts without samples destruction.

1. Introduction.

Functionalization of textile materials is of major importance and an essential component in textile processing for imparting of additional properties. Textile materials have intrinsic properties that make them very valuable: flexible, light weight, strong, good touch, softness, etc, because of this, they are excellent for imparting additional functionalities. Nanotechnology can enrich the textile industry in several ways. First of all, and most obvious, it creates many new possibilities and improved functions in textiles. New materials can be developed, and new properties can be added to existing materials by the use of special-treated fibres, coatings and e-textiles. [1] Therefore, beside all new functions, the trend towards even thinner fiber creates a need for thin nano-scaled or nano-structured finishing. [2]

2. Materials and Methods.

Commercial woven plain wave 100% cotton fabric with the linear surface density 276.19 g/m^2 from yarns of linear density 9.2 Tex has been used in the experiment. The thickness of the textile fabric is ~ 0.25 mm; the measurement was taken by the textile thickness tester (“TH-25”, “Zapadpribor”, Ukraine). To provide good interfacial contact between fiber surface and deposited metal, the surface of cotton fabric samples were washed at temperature 90°C with detergent without optical brighteners, nevertheless, the washing does not remove all the oil, because of that after samples were immersed in 80% acetone solution at room temperature for 5 minutes [3, 4], and were washed twice with distilled water (ISO 9001, ISO 14001), the drying step was carried out on a horizontal surface.

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2.1. Surface modification technique for textile materials.

Low temperature plasma treatment is a quite new technology for the natural fiber textiles; plasma is essentially a dry process providing modification of the top nanometer surface. [5] This means that the plasma only affects the outermost thin layer of the substrate, i.e. it is a real surface modification technique. In this way, the typical textile properties (hand, softness, flexibility, etc.) are not influenced. The advantages of plasma technology are its potential environmental friendliness and energy conservation benefits in developing high-performance materials. [5]

The results of the previous research evince that pre-treatment knitted fabrics from cotton+PA and elastan threads with low temperature plasma have significant impact on sustainability of coating. [3, 4] In current research plasma treatment apply as activation method of the surface of 100 % cotton fabric is used too.

2.2. Vacuum evaporation technology and magnetron sputtering technology.

Vacuum evaporation is one of the most commonly used methods for deposition of functional films on to the substrates, as process allow vapor particles to deposit directly on to the substrate, where vapor particles condense back to a solid state, forming a functional coating. The vacuum evaporation process involves two basic stages: the evaporation of a functional material and the condensation on the substrate. Vacuum is used to prevent the collision of the evaporated particles with the background gas or other unwanted particles. [5] A control sample, sital (glass ceramic) plate, was placed near the fabric samples, to control the thickness of copper coating by vacuum evaporation technology. After thickness of copper coating have been measured by microinterferometer “MII-4”. [4] Short time of deposition (1-2 seconds) in vacuum chamber “UVN-2U” is sufficient to get a thin copper coating without destruction of substrate from natural fibres. The results of the experiment evidence that during 1 second deposition 60-70 nm thick coating of copper is formed on the surface of cotton fabric sample. [3, 4]

From publicized materials analyzing [2, 6] followed, that coatings deposited by magnetron sputtering process in exploitation are more sustained as it is a plasma coating process whereby sputtering material is ejected due to bombardment of ions to the target surface. During the experiment the vacuum chamber of the PVD coating machine were filled with an inert gas - argon. By applying a high voltage (700V) a glow discharge is created, resulting acceleration of ions to the target surface. The argon-ions will eject sputtering materials from the target surface (sputtering), resulting in a sputtered coating layer on the products in front of the target. The results of experiment evidence that during 1 minute of sputtering 60-70 nm thick coating of copper is formed on the surface of each of seven fabric samples fixed on the rotating disk (12 min^{-1}).

2.3. Non-contact method of surface examination.

Diffuse light reflection is peculiar for woven plain wave 100% cotton fabrics, due to geometry of the it’s surface (relief, texture, trim). Most of the metals reflected well incident visible light rays. The hypothesis is based on the difference of uncoated and coated fabric surface reflection mechanisms that leads to opportunities to control deposited coating properties. In order to get a detailed insight of the textile surface changes after it’s coating by metal and after it’s exploitation, as well as to develop a tool for comparative analysis, the surface of the sample has been examined with the laboratory laser complex. Micro-laser 10STA-01-10 with wavelength 560 nm has been used, reflected/transmitted light intensity measurements have bee taken with a digital oscilloscope Peak Tech 1145 (80 MHz). The experiment has been carried out by the laser beam, changeable angles between falling and reflected light beams were -20°, -15°, 5°, 10°, 15° and 20° degrees (figure 1). The obtained measurements show the level of impact on the changes of surface reflective properties the result of its coating with copper and after exploitation process. [3, 4]

Three sample groups with four samples for each have been prepared – one for sputtering technology, one with plasma pre-treatment and other group without pre-treatment for vacuum evaporation corresponding to methodic mentioned above. After copper deposition and washing, reflected light has been measured in three different surface spots for each sample; the average values of twelve different measures for every sample group before and after washing have been calculated and presented in graphs for each deposition.

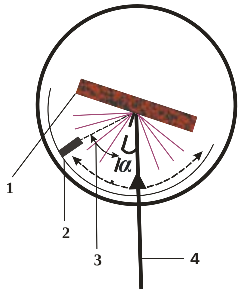

Figure 1. Laboratory laser complex for the measuring of reflected light: 1- textile sample on holder; 2- detector of reflected light; 3- changeable angle between falling and reflected light beams β; 4- laser beam;

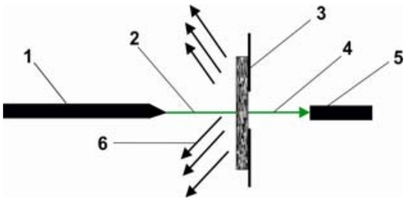

Figure 2. Scheme of the measuring of transmitted light: 1- laser; 2- incident laser beam; 3- textile sample on holder; 4- passing laser beam; 5- detector of transmitted light; 6- scattered light.

The transmitted light measurements make it possible to determine the evenness level of the copper layer and the copper layer changes after exploitation impacts (figure 2). Transmitted light has been measured in ten different spots of sample for each one sample of mentioned groups. The average values of 40 different sample spots measurements have been calculated and presented in graphs for each deposition.

3. Results and Discussion.

The graph represented in figure 3 demonstrates the changes in reflectance intensity of the samples before washing, after one washing and after second washing cycles. The amount of reflected light decrease rapidly by 24% after the first washing, what signifies that a part of the copper coating has been washed out, but the second washing cycle influences the copper coating insignificantly light reflectance intensity decreases by 6%.

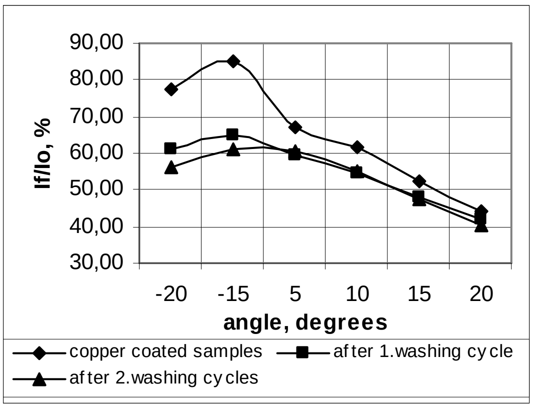

Figure 3. Comparison of reflected light of samples coated by magnetron sputtering.

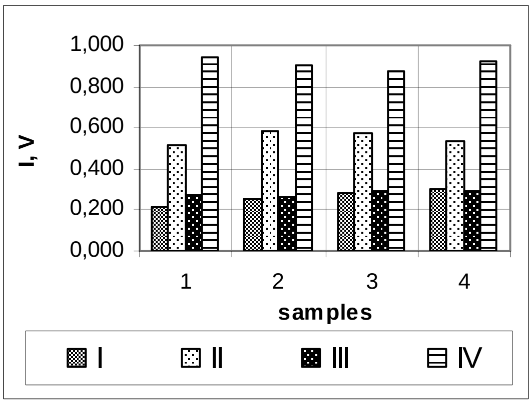

Figure 4. Transmitted light measurements after samples washing: I – coated by magnetron sputtering technology; II – coated by vacuum evaporation technology with plasma; III – coated by vacuum evaporation technology; IV – non-coated material.

The graph represented in figure 4 desinates that the highest light transmittance of the samples after washing is inherent to non-coated cotton fabric (□94 %), whereas the sample group subjected to to plasma pre-treatment before vacuum evaporation has significantly lower indicators of light transmittance (□55 %), that evidences that copper particles still remaining on the surface of the samples surfaces. Level of transmitted light of samples of first and third groups are almost equal (□27 %) or from 3 to 3.5 times lower than for non-coated samples and from 1.7 to 2.4 times lower than transmittance of the second group samples. As consequence conclusion could be drawn out that plasma pre-treatment is not appropriate for pure cotton fabrics before copper deposition by both technologies.

From SEM micrographs (Tescan, Mira//LMU Schottky) in (figure 5 b) are seen that after with copper coated textile washing copper coating of samples have microdefects that explain the reflected light measurements decreasing and transmitted light measurements increasing.

Figure 5. Micrograph of sample coated with copper by vacuum evaporation technology a) before and b) after washing

4. Conclusion.

From results present that modification of surface by plasma for 100% cotton fabric reduces the copper coating adhesion.

Laser laboratory complex measurements of reflected and passing through covered material light can be applied to trace the unevenness of deposited metal film on the covered fabric surface and its changes from exploitation impacts without destruction of samples.

Transmitted light measurements methodology limits the thickness of the fabric, this methodology can be applied only for plain fabrics, but thick fabrics will not transmits the light, but reflected light measurements methodology can used for all types of materials.

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