The Effect of the Amount of Deposited Copper on Textile Surface Light Reflection Intensity

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Abstract: **Main tasks of presented research are to impact the additional value on natural fabrics by adding them new properties with a metal nano-scale coating, evaluate coating technologies. The paper describes the process of 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 light can be applied to trace the changes of the covered fabric surface without samples destruction.**

Keywords: **magnetron sputtering, cotton textile, laboratory laser complex.**

I. Introduction

Functionalization of textile materials is of major importance and an essential component in textile processing for imparting of additional properties. [1]

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. [1]

The techniques used to functionalize textiles surface generally are grouped onto two major categories: chemical and physical. Physical methods are based on usage of non-chemical forces to control deposition of functional material on the textile surface. [2]

Sputter coatings also provide new approaches to the functionalization of textiles, using metallic, oxide, polymer and composite coatings to achieve various performance properties. [2]

Magnetron sputtering techniques are widely used to deposit different kinds of coatings, such as metallic coatings, polymer coatings and composite coatings. Magnetron sputtering was developed to solve the electron problem by placing magnets parallel to the target surface, which can constrain the motion of secondary electrons ejected by the bombarding ions the close vicinity of the target surface. The ion current is also increased by an order of magnitude over conventional diode sputtering, systems, resulting in faster deposition rates at lower pressure. [2]

In comparison with other deposition methods, a most important advantage of sputtering is that even the highest melting point materials are easily sputtered. Sputtered films typically have better adhesion on the substrate than evaporated films. The thickness of a sputtered film is much more easily controlled by fixing the operating parameters and simply adjusting the deposition time. [2]

Nearly all metallic materials can be deposited on textile substrate by sputtering. [2] Metallization is a metal coating process that adds value to and improves the functions of textile materials. Textile materials modified with different metals have attracted a great deal of attention owing to their potential applications in technology and design fields. [2]

SEM observations clearly revealed a significant difference in surface morphology before and after the copper sputter coating. The functionalization of textile materials using a sputter coating of copper can significantly modify surface properties of materials. The development of modified materials with improved properties will open up new possibilities for applications of these materials. [2]

Copper is considered to be safe for humans, as demonstrated by the widespread and prolonged use by women of copper intrauterine devices (IUDs). [3] Animal studies have demonstrated that copper fibbers do not possess skin sensitizing properties. These findings are in accordance with the very low risk of adverse skin reactions associated with copper. [3, 4] In contrast to the low sensitivity of human tissue (skin or other) to copper, microorganisms are extremely susceptible to copper [5, 6]: copper surface kills over 99.9% of bacteria (Escherichia coli, Enterobacter aerogenes, MRSA, Pseudomonas aeruginosa, Staphylococcus aureus) for 24 hours. [7]

II. Materials and Methods

A. Materials

Commercial woven 100% plain wave cotton fabric with surface linear 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 approximately 0.25 mm; the measurement was taken by the textile fabric thickness tester (“TH-25”, “Zapadpribor”, Ukraine).

B. Surface preparation technique for textile surface

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 [8, 9], and were washed twice with distilled water (ISO 9001, ISO 14001), the drying step was carried out on a horizontal surface.

C. Magnetron sputtering technology

Magnetron sputtering is a plasma coating process whereby sputtering material is ejected due to bombardment of ions to the target surface. The vacuum chamber of the PVD coating machine is filled with an inert gas - argon. By applying a high voltage (~700V), a glow discharge is created, resulting in acceleration of ions to the target surface and a plasma coating. 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.

Sputter coating also provides the most promising technology for the surface functionalization of textile materials as offers a number of advantages over other technologies for textile materials [2]:

1) an abundance of deposition materials exists, such as metals, metal oxides and polymers;

2) deposition takes place at low temperatures for polymer fibers;

3) deposited material adheres well to the fibrous substrates;

4) different deposition materials can also be combined.

Physical vapor deposition, especially sputtering technology, has been regarded as environmentally friendly technique for the functionalization of textile materials. [1]

The results of experiment evidence that during 1 minute of sputtering 60-70 nm thick coating of copper (Fig. 1) is formed on the surface of each of seven fabric samples fixed on the rotating disk (12 min^{-1}).

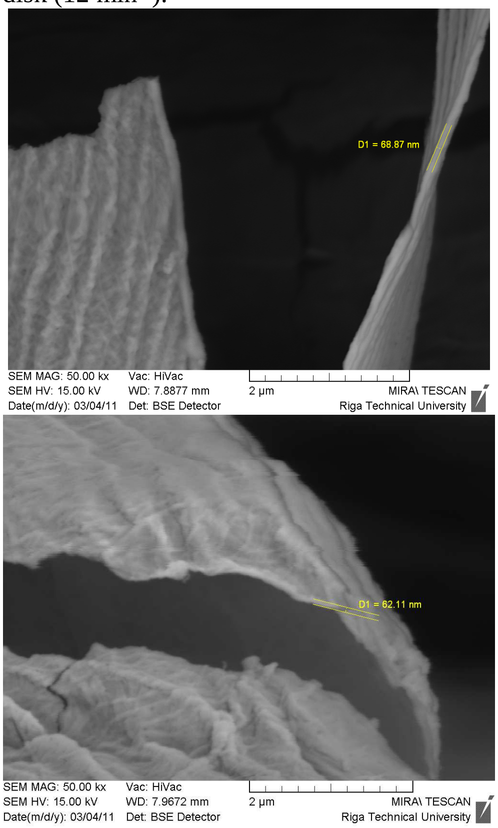

Fig. 1. Copper coating thickness measurement by SEM; magnetron sputtering time 60 seconds

D.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). 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 surfaces of the samples were examined with the laboratory laser complex.

For reflected light intensity was used "Micro-laser 10STA-01-10" with wavelength 546 nm, reflected light intensity measurements were taken with photo diode, after that light reflection intensity were displayed on digital oscilloscope "Peak Tech 1145 (80 MHz)". The angle between incident and reflected angles were varied from -45° to 45° with a step 5°.

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.

After copper deposition, reflected light was measured in three different surface spots for each from 3 samples prepared corresponding to each deposition time mentioned before; the average values of nine different measures for every sample group before and after abrasion and washing tests were calculated and presented in graphs for each deposition time.

According to the Lambert's cosine law, if the solid is reflected uniformly in all directions, then the radiation intensity of the wave of a given direction (I_β) must be proportional to the cosine of the angle β and the incident beam intensity (I_0) (1).

An important consequence of Lambert's cosine law is that when such a surface is viewed from any angle, it has the same apparent radiance.

$$I_\beta = I_0 \cos\beta \qquad (1)$$

Taking into account, that natural fibres and surfaces of textile materials from them had diffuse light reflection, and surface roughness partially even out during metal deposition process, as well changes of other surface properties occur, the comparison of light reflection intensities measurements of textile samples with different surface properties and treatments gives the opportunity to received information about textile surface properties, including structure changes [4].

III. Results and Discussion

A.Scanning Electron Microscopy

For experiment were prepared seven samples at each sample groups with different time of sputtering 40, 70, 90, 120 and 135 seconds.

The results of experiment evidence that the thickness of samples prepared by sputtering time 40 seconds is approximately 40 – 50 nm, 70 seconds is approximately 70 – 80 nm, 90 seconds is approximately 90 – 100 nm, 120 seconds is approximately 120 – 140 nm, 135 seconds is approximately

135 – 150 nm. The deposited copper coating thickness has almost the straight-line correlation with sputtering time from 40 to 135 seconds.

Copper coating can be deposited on cotton textile by magnetron sputtering technology without destruction of substrate from natural fibers (Fig. 2 – Fig. 7).

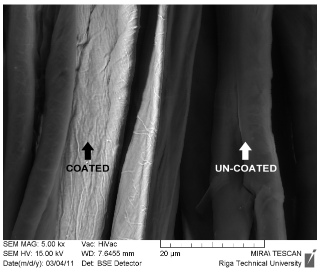

Fig. 2. Difference between coated with cooper yarns and non-coated yarns of cotton textile.

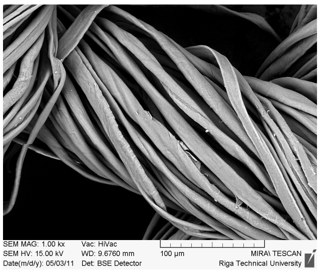

Fig. 3. Copper coating - sputtering time 40 seconds

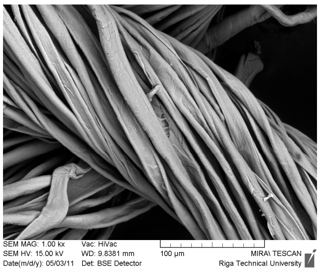

Fig. 4. Copper coating -sputtering time 60 seconds

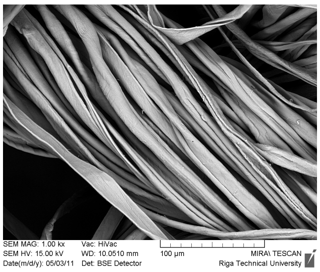

Fig. 5. Copper coating - sputtering time 90 seconds

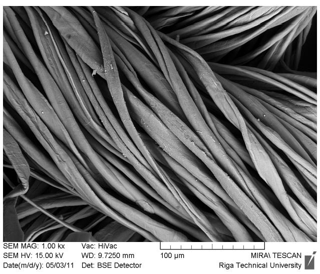

Fig. 6. Copper coating - sputtering time 120 seconds

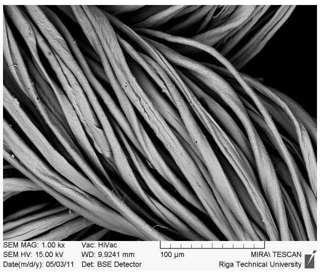

Fig. 7. Copper coating - sputtering time 135 seconds

From SEM micrograph seen that prepared with magnetron sputtering technology copper coatings on cotton material surface are without defects, even distributed not only on the surface of yarns but as well in depth of textile material. Micrographs also evidence, that the sputtering time over 40 seconds not influenced the surface coating quality, with longer coating processing 70, 90, 120 and 135 seconds only was increased copper coating thickness.

SEM micrographs (Fig. 2 - Fig. 7) as and optical microscope picture (Fig. 8) evince that copper coating is not a flat film on cotton textile surface and copper particles are deposited on fibers without changing the textile surface structure.

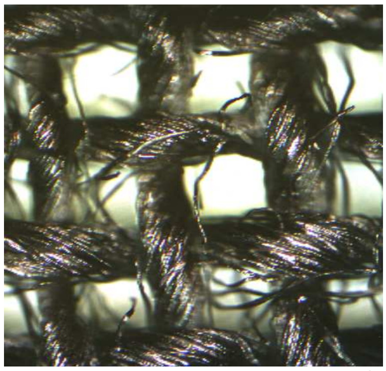

Fig. 8. Optical microscopy image with 250 enlargement of cotton textile coated with copper.

For evaluation of the exploitation impact on copper coating abrasion and washing tests were carried out. For abrasion test samples from all sputtering time groups were exposed to 3000 abrasion cycles with abrasive material press strength 1 kg or 71g on 1 cm² that imitated textile surface friction on shoes leather inside surface; for washing test samples from all sputtering time groups were washed at temperature 30ºC with detergent without optical brighteners, drying step was carried out at a horizontal surface.

From SEM micrograph (Fig. 9.) seen changes after abrasion test of copper coating on cotton textile surface received during sputtering time 40 seconds, the micrographs evince that approximately 30% of copper coating was gone from samples surfaces, the same results were received after samples washing (Fig. 10.). At the same time, approximately 70% of copper coating was remaining on the surface of samples and it is enough for cooper antimicrobial and antifungicidal impact.

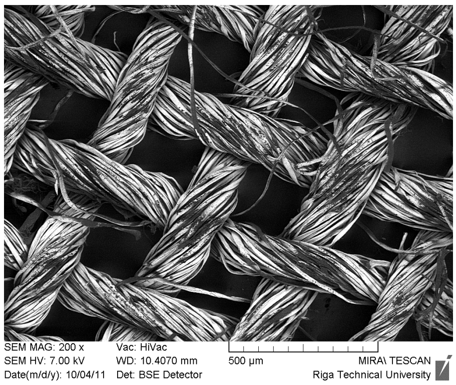

Fig. 9. Copper coating after abrasion test; magnetron sputtering time 40 seconds

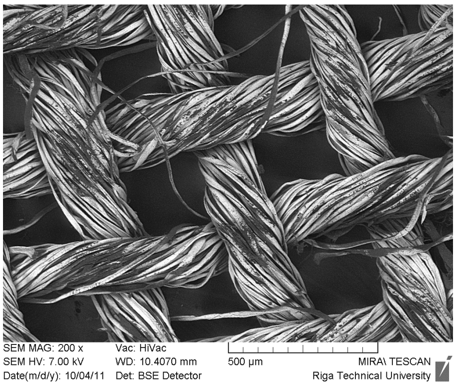

Fig. 10. Copper coating after washing test; magnetron sputtering time 40 seconds

B.Non-contact method of surface examination

Measurements of the reflected light represent changes of the textile surface in result of metal deposition and covered textile exploitation.

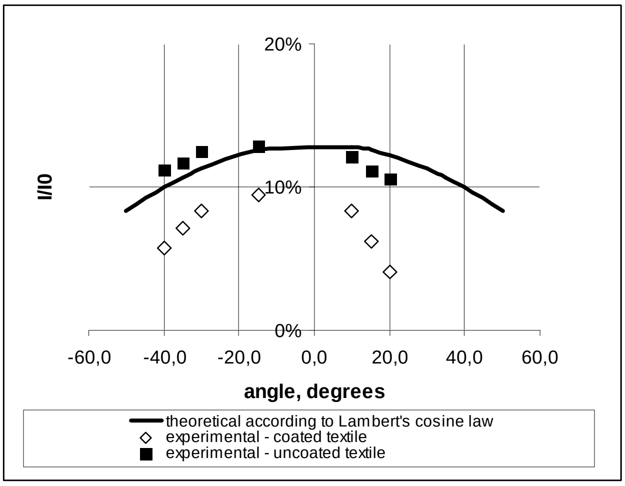

Fig. 11. Theoretical diffuse reflection curve in comparison with experimentally received reflection curves, (I/I0 - beam intensity level, rationed to the maximum)

From graph represented in Figure 11 can make a conclusion, that experimental light reflection curve (light reflection intensity of coated with copper textiles) in comparison with uncoated textile experimental light reflection curve still have diffuse reflection, this implies, that deposited copper coating on textile surface repeat textile material surfaces structures, at smallest and biggest angles deposited copper coating has observable deviation from diffuse reflection it can be explained by fact that copper coating absorbed the part of incident light.

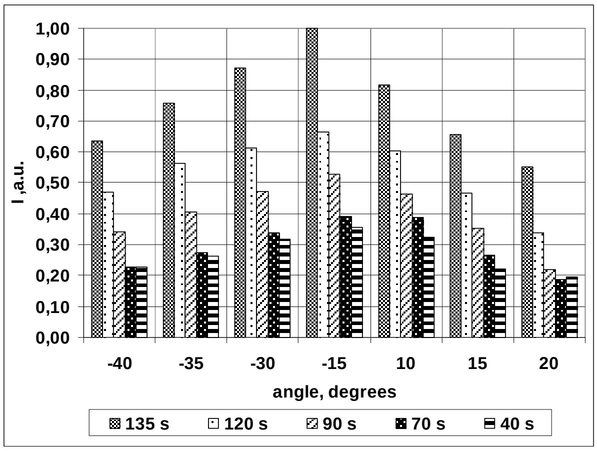

Fig. 12. Comparison of reflected light of samples coated by magnetron sputtering technology (arbitrary units – intensity level rationed to the maximum)

The graph represented in Figure 12 demonstrates the changes in reflectance of the samples depending on sputtering time; it is seen that with a longer sputtering time reflectance increase, that evidences that copper amount on the surface of textile increase. Reflected light intensity with increasing of sputtering time rapidly increased to sputtering time 135 seconds and for 60% exceeded the relevant indicator obtained by samples with sputtering time 40 seconds. It's understandable, because increased sputtering copper coating thickness and at the same time increased mirror reflection. As show micrographs of coated samples (Fig. 2 - Fig. 6), even sputtered copper coating already received with the sputtering time 40 seconds and for many applications it is inexpedient to increase this time.

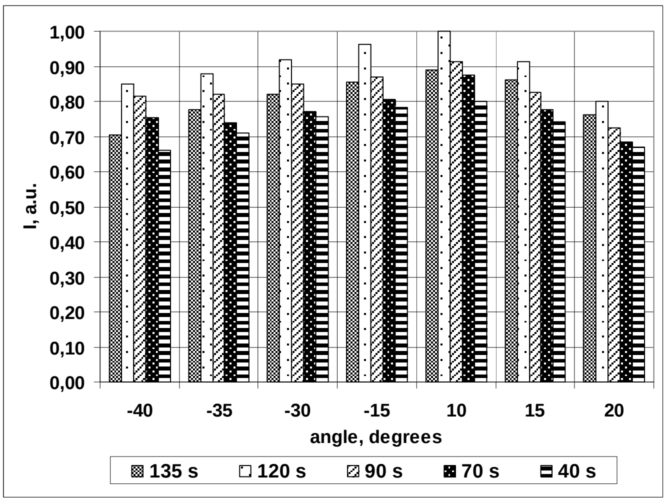

Fig. 13. Comparison of reflected light of samples coated by magnetron sputtering technology after washing test (arbitrary units – intensity level rationed to the maximum)

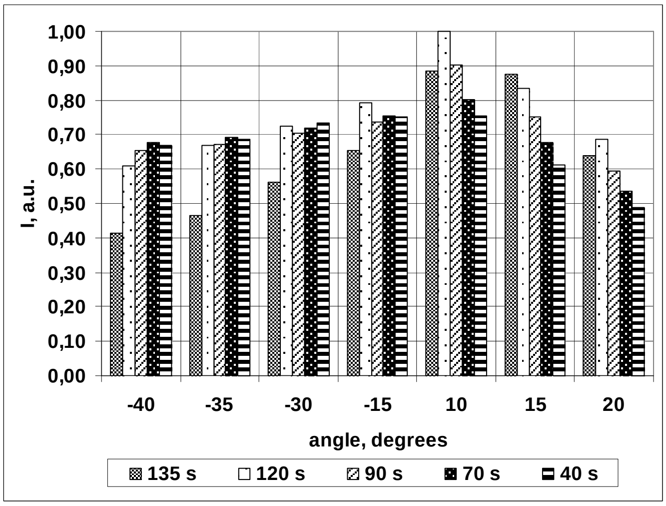

Fig. 14. Comparison of reflected light of samples coated by magnetron sputtering technology after abrasion test with 3000 cycles (arbitrary units - intensity level rationed to the maximum)

Graph (Fig. 13) evidence that for samples coated by copper at different sputtering time after washing test differences between reflected light intensity of samples groups were reduced, after washing differences in light reflection intensity between samples groups does not exceed 15%.

Graph (Fig. 14) show that for samples coated by copper at different sputtering time, after abrasion test differences between reflected light intensity of samples groups were reduced, too. To smaller angles reflected light intensity for samples groups with different sputtering time adjusted almost the one level.

This implies that in spite of sputtered coating thickness and time of sputtering, after washing and abrasion tests on all textile samples remains approximately equal quantity of copper.

Reflected light measurements as and micrographs evince that copper coating is not a flat film at cotton textile surface, but copper particles deposited at fibers and textile is not changed it's surface relief, texture, trim.

IV.CONCLUSION

Based on the results obtained and discussed previously, the following conclusions may be drawn. Copper coating can be deposited on pure cotton textile by magnetron sputtering technology with rotating disk without destruction of substrate from natural fibers and in this process, the typical textile properties (hand softness, flexibility, etc.) and surface relief and texture are not influenced.

With a longer sputtering time light reflectance increased, that evidences that copper amount on the surface of textile increased.

Carried out light reflection intensities measurements and it comparative analyses show, that sputtering time 40 seconds are enough for qualitative even copper coating establishment on cotton textile surface, 40 second long sputtering allow to achieve qualitative copper coating, even distributed not only on the surface of yarns but as well in depth of textile material.

Obtained results of light reflection intensity after washing and after abrasion test carry inference, that in spite of different sputtering time and copper coating thickness, after textile exploitation on textile surface remains approximately equal quantity of copper, it means that not independent from sputtering time define copper amount attached to textile material, but the rest copper amount come out during the exploitation of textile material, that can be explained with similar substrate and with similar formed adhesion forces between coating and substrate.

Measurements of reflected light could be used to trace the metal coating changes from exploitation impacts.

ACKNOWLEDGEMENT

This work has been supported by the European Social Fund within the project "Establishment of interdisciplinary research groups for a new functional properties of smart textiles development and integrating in innovative products", Nro.2009/0198/1DP/1.1.1.2.0./09/APIA/VIAA/148.

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Svetlana Vihodceva, Silvija Kukle, Juris Blūms, Gunta Zommere. Uzputināšanas laika ietekme uz tekstiliju virsmas gaismas atstarošanas intensitāti
Galvenais pētījuma mērķis ir papildus funkciju pievienošana kokvilnas materiāliem, ko var sasniegt, izveidojot materiāliem metāla pārklājumu.
Dabisko šķiedru un no tām izgatavoto tekstilmateriālu virsmas gaismu atstaro difūzi. Tās pārklājot ar metālu, virsmas raupjums daļēji izlīdzinās, kā arī mainās citas virsmas īpašības. Tika izvirzīta hipotēze, ka atstarotās gaismas intensitātes mērījumu salīdzināšana tekstilparaugiem ar dažādām virsmas apstrādēm ļaus iegūt informāciju par virsmas īpašību, tai skaitā struktūras izmaiņām.
Veiktie gaismas atstarošanas intensitātes mērījumi un to salīdzinošā analīze rāda, ka uznešanas laiks - 40 sekundes - ir pietiekams, lai izveidotos nepārtraukts vara pārklājums, SEM mikrogrāfijas rāda, ka 40 s ilga apstrāde nodrošina kvalitatīvu pārklājumu, kas uznests ne tikai uz virsējiem auduma pavedieniem, bet arī uz tā dziļākajiem slāņiem. Iegūtie gaismas atstarošanas intensitātes mērījumi pēc mazgāšanas un berzes cikliem ļauj secināt, ka neatkarīgi no uzputināšanas laika noteikts metāla daudzums piestiprinās pie tekstilmateriāla, bet pārējais metāla daudzums ekspluatācijas laikā noiet nost.
Piedāvātā metodika atstarotā stara intensitātes mērījumu salīdzināšanai tekstilparaugiem ar dažādām virsmas īpašībām un apstrādēm ļauj iegūt informāciju par virsmas īpašībām, tai skaitā struktūras izmaiņām pēc metāla uznešanas un tekstila ekspluatācijas.

Светлана Выходцева, Силвия Кукле, Юрис Блумс, Гунта Зоммере. Влияние времени напыления на интенсивность светоотражения поверхности текстиля
Главной задачей исследования является присвоение дополнительных функций текстилю из хлопка, что можно достигнуть при помощи нанесения покрытия из металлов. Натуральные волокна и изготовленный из них текстиль отражают свет диффузно. При покрытии текстиля металлами, частично выравнивается шероховатость поверхности текстиля, а так же меняются другие свойства поверхности. Была выдвинута гипотеза, что сравнение результатов измерений интенсивности светоотражения текстильными образцами с различной обработкой поверхности даст возможность получить информацию о изменениях свойств поверхности, а также её структурных изменениях.
Проведённые измерения отражения света и сравнительный анализ, показывают, что время нанесения 40 секунд достаточно для получения непрерывного покрытия металла, анализ СЭМ микрографий показал, что продолжительность напыления 40 секунд обеспечивает качественное покрытие, которое нанесено не только на верхние нити текстиля, но и так же на глубокие слои. Полученные измерения отражения света после мытья образцов и после циклов трения, позволяют сделать вывод, что независимо от времени напыления, определённое количество металла закрепляется на поверхности текстиля, а остальное количество металла во время эксплуатации сходит с поверхности.
Предложенная методика сравнения измерений отражения света у образцов текстиля с различными свойствами и обработками позволяет получить информацию о свойствах поверхности текстиля, а так же об изменениях его структуры после нанесения металла на поверхность текстиля и его эксплуатации.