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Investigation of Staple Yarns Modified by the Physical Vapour Deposition Method

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Abstract

The aim of the research presented in this paper was to develop a technology for obtaining electroconductive yarns using the physical vapour deposition (PVD) technique, as well as methods for testing the yarns. The authors presented a method of production of metallic coatings for staple and continuous yarns with the use of PVD processes which enabled the production of yarns with electroconductive properties. The article also presents the properties of yarns after the PVD modification processes and the sustainability of these properties after utility testing such as laundry, bending and abrasion. Yarn thermographic analysis, which reflected the quality of the outer metallic layers, is also presented.

Key words: *electroconductive yarn, yarns electrical resistance, thermovision, metal coating, physical vapor deposition, atomic force microscopy.*

Introduction

Modern technologies imply the development of various fields of industries. There are new products with new features and functions which were not known previously in such fields as textile and materials engineering. As a result of a synergistic combination of these disciplines there are new products which could be used in various applications. Modifying well-known textile accessories with the use of new technologies contributes to enlarging the range of textile items used, for example, in textronics [1 - 5].

Despite the various types of electrical conductive yarns, scientists still explore and conduct researches on new textile products and methods of their preparation. The need for “new” textile structures with electroconductive properties is caused by the need to create yarns which enable the use and maintenance of these products in a wide range.

Different types of metal have been the components most frequently used in order to impart electroconductive properties to textiles, which are used not only for decorative aims [6], for example they are more often utilised in technologically advanced products referred to as “smart” textiles.

In addition to metal there are also used various kinds of electrically conductive polymers [7, 8] and various forms of carbon [7 - 10] as one of the components, or in the case of polymers as fibre-forming material in the manufacture of yarns and other textile products.

Metal fibres and yarns are prepared using a variety of methods for placing an electrically conductive component, namely by introducing metal into synthetic fibres, impregnating the yarn surface with powdered metal, applying a metal coating to the surface of the yarn, joining metal fibres or metal yarn with other conventional yarns, as well as by a deposition on the surface or implementation inside the volume of the fibres and yarns of conductive carbon additives such as carbon black or graphite [6, 7, 11, 12].

Yarns and metal fibres are formed from various types of thin wires of different cross-sections (round, triangular or other). Metals used for such yarns are typically stainless steel, silver, copper, brass and nickel [6, 7, 11, 14, 15].

Metallised yarns and fibres are produced in several ways, the most common of which is to combine metal fibres or yarns by twisting with other fibres (yarns). Another common method is metalization of the surface using chemical processes [17-20].

Another method is the addition of a volume or surface of a fibre-forming polymer additive metal or other conductive additive [6, 7].

The metallization of polymer materials can be carried out with the help of new textile techniques such as laser, plasma and physical vapour deposition (PVD) technologies.

Methods using plasma, laser and PVD technologies are more often used because of lower costs and to avoid an excessive

burden on the environment, as in the case of chemical methods of fibre and yarn metallisation [21, 22].

These are well-known technologies when it comes to their application in mechanics, optics and electrical engineering, but are quite new in the production of a technically advanced range of textiles [4, 20, 23 - 26].

The method of sputtering thin layers of metal on textiles makes it possible to extend the range of textiles which can be used for constructing various interactive and sensor applications such as signal transmission lines, temperature or humidity sensors in clothing, and textile radiators.

Modern textiles should be targeted and open to adopting various technologies such as electronic or optical ones for producing a new generation of textile products, which contributes to enlarging the range of textiles and textile industry development [4, 27 - 31].

The aim of our work was to obtain electroconductive yarns with good utility properties as well as develop an appropriate technology allowing to manufacture such yarns in a modern advantageous process.

In this paper the results of yarn modification by means of the PVD method are presented. The investigation was focused on the electroconductive properties of yarns added by the deposition of thin silver and copper layers. Technological details and properties of the materials obtained are shown.

Materials and methods

Yarns and PVD modification

Two yarns, manufactured in a laboratory spinning process at Lodz University of Technology, were selected for surface modification using physical vapour deposition (PVD) processes. The first yarn with a linear density of 65 tex was formed from polyacrylonitrile (PAN) staple fibres by a converter system on a ring spinning frame. The second yarn was a monofilament with linear density of 212 dtex obtained from polyester, (polyethylene terephthalate - PET).

These yarns were surface modified by the formation of a metal coating using the PVD process - evaporative deposition of metals [27 - 30].

The evaporative deposition of two metals: silver and copper was carried out using the Classic 500 PVD system (Softrade Pfeiffer Vacuum, Germany).

Before metal deposition, the yarns samples were cleaned in ethanol in an ultrasonic bath for 3 minutes. Then the yarns were dried at room temperature of 21 ± 2 °C. The purified and dried yarns were put in a coater chamber under a pretension of 0.5 cN/tex. The processes of vapour deposition for both metals were carried out to obtain a layer thickness of 400 nm on the yarn. The thickness of the metal layer deposited was determined during the evaporative deposition process by the use of a quartz crystal microbalance (QCM), an integrated part of the Classic 500 PVD system.

The yarns treated by surface modification using the PVD technology were subjected to the following tests: evaluation of the surface morphology of the metallic layers formed on the yarn, assessing the level of the surface resistance, thermal imaging tests during electric current flow through yarns as well as usability testing.

Evaluation of surface topography

Evaluation of changes in surface topography with the metal layers applied to a standard material of relatively homogeneous topography (PET monofilament) was carried out using an atomic force microscope (AFM) - Multiview 1000 (Nanonics, Israel). The measurements were carried out in the tapping mode with the use of a silicon cantilever and tip with

a radius of curvature of 10 nm working at a resonant frequency of 52 kHz, customized for fibrous structure measurement (SuperSensor TM, Nanonics, Israel). Image analysis was performed using WSxM v5.0 (Nanotec, Spain) [32].

Assessment of electrical resistance

The electrical resistivity of yarns was measured using a special measuring stand developed (*Figure 1*) [33]. Examination of the surface resistance was based on stretching of the yarn between the clamps under a load of 0.5 cN/tex. The clamps were also the electrodes performing the measurement (the distance between the clamps-electrodes was 10 mm). The electrodes were equipped with pressure sensors enabling the yarns to be tested at various values of pressing force from about 22 N to about 76 N. In the case of our measurements, the force was 57 ± 0.5 N. The yarns before measuring the surface resistance were conditioned in an atmosphere of 21 °C and relative humidity of 25%. The resistance of the yarns was measured every 30 seconds for 3 minutes over a distance of 10 mm at five different locations along the length of 200 mm of the yarn (pieces of yarns of 250 mm length and sections of about 50 mm were included in the clamps). Surface resistance values were measured with the use of an Agilent 34401A multimeter (USA).

Usability testing

The surface resistance of yarns after steaming and utility tests was determined in studies using a measuring stand shown in *Figure 1* [33]. Both yarns were tested after laundering, repeated bending and abrasion. Washing was performed in accordance with the standard PN-ISO 105-C06:2010 "Textiles -- Tests for colour fastness -- Part C06: Colour fastness to domestic and commercial laundering". A bath containing a detergent solution with a concentration of 10% at 40 °C was used. The samples were rinsed and dried at ambient temperature.

Modified yarns with a length of 250 mm were tested for maintaining their electrical conductivity properties after bending in a cycle of $200 \times 180^\circ$.

The resistance to abrasion of the yarns studied was tested with the use of a stand containing rubbing material with gradation P1000. Due to the lack of standards regarding yarn abrasion, a test was carried out at a selected experimental load of 250 g and for 20 cycles of friction. The conditions selected caused the abrasion of the metallic layer without damaging the yarn. A lower load (less than 250 g) and smaller number of cycles (less than 20) did not cause significant changes in the surface structure.

Thermal imaging tests

Thermal imaging tests were conducted in order to evaluate the homogeneity

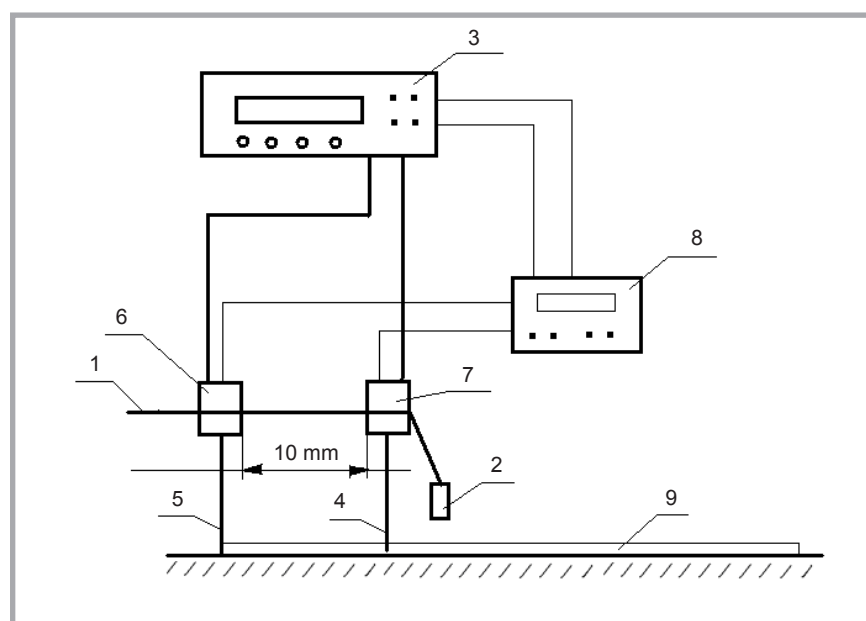


Figure 1. Scheme of measuring stand of yarn resistance: 1 – yarn tested, 2 – preloading of yarn, 3 - multimeter, 4 – moving left clamps, 5 –stationary right clamps, 6, 7 - clamps with pressure sensors and simultaneously measuring electrodes, (7 – movable clamp), 8 –measuring system of clamp pressure for yarns, 9 –slider.

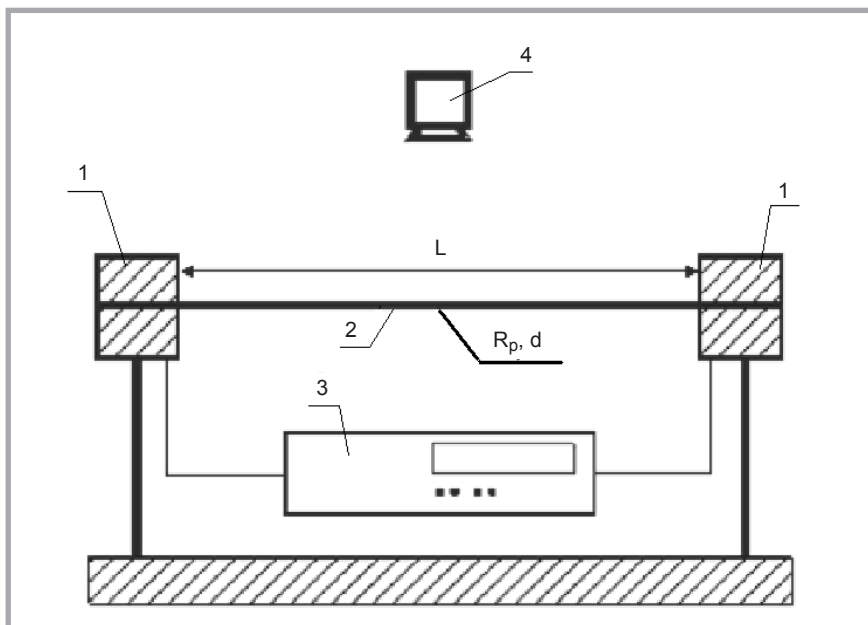


Figure 2. Scheme of the laboratory measurement system; 1 - electrical and mechanical clamps, 2 - electroconductive yarns, L - yarn length, d - yarn diameter; R_p - yarns electrical surface resistance, 3 - power supply unit, 4 - thermovision camera.

of the metallic layer covering the yarn. The temperature distribution on the surface of the modified yarn during an electric current flow through the yarns was investigated. This test is useful to assess the quality of the resulting metal coating. During an electric current I flow in the yarn with electroresistance R_p power losses of a value $\Delta P_p = I^2 \cdot R_p$ (see **Figure 2**) are dissipated. The product of the heat losses at a given time t is the energy Q_p heating the metal layers. The heat energy causes a temperature rise ΔT of the yarn and irradiation of electromagnetic waves of a wave spectrum determined. Thus the radiation distribution reflects the homogeneity of the metal layers according to **Equation 1**. Thinner sections of the layer have higher resistance thus they glow faster.

$$\gamma = \frac{l}{R_p \cdot S} \quad (1)$$

where is: γ - electrical conductivity of yarn, R_p - electrical surface resistance of yarn, S - cross-section of the yarn's metal layer through which the current flows, l - length of yarn.

Results

Results of surface topography study using AFM

The deposition of metallic layers on the linear structures selected requires their evaluation also on a nanometer scale. PET monofilaments were selected as a model material for visualization of the metal layer growth with the use of an atomic force microscope. Due to

the operating principle of the AFM microscope, studies could not be made for yarns whose surface is highly expanded and difficult to study using an AFM tip. **Figure 3** presents topography changes to a monofilament surface caused by applying a metal layer. Applying a layer with a thickness of 400 nm does not cause significant changes in the topography, and the deposited layer has a granular structure. In order to compare changes in the surface topography of a monofilament with increasing thickness of the metal layer, numerical analysis of the images received were carried out, and changes in the surface roughness were characterized by the ratio of RMS calculated according to the formula:

$$RMS = \sqrt{\frac{\sum_{i=1}^n (z_i - \bar{z})^2}{n}} \quad (2)$$

where: RMS - roughness factor, which specifies the root mean square of dimension in z direction, z_i - distance of the i -point from the average level \bar{z} , n - number of measurement points.

The results of changes in the surface roughness measured as RMS factor are presented in **Table 1**. The surface roughness of the monofilament varies with the deposition of layers. In the case of the estimated layer thickness of 50 and 100 nm, the surface roughness increases from 22.5 to 30 nm. After applying a metal layer thicker than 200 nm the metal surface is less rough than it was beforehand. The metallic layer becomes smoother and more uniform, which improves electrical conductivity. These observations indicate the grains increasing of the metal layers on a substrate of a circular cross section. Similar observations were observed for the layers deposited by

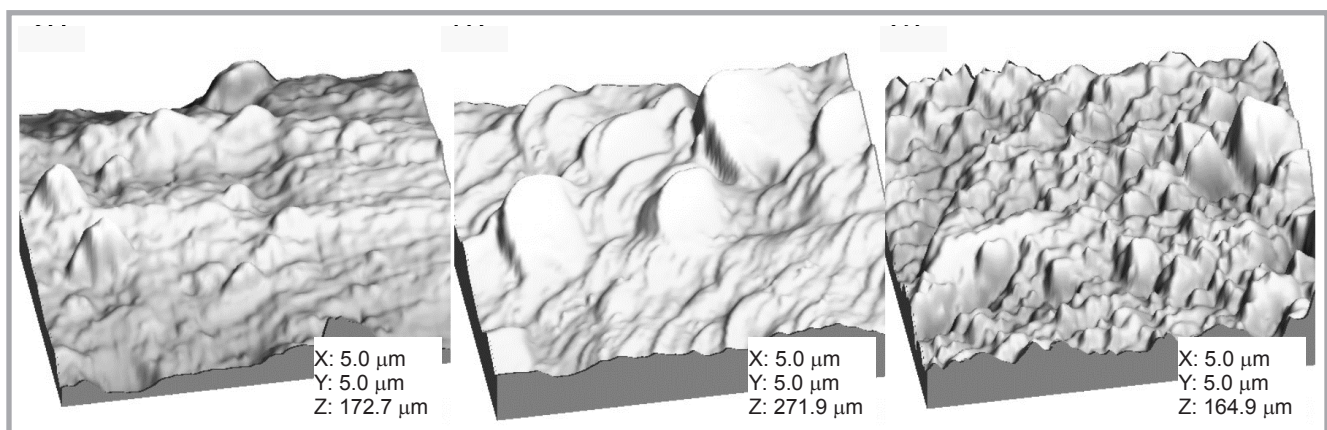


Figure 1. AFM images of the surface topography of the monofilament before (a) and after applying the layer of 400 nm of silver (b) and copper (c).

PVD process on smooth substrates such as foil [34]. Such results were expected when planning to reduce the roughness, but it had to be confirmed in studies to enable accurate observations of the surface topography changes to yarns with metal layers. Based on the results shown in **Table 1**, yarns with metal layers with a thickness of over 200 nm were selected for further testing.

Results of yarns resistance tests

The electroconductive properties of yarns with metal layers were thoroughly tested. These studies demonstrated that the PVD method will give good electrically conductive layers of the yarns processed.

An indicator is the surface resistance of yarn with metal layers. In order to determine whether the layers are stable enough to be used, for example, for a heater in mattresses, yarns modified by PVD technology had to undergo usability testing. The results are summarised in **Tables 2** and **3**.

The monofilament yarn before modification was characterised by a resistance value of 101.9 G Ω , while that of ring yarn was 177.5 G Ω . Both values were measured with the use of the earlier-described test bench. Using the PVD methods for yarn modification, it is possible to obtain good electroconductive properties from several Ω to over a dozen Ω . According to the data in **Tables 2** and **3**, the resistance values for yarns after abrasion and bending tests slightly increased or slight declined. In the abrasion and bending tests, there was a slight loss in the electrical conductive properties of a few ohms.

The lowest increase in the resistance value after the bending test was observed for yarn produced by ring spinning and coated with copper (14.24 Ω), while the biggest growth in this parameter was for monofilament yarn coated with silver (8.13 Ω).

In the abrasion tests, the electrical properties of ring spun yarn with a copper layer showed the least loss (14.73 Ω), while the greatest losses in conductivity were determined for the monofilament yarn coated with silver (8.21 Ω).

After the laundering test, the resistance of all samples studied significantly increases. The biggest loss in electroconductive properties (which means increase

Table 1. Summary of RMS coefficients for metal coatings of silver and copper.

Metal	The thickness of the layer, nm	RMS, nm	Metal	The thickness of the layer, nm	RMS, nm
none	0	22.5	none	0	22.5
Ag	50	31.4	Cu	50	29.6
	100	32.2		100	27.7
	200	14.8		200	19.0
	400	17.7		400	18.1

Table 2. Surface resistance after applying the metal layer on the yarns and after the functional properties tests.

MONOFILAMENT 85 dtex, layer Ag 400 nm, the length of 1 cm					
Lp.		Resistance after modification	Resistance after bending	Resistance after abrasion	Resistance after laundering
1.	Average value, Ω	1.53	8.13	8.21	478.13
2.	Standard deviation, Ω	0.00	0.12	7.77	657.25
MONOFILAMENT 85 dtex, layer Cu 400 nm, the length of 1 cm					
Lp.		Resistance after modification	Resistance after bending	Resistance after abrasion	Resistance after laundering
1.	Average value, Ω	2.05	8.79	4.58	173.24
2.	Standard deviation, Ω	0.003	14.67	2.64	23.50

Table 3. Surface resistance after applying the metal layer on the yarns and after the functional properties tests.

RING yarn 65 tex, layer Ag 400 nm, the length of 1 cm					
Lp.		Resistance after modification	Resistance after bending	Resistance after abrasion	Resistance after laundering
1.	Average value, Ω	6,52	9,24	9,29	207,78
2.	Standard deviation, Ω	0,07	0,73	11,74	47,96
RING yarn 65 tex, layer Cu 400 nm, the length of 1 cm					
Lp.		Resistance after modification	Resistance after bending	Resistance after abrasion	Resistance after laundering
1.	Average value, Ω	13,87	14,24	14,73	405,27
2.	Standard deviation, Ω	0,42	12,83	3,35	72,51

in resistance) was observed for PET monofilaments with silver layer at a value of 478.13 Ω . For the same yarn with the copper layer the smallest loss in electroconductive properties (small increase in resistance) of 173.24 Ω was observed.

Results of thermal imaging tests

Power losses were determined for the current from the minimum 0.1 to 0.5 A for different types of yarns, where the yarn temperature was measured using an infrared camera - Therma Cam E65 (Flir, USA). In particular cases, the temperature rise ΔT on the conductive path exceeded 70 $^{\circ}\text{C}$. This indicated a too high resistance, and a heterogenous structure of the applied metallic coating.

$$\Delta P_p = R_p \cdot I^2 \quad (3)$$

For example, for the ring spun yarn 65 tex with a silver layer (Ag) 400 nm, and with a length of 1 cm, the power losses were 0.65 W, while at the distance of 10 cm - 2.7 W.

$$\Delta P_{p(1\text{ cm})} = 6.52 \cdot 0.1^2 = 0.65 \text{ W}$$

$$\Delta P_{p(10\text{ cm})} = 27 \cdot 0.1^2 = 2.70 \text{ W}$$

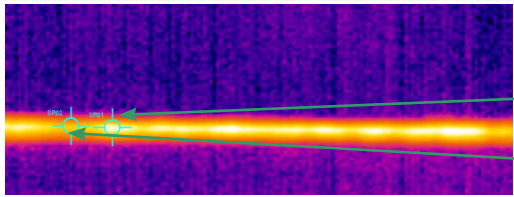
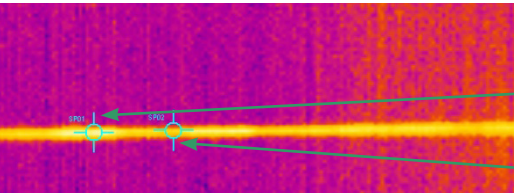
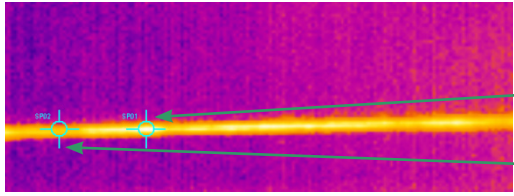
Table 4 presents examples of thermal imaging photos of the test yarns with marked selected temperature points: Sp1 and Sp2. Differences in the temperature values show the heterogeneity of the metal layer applied.

Thermal imaging studies show temperature distributions on the surface of the electroconductive yarns produced. Analysing the temperature distribution, the local temperature increases (white areas) can be determined, reflecting local resistance increases, which are caused by a partial discontinuity of the metal layer applied. According to these data, the continuity or discontinuity of the metal layer on the yarn surface can be qualitatively evaluated. In the cases studied, the unevenness of the metal layer on the cylindrical surface of the yarn could be observed.

Discussion and conclusions

Depending on the future application, there are different kinds of requirements imposed for yarns regarding electrocon-

Table 4. Example of thermographic photos of yarns.

I, A	Ring spun yarn 65 tex, layer Ag 400 nm	
0.1		$T_{max} = 34.8\text{ }^{\circ}\text{C}$ $Sp_1 = 34.1\text{ }^{\circ}\text{C}$ $Sp_2 = 30.4\text{ }^{\circ}\text{C}$
	Monofilament 212 dtex, layer Ag 400 nm	
0.2		$T_{max} = 28.6\text{ }^{\circ}\text{C}$ $Sp_1 = 26.3\text{ }^{\circ}\text{C}$ $Sp_2 = 25.2\text{ }^{\circ}\text{C}$
	Ring spun yarn 65 tex, layer Cu 400 nm	
0.1		$T_{max} = 30.0\text{ }^{\circ}\text{C}$ $Sp_1 = 30.0\text{ }^{\circ}\text{C}$ $Sp_2 = 26.2\text{ }^{\circ}\text{C}$

ductive properties. Therefore, in some cases, the yarns do not necessarily have perfect quality of the metallic coating, an example of which are anti-static products, e.g. different types of mats, aprons and gloves. However, in the case of specialised medical devices, such as electrodes for electrical stimulation, the uniform and homogeneous electro-conductive properties of the yarn surface are required. Otherwise the local discontinuity of the metallised layer could cause, in particular, point temperature increases that could cause local burns on the patient's skin.

In this study PVD technology was successfully adapted to produce a new type of textile that can be applied to a range of hybrid textile products.

In this work the textile products in the form of yarns with metal coatings were characterised by a good level of surface resistance after the modification process and after the functional property tests.

Summing up, the properties of electro-conductivity produced by PVD are characterised by increased conductivity, and stable mechanical properties, sufficient resistance to washing, among others, which determines the range of their applications for electronics in textiles. Modified yarns of that all PVD technology can

be used, for example, as textile heaters or shielding electromagnetic fields.

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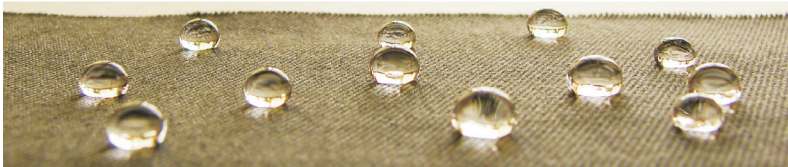
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The Scientific Department of Unconventional Technologies and Textiles specialises in interdisciplinary research on innovative techniques, functional textiles and textile composites including nanotechnologies and surface modification.

Research are performed on modern apparatus, *inter alia*:

- Scanning electron microscope VEGA 3 LMU, Tescan with EDS INCA X-ray microanalyser, Oxford
- Raman InVia Reflex spectrometer, Renishaw
- Vertex 70 FTIR spectrometer with Hyperion 2000 microscope, Brüker
- Differential scanning calorimeter DSC 204 F1 Phenix, Netzsch
- Thermogravimetric analyser TG 209 F1 Libra, Netzsch with FT-IR gas cuvette
- Sigma 701 tensiometer, KSV
- Automatic drop shape analyser DSA 100, Krüss
- PGX goniometer, Fibro Systems
- Particle size analyser Zetasizer Nano ZS, Malvern
- Labcoater LTE-S, Werner Mathis
- Corona discharge activator, Metalchem
- Ultrasonic homogenizer UP 200 st, Hielscher

The equipment was purchased under key project - POIG.01.03.01-00-004/08 Functional nano- and micro textile materials - NANOMITEX, co-financed by the European Union under the European Regional Development Fund and the National Centre for Research and Development, and Project WND-RPLD 03.01.00-001/09 co-financed by the European Union under the European Regional Development Fund and the Ministry of Culture and National Heritage.



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