Effect of Corona Discharge on the Morphology of Polyester Fiber Top Layer

Stefan Brzeziński,[1] Adam Tracz,[2] Stefan Połowiński,[3] Dorota Kowalczyk[1]

[1]*Department of Non-conventional Techniques and Textiles, Textile Research Institute, Gdańska 118, Łódź 90–520, Poland*
[2]*Center of Molecular and Macromolecular Studies, Polish Academy of Science, Şienkiewicza 112, Łódź 90-363, Poland*
[3]*Department of Physical Chemistry of Polymers, Technical University of Łótź, Żeromskiego 116, Łódź 90-543, Poland*

Received 29 September 2009; accepted 4 December 2009
DOI 10.1002/app.31926
Published online 22 February 2010 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The penetration of a woven fabric by corona discharge is of paramount importance for the achievable result of its modification. It was examined how the fiber top layer structure of polyester woven fabric was changed because of its treatment with corona discharge under atmospheric pressure. The corona discharge used was generated with the use of an original generator equipped with a set of multisegmental electrodes that made it possible to obtain a required level of activation energy in successively supplied "portions," which simultaneously prolonged the time of plasma impact. Changes in the topography of fiber top layer were examined by the AFM method. It has been found that this corona discharge can shape the fiber top layer in a qualitatively similar way as in the case of polyester films (characteristic globular structure). It has also been established that the achievable effect of modification is not limited exclusively to the fibers that form the external surface of the fabric activated, but it also affect, though to different extent, the fibers from various areas of the fabric structure. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 116: 3659–3667, 2010

Key words: corona; atomic force microscopy (AFM); fibres; polyester; surface modification

INTRODUCTION

The conventional technologies of chemical modification of fibers and fabrics concern mostly surface processing and are mainly performed in aqueous media. Generally, these processes are very energy- and material-consuming and constitute a source of hardly degradable post-production effluents. As opposed to them, plasma processes are carried out in a gaseous medium, do not use water and do not produce effluents, being also considerably less material-consuming. Particularly great hopes are set on the surface modification of textiles by low-temperature plasma.[1–8] There are two basic types of this plasma: low pressure plasma (LPP) generated and used under a low pressure, mostly 0.1–1.0 h Pa, and atmospheric pressure plasma (APP) generated and used under atmospheric pressure, commonly called atmospheric plasma.

APP, and especially its historically first variant described as corona discharge, has been studied for many years. The high intensity of corona discharge effects on polymeric materials makes it possible to use short treatment durations and consequently its performance in a continuous process. Such a process provides a considerably higher yield than that of batch processes being necessary in the case of LPP.[1,5] These studies were, however, directed mainly toward the finishing of polymer film surface, and now the corona discharge treatment is a commonly used, routine process for the preparation of the surface of these materials for printing, joining, and laminating.[1,9,10] Nevertheless, the effects of corona discharge on the surface of polymeric films and the resultant physical changes in the topography of their top layer and chemical composition are still the object of many studies. Strobel et al.[11] have reported changes in the plasma-treated polypropylene (PP) film in the form of irregularly located blisters with a diameter dependent on the relative humidity within the discharge zone. These changes are attributed to the presence of low-molecular-weight oxidized material (LMWOM) on the water-soluble film surface, with the oxidation degree being evidenced with the increase in O/C ratio, measured by the XPS technique. Similar changes have been observed by Mahlberg et al.[5] investigating the effect of LPP

Correspondence to: A. Tracz (atracz@cbmm.lodz.pl).
Contract grant sponsor: Polish Ministry of Science and Higher Education in 2006–2009; contract grant number: Research Project No. 3 R08 035 01. L.

under oxygen atmosphere. They obtained a surface roughness at a level of 2–10 nm as measured by the AFM microscopy. These studies, aimed at the assessment of possible optimization of film adhesive properties, have allowed the authors to state that the adhesion between two surfaces results from two factors: the size of adherent surface and the attractive forces between the components being joined. O'Hare et al.,[12] using dielectric barrier discharge (DBD), have examined the physical changes in the PP film surface in the nanoscopic scale, founding the presence of a characteristic globular structure, whose roughness depends on the discharge energy used. These authors[13] have also studied the action of corona discharge on a film from poly (ethylene terephthalate) (PET). Using the XPS technique, they have determined the dependence of oxygen-containing groups (phenol, acidic, carbonyl) on the discharge energy used. Using the AFM technique, there were also observed globular structures formed on the PET surface being similar to those obtained on PP film surface.[13] The formation of these structures and their shapes, are due to the fact that the surface energy of LMWOM is higher than that of PP.

The success of the activation of polymer film surface with corona discharge became a stimulus to use it for the modification of textiles, first of all those from synthetic fibers being produced from the same polymers as polymeric films. Films are characterized by a smooth, compact and homogeneous surface, while textiles have heterogeneous and developed surface and a complex three-dimensional structure of fabric, whose characteristics include high macroporosity and microporosity, which significantly affects the results of corona discharge treatment.[1,14–18] Because the specific surface of textiles is very high it is necessary to use considerably higher discharge energies than those for the modification of the film top layer. However, as the discharge energy is increased, the nonuniformity of discharge is also increased at the same time and there appear a high-power brush discharge in the form of plasma channels, commonly called streamers. They bring about local disqualifying thermal damages to the fabrics treated with corona discharge[1,7,14,19,20] which became the main impediment to the use of this technique in the textile industry.

Various modifications of corona discharge generation were proposed to overcome the problem.There should be mentioned dielectric barrier discharge (DBD) generated within the area of low frequencies from 20 to 100 kHz in air as well as in a controlled atmosphere of various neutral carrier gases, mainly He, Ar, and N or their mixtures with air, directly applied[1,6,8,21–29] or in the stream of blowing-in gas (DBDJ)[30] and dielectric coplanar surface barrier discharge (DCSBD).[31] Both of these discharge types, are generated within the area between two isolated electrodes with different shapes. Another variety of atmospheric plasma, is the atmospheric pressure glow discharge (APGD), generated mostly in the medium of helium or nitrogen at a voltage with radio frequency (13.56 and 27.12 MHz), being characterized by a high uniformity and relatively higher penetration across fabric than that of discharge generated with lower frequencies.[7,16,17,32,33]

To obtain the varieties of atmospheric plasma presented, there have been developed special constructions of discharge electrodes, e.g. electrodes coated with ceramic insulation or sealed in a ceramic block, as well as prototypes of new-construction generators.[1,16–19,22–25,34–36]

A very important research problem is the plasma penetration inside fabrics to modify not only the fibers forming the external, exposed to discharge fabric surface but also those inside its structure. This is of paramount importance for the performance effectiveness of the textiles activated.[1,16–19] Poll et al.[18] have studied the penetration of fabrics by plasma generated and applied under various pressure conditions from a low pressure to atmospheric pressure and concluded that the penetration depends on pressure and the size of macropores and micropores in the fabric treated. They have found that the best results can be obtained under pressure from 1 to 100 mbars, while under atmospheric pressure the penetration is impossible, even when the treatment duration is considerably prolonged. Investigating the plasma penetration across the layers of textiles, De Geyter and coworkers[37] have found a significant effect of the process pressure (medium-pressure plasma - 5 kPa). Wang et al.[16,17] have examined the penetration of fabrics by atmospheric glow plasma blown in a stream of He/O_2 as a carrier gas onto the surface of multilayer sets of polyester woven fabrics. They have concluded that the size of macropores in fabrics and the exposure time play the basic role in the penetration: the greater the pore dimensions and the longer treatment time, the better is the penetration.

It should be added, however, that the new developments of special electrodes and generators resulting from research and development works are relatively expensive. Hence, despite important technological results, the interest of textile companies in this regard is limited, which considerably impedes the spreading of the modern, effective and environment friendly treatment of textiles with APP.[1,3]

The difficulty in setting the conditions for the corona treatment of textiles. is due to the fact that two inverse conditions should be fulfilled: the necessity of supplying a high activation energy E_j to the fabric under modification to obtain expected modification

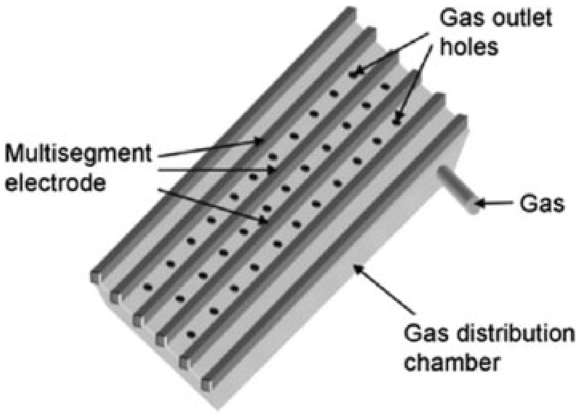

Figure 1 Scheme of multisegmental electrode.[20]

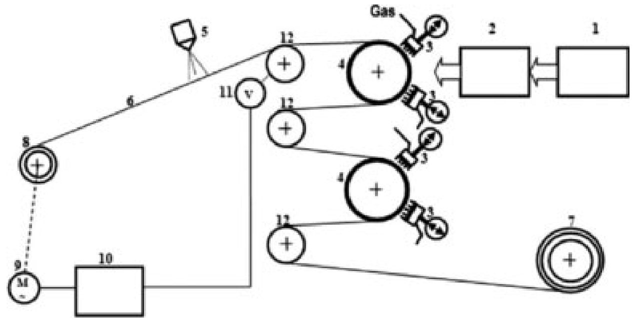

Figure 2 Block diagram of the experimental generator for the treatment of textiles with corona discharge by a continuous method[19]; 1: generator, 2: high-voltage transformer, 3: five-segment discharge electrode, 4: cylindrical electrode, 5: nozzle for aerosol application, 6: fabric to be treated, 7: rewinder, 8: winder, 9: drive motor of rewinder, 10: frequency converter to control the rotational speed of the winder drive motor, 11: sensor of the fabric linear speed, 12: auxiliary roller.

effects, which requires a high power of the generated discharge, and on the other hand, the energy of destructive steamers should be reduced to a level that eliminate the hazard of local thermal damages to textiles and provides possibly the highest uniformity of the discharge.

In this work, we have decided to solve the problem by dividing the energy dose of corona discharge required for the modification, whose quantified measure is E_j [J/cm^2], into series "n" of successively proportioned partial energy doses, E_{jn}, whose total and, from the nature of things, prolonged action on fiber/fabric provides the expected level of their plasma modification

$$E_j = n * E_{jn}$$

At the same time, such "component" energy doses of discharge can be low enough to provide the indispensable high uniformity of discharge and to considerably reduce the devastating effect of streamers. Moreover, such a successive metering of the required activation energy makes it possible to considerably prolong the action of discharge on the fabric causing no damage to it. Based on the experience of other researchers,[16,17] one can assume that such a prolonged treatment with corona discharge should beneficially affect its penetration into the fabric structure and modify not only the fibers of the external exposed side of the activated fabric but also those in its deeper layers.

EXPERIMENTAL

Equipment used for the treatment of textiles with corona discharges

To accomplish the concept of dividing the energy dose of corona discharge into series of successively proportioned partial energy doses there was designed a set of multisegmental electrodes, which is characterized by appropriately arranged orifices for supplying a gas that forms the discharge medium. The construction principle of this electrode is shown in Figure 1.[20]The above construction of multisegmental electrodes was used in the designed experimental generator for the treatment of textile with corona discharge by a continuous method (Fig. 2). Its basic advantage is its suitability for use in a process similar to real industrial conditions.

Materials

The woven fabric used was a commercially produced fabric (WISTIL S.A., Kalisz, Poland) from

TABLE I
Characteristics of PET Woven Fabric

Warp		Weft		Fabric weight (areal density) g/m^2
Yarn characteristics	Threads/10 cm	Yarn characteristics	Threads/10 cm	
84 dtex f48 t0 spot tacked	390	150 dtex f216 t0	320	89

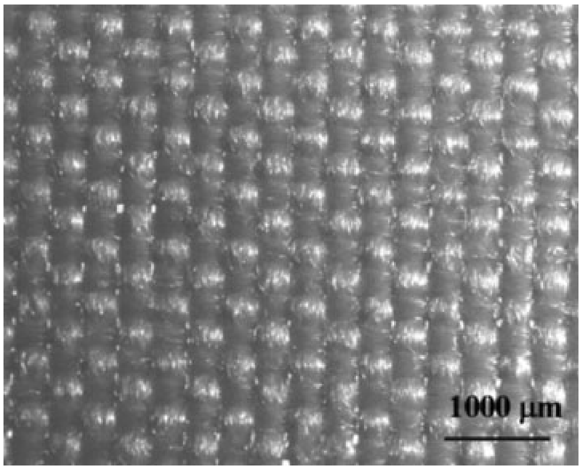

Figure 3 Fabric image under an optical microscope.

twistless multifilament polyester yarns with the characteristics given in Table I.

The woven fabric shown in Figure 3 with a plain weave 1/1 and a thickness of 0.19 mm, is characterized by a very compact structure and a high cover factor exceeding 99% and small interthread clearances (macroporosity); the average size of pores ranges from 30 to 50 μm.

Before plasma treatment the PET fabric was washed and thermal stabilized at 190°C for 20 s.

Microscopic examination of the fiber surface

The microscopic examinations of the fiber surface topography were performed in oscillatory mode using an atomic force microscope (AFM), Nanoscope IIIa Digital Instr., (Santa Barbara - USA). In addition to the topographic image, the image of amplitude or phase contrast was recorded at the same time. Rectangular siliceous probes with a radius of curvature R $\sim$ 15 nm, (Nano Probe - Germany) were used. All the examinations were carried out under the air atmosphere at room temperature, with a scanning frequency below 2 Hz. Images with a resolution of 512 $\times$ 512 lines were recorded.

EDX analysis

The determination of the oxidation number of top layer (and near-surface layer) of fibers, expressed by the O/C ratio corresponding to the oxygen quantity defined as a quotient of oxygen atoms weight and carbon atoms weight in this layer, was performed by the method of energy dispersive x-ray micro-analysis (EDX).[14,38] by means of an EDX micro-analyzer, system ISIS from Oxford Instruments. The examination and observation of the topography of the surfaces under investigation were performed by means of a scanning electron microscope Vega TS 5135 MM from Tescan. The resolution of the x-ray micro-analysis was about 0,5 μm. The measurements were repeated in at least 5 independent areas of the fabric sample tested and the results were arithmetic averages of these determinations. The content of elements in the top layer tested was calculated using the SEM Quant program with ZAF correction procedure allowing one to obtain a measurement accuracy of 1%.[14]

Determination of acidic group content

The content of acid groups was determined by the comparative method. To that end each of the samples under testing, with a weight of 1 g, was flooded with 50 mL of water and then 5 mL of 0.01 m NaOH solution was added to it. After 30 min, the solution was made up to a volume of 100 mL with distilled water. The pH-metric titration of NaOH with 0.01 *M* HCl solution was carried out with the use of a glass electrode. Based on the titration curves obtained, the number of acid groups per 1 g of the ample weight was estimated.

Measurements of contact angle and free surface energy

The contact angle of woven fabrics was determined by the dynamic method using a Sigma 701 tensiometer from KSVInstruments. (Finland). This method consists in recording the force acting on the sample tested being immersed in and emerged from the measurement liquid at the same rate. Five measurements were performed for each sample. Based on the contact angle values obtained, the free surface energy was calculated by the Wu method (harmonic mean).[14]

Capillarity measurement

Capillarity measurement is a commonly accepted method for the determination of wettability and the degree of plasma modification of textiles.[7,14,16–18] This method, described in PN-67/P-04633,[34] consists in determining the height of the colored liquid ascent in a fabric strip whose lower end is immersed in this liquid.[14]

RESULTS AND DISCUSSION

Optimized activation conditions

The newly designed set of multisegmental electrodes, allows one to obtain a considerable improvement in the uniformity of corona discharge and significant decrease in the streamer energy in the generation of discharge with a high activation energy E_j, considerably higher than that obtainable

TABLE II
Selected Surface Properties of PET Woven Fabric with a Characteristics Given in Table I

	Free surface energy [J/cm^2]	Capillarity [cm]	Contact angle (water) [deg]	Acid groups content [mol/g]	O/C ratio (EDX)
Before activation	38.45	5.45	63.35	0.00	0.76
After activation 75.6 [J/cm^2]	48.16	10.65	51.03	1.7×10^{-5}	0.80

with a single-segment electrode. All in all, there were obtained expected results of the surface modification of fibers/fabrics with the elimination of the hazard of fabric strength loss and local thermal damages to the corona activated textiles.

From the performed investigations, it follows that to obtain the best results of activation, it is necessary to adapt the process conditions of corona discharge treatment to the characteristics of fabrics: type of fibers, yarn and flat fabric structures as well as to the characteristics of the generator used and expected technological effects of the treatment.

There was proposed a concept of so-called optimized activation energy E_{jopt} and a procedure of its determination. It expresses the maximal dose of activation energy supplied per a unit of fabric surface that makes it possible, under specified conditions (type of fiber, yarn and fabric, generator characteristics) to obtain the possibly higher extent of surface modification of fibers, without time thermal damages to textiles resulting in deterioration in the strength parameters of fabrics.

Thus, E_{jopt} is a relative index, dependent on both the characteristics of fabrics subjected to activation and the characteristic of discharge generator as well as the technological purpose of corona discharge activation and the expected modification effect.

The optimized unit doses of activation energy E_{jopt} (for the woven fabrics under modification and the characteristics of the experimental generator) and the parameters describing the surface properties of woven fabrics obtained under these conditions are listed in Table II.[20]

Morphology

The corona discharge treatment of fabrics under optimized conditions resulted also in considerable changes in the microtopography and nanotopography of PET fiber top layer[20] as shown in AFM images (Fig. 4). The surface of unmodified fibers is smooth and the roughness of surface (root mean square (RMS) parameter) does not exceed 2 nm [Fig. 4(a)]. After fabric treatment with corona discharge, the fiber surface shows a characteristic globular structure [Fig. 4(b)], analogos to that described by O'Hare et al.[13] for the polyester film modified with corona discharge. It should be stressed that under optimized conditions the corona discharge treatment of fabrics brings about changes in their surface topography in a nanoscope scale (RMS = 13 nm). Therefore, AMF microscopy showing the images of surface without metallic layer covering allows considerably better assessment of the surface activation effects than that made by SEM, even at low discharge power. The vertical resolution of AFM microscope is considerably higher than that of SEM, and besides the surfaces examined are not dusted with gold.

The AFM images, especially those of phase contrast, prove that on the fiber surface are developed granules with various sizes, while between greater granules appear considerably smaller ones [Fig. 4(b,c)]. The granular morphology of surface modified by corona discharge is ascribed to the dewetting process of the layer of oligomeric polar products of degradation.[14,15,39] Washing down the activated surface with water or water-alcohol mixture results in to the removal of oligomers and so it can bring about considerable changes in the topography of top layer of the modified polymer. In their previous studies, O'Hare et al. have shown that the granules formed as a result of the PET film modification with corona discharge plasma can be completely removed.[13] The smoother surface free from granules was, however, different from the unmodified surface. This indicates that the granules formed by the plasma modification under the conditions used by these authors were created from noncross-linked, polar, soluble products of degradation; therefore, the effect of strong surface granulation was not a durable result.

The image of PET fiber surface activated under optimized conditions (E_{jopt}), after washing down with water-ethanol mixture (1 : 1) is shown in Figure 4(c). As follows from the comparison of the images shown in Figure 4(b,c), the granulation does not disappear after washing down, but it becomes even more visible. The surface roughness (root mean square parameter RMS) increases after washing from 13 nm to 18 nm, which suggests that the low-molecular-weight soluble products of degradation filled the surface between granules. This results indicates that the considerable roughness of PET fiber surface, obtained with the use of our generator is a durable effect and resistant to washing. Thus, the granules observed by us are formed from crosslinked

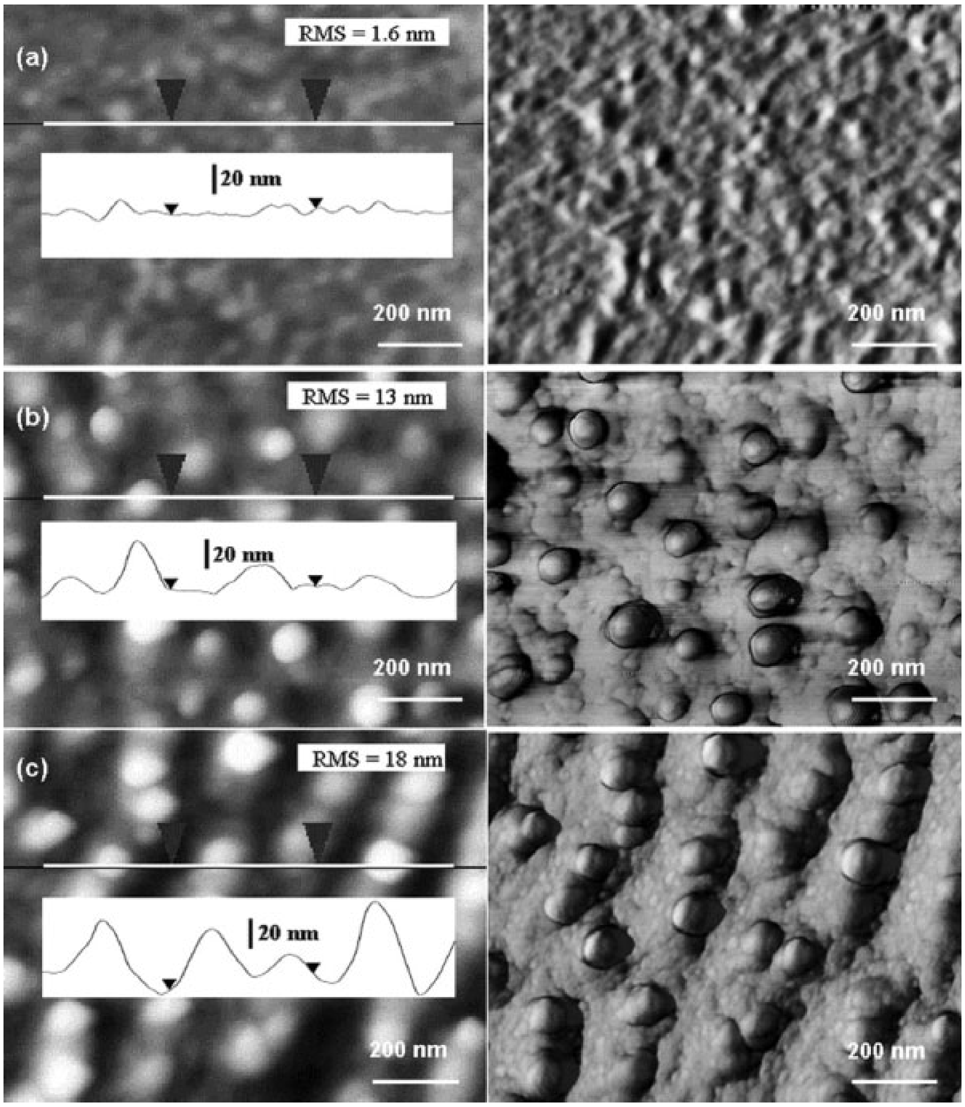

Figure 4 (a) AFM images (left side: topography, right side: phase contrast) and the profiles of PET fiber surface: (a) – before modification, RMS = 1.6 nm, (b) – after corona discharge modification under conditions: E_{jopt} (E_j = 75.6 J/cm^2), RMS = 13 nm, (c) – after modification and rinsing off in the mixture of water and methanol, RMS = 18 nm.

products of macromolecule degradation, whereas the distinct surface roughness (in various scales) results from the plasma etching process.

From the presented test results of the nano-topography changes in polyester fibers brought about by corona discharge (under optimized conditions, E_{jopt}) it results that the morphological changes in the top layer of fibers treated with corona discharge are qualitatively similar to those observed in the case of films made from similar polymers. This indicates a qualitative similarity between physico-chemical processes taking place on the surface of polymeric materials regardless of the material form (film vs. woven fabric).

The relatively long time of corona discharge treatment, obtained with the use of multisegmental electrode set, amounting, for the PET woven fabric tested and conditions E_{jopt}, to about 33 s, in comparison to about 1 s in the case of a single-segment electrode, should bring about changes in the penetration of fabrics by corona discharge plasma.

For that reason there were performed examinations of the top layer topography of fibers derived from different fabric areas: from the external surface exposed to corona discharge, external surface on the other side of fabric and areas in the middle of fabric. The woven fabric, from which fibers were taken for AFM analysis, was treated by corona discharge under optimized conditions (E_{jopt}), and the fiber withdrawing was facilitated by the fabric structure, twistless yarns. The selection of fiber locations and positions in three fabric areas are shown in Figure 5.

It should be stressed that all the AFM examinations concerned the fiber fragments present on the fabric surface from the side of nonisolated metallic electrode, thus fibers from area I shown in Figure 5. Thus it is expedient to assess the modification degree of the surface of fiber fragments on the other side of fabric (from the side of grounded electrode, area II). Moreover, for the sake of proper interpretation of the changes in wetability of fabrics modified with corona discharge, tested by the method of capillarity measurement, it is especially important to find out whether the surface of fibers "covered" by other fibers are also modified, e.g. at the points of thread interlacing (area III).

To uncover the fibers at the points of thread interlacing that during the activation of fabric in the zone of corona discharge were "covered" (area III), one of the threads was taken out from a small sample of the modified woven fabric. The spot, from which the single thread was taken out, is marked with a discontinuous line in the SEM images of a fabric fragment shown in Figure 6.

After the preliminary assessment made by means of SEM, the examinations were continued using AFM. Under the control of the camera recording the image of fabric fragment with the thread removed, the microscope tip could be located over the fibers from fabric surface in area I and over fibers from area III, which, during the treatment of fabric with corona discharge, was "covered" by the thread removed afterward.

The AFM images of the top layers of fibers derived from three differently localized areas during modification are shown in Figure 7. The surfaces of fibers in all the three areas have been modified. The most important discovery is that the top layer of fibers at the fabric bottom [Fig. 7(b)] and even that in area III "covered" during fabric activation, have been modified as confirmed by the characteristic globular structure [Fig. 7(c)]. The roughness of fiber surface in area III is small and does not exceed 3 nm and individual granules are considerably flatter. The crosswise dimensions of the greatest granules amount to about 250 nm, while they protrude only about 10 nm from the "bedding". Thus, it is clear why one cannot depict the surface with such a granulation by means of SEM.

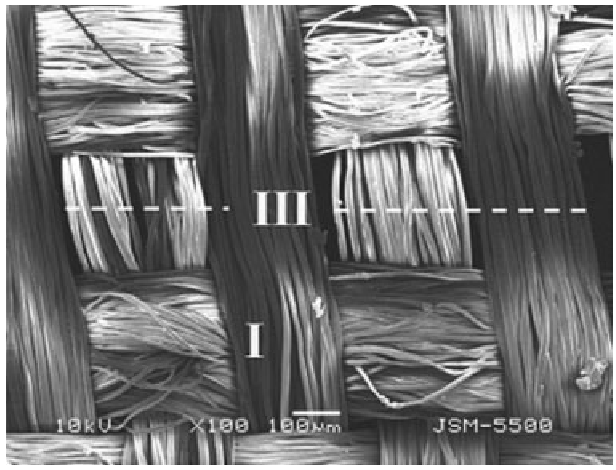

Figure 6 SEM images of PET fabric fragment after modification with corona discharge under optimized conditions (E_{jopt}, treatment time: $t = 33$ s); the discontinuous line indicates the spot, from which a single thread was taken out. I and III areas as in Figure 5.

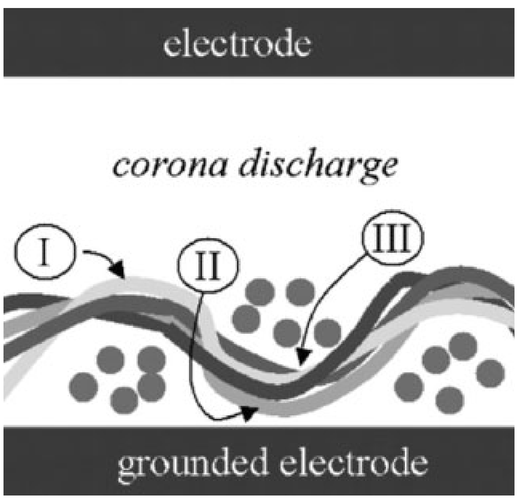

Figure 5 Schematic cross-section of woven fabric in the zone of corona discharge. Lines represent fibers that form the thread of weft and circles: cross-sections of fibers that form warp. There are three distinct areas: **I**—external fibers on the fabric surface from the side of the upper electrode; **II**—external fibers on the other side of fabric sliding on the grounded electrode; **III**—fibers at the thread interlacing "covered" by the upper thread.

The results obtained indicate that the surface of all fibers in the woven fabric treated with corona discharge is modified, though to various extents depending on fiber location. This discovery is of significant cognitive and practical importance.

It has been found that fibers derived from various fabric areas show characteristic changes in the topography of fiber top layer and its globular structure. This concerns also the fibers from areas III being completely covered during the fabric activation with corona discharge. These changes, however, show significant quantitative differences resulting from different degrees of accessibility of fibers from the areas tested to the penetrating plasma, which is clear considering the very compact structure of woven fabrics with small-size pores.

The results obtained are confirmed by the reports of other researchers who, however, use completely different type of atmospheric plasma,[16,17] which owing to its nature is characterized by a high

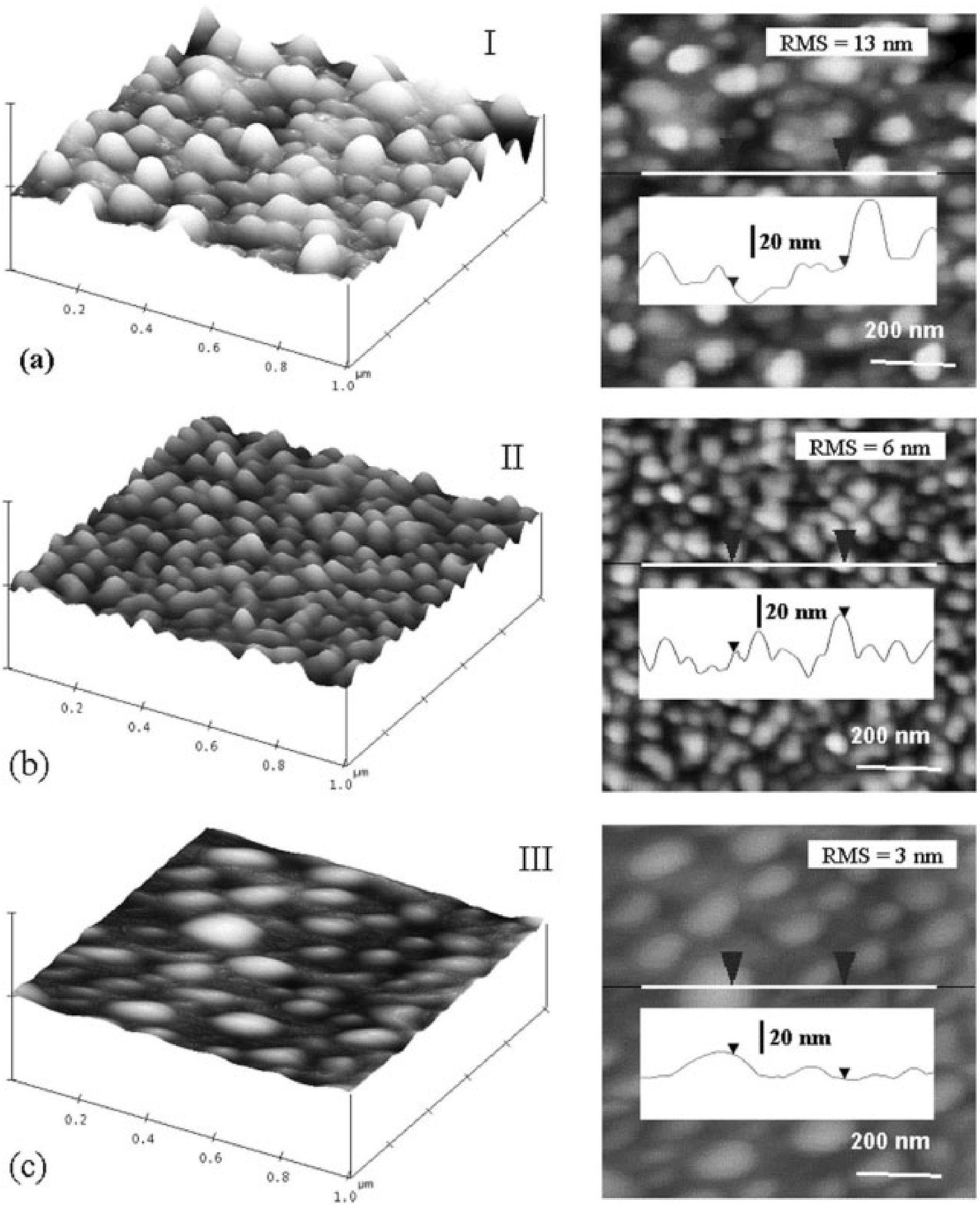

Figure 7 AFM images and corresponding profiles of surface section of fibers from various areas: (a) area I, (b) area II, and (c) area III.

penetrating capability; moreover, it is forced in to the fabric structure with the stream of He/O_2.

Chemical changes

The composition of top layers of fibers from areas I and II was determined using the EDX technique. The results of chemical composition confirmed those of the physical changes in fiber nanotopography. The following values of the O/C ratio with the accuracy of 0.01 were obtained:

- fibers from the upper fabric surface O/C = 0.82
- fibers from the lower fabric surface O/C = 0.79
- fibers from unactivated fabric O/C = 0.76 (upper side = lower side)

This indicates that the oxidation degree of the fiber top layer from the upper exposed fabric surface, measured with the O/C ratio, has increased by 0.06, while that of the inaccessible lower surface by 0.03. This and the results of AFM examinations confirms the modification of the fibers from area II. The results of AFM examinations as well as those of the determination of the oxidation degree of fiber top layer by the EDX method indicate that the surface of all the fibers in woven fabrics has been modified, tough to different extent dependent on the fiber location in the fabric structure.

CONCLUSSIONS

1. The treatment of PET woven fabrics with corona discharge generated by the multisegmental electrode set designed and made for this purpose makes it possible to obtain a high degree of physical and chemical modification of the fiber top layer and consequently to improve the surface properties of woven fabrics.
2. This modification, performed with the use of corona discharge under optimized conditions of successive dosing the partial activation energies, $E_j = n * E_{jn}$, for appropriately longer time "t" (E_{jopt}), results in physical changes in the nanotopography of the fiber top layer showing a characteristic globular structure. This structure is analogous to that found in the case of polyester film treated with corona discharge.
3. Because of the very high porosity of textiles consisting of variously ordered filaments, the specific surface of fiber/fabric being subject to modification is incomparably larger than that of a film. This porosity, including interfilament and interthread cavities or clearances, causes a considerable portion of the supplied corona discharge energy to permeate through the structure of fabric, while the plasma "washes down" fiber surfaces inside the fabric.
4. The implementation of the conception of successive dosing partial activation energies, $E_j = n * E_{jn}$, for appropriately longer time "t," by means of the newly designed multisegmental electrode set, makes it possible for the plasma to penetrate into textile fabric and to modify fibers in various areas of the fabric structure, though to different extents.

Lukasz Pietrzak is acknowledged for SEM measurements.

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