Modification of textile properties using plasma

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Overview

An overview of the published investigations undertaken in the field of the target applications (modification) of the properties of textile materials using different types of plasma and the plasma in combination with other physical and chemical agents is provided. The emphasis lies on the properties which are the consequence of the characteristics of textile surface materials (hydrophilicity, hydrophobicity, antistatic property, electrical conductivity, multifunctionality). After explaining plasma phenomenology as a special state of matter, the possibilities of generating and obtaining plasma are described. The classification of plasmas according to different criteria is explained. The plasmas suitable for the treatment of textile materials are emphasized and their basic parameters relevant for the implementation of material treatments are explained. Numerous investigations described in many papers over the last ten years or so are shown according to the type of textile materials on which they were performed (fibers, yarns and fabrics).

***Key words**: textile materials, plasma, plasma treatment, textile surface, modification of properties, multifunctionality, ecological processes*

1. Introduction

Due to the increasingly stringent environmental requirements set in modern textile technology processes, increasing importance is attached to treatment by agents that are acceptable from an environmental point of view. In this sense, there has been more research related to the use of plasma as an environmentally friendly physical agent. Although known from before, a special interest in the application of plasma technology in the field of textiles has been recorded in the last ten years, especially in the processes of pre-treatment and finishing of textile materials for the purpose of obtaining multifunctional textile products. In this sense, the emphasis of modern treatment is focused on obtaining favorable effects by modifications of the fiber surface (textiles), which ultimately contribute to the overall quality of the textile material. Surface treatments with plasma, ozone, biopolymers, etc. which are being researched and implemented nowadays for the purpose of modifying the properties of textile materials, are, in addition to being environmentally friendly, also more energy efficient compared to conventional finishing methods. The development and commercialization of plasma technology in the design and production of modern textile materials contributes to reducing the use of chemical agents, which is gaining increasing importance - both from an environmental and economic point of view.

Research on the possibility of achieving multifunctional properties of textile materials by treatment using plasma as one of the physical agents, is carried out at the TTF under the scientific project "*Multifunctional textile materials for personal protection*" (led by prof. E. Pezelj, PhD). The project is financed by the Ministry of Science, Education and Sports, and the necessary equipment has been purchased with donated funds from the Ministry, the scientific project funds and with some assistance from TTF.

2. Plasma as a physico-chemical agent for modifying the properties of textiles

2.1. What is plasma?

The word plasma comes from the Greek word *plasma*, which in free translation means *self-shaping material*. In physics, plasma is defined as an ionized (or partially ionized) gas, mostly composed of free charge carrier particles, such as ions and electrons. In essence, plasma is neutral and is considered to be the fourth state of substance.

In nature, plasma is the most common physical state encompassing as much as 99% of visible matter in space, and known examples of plasma include: *Aurora Borealis (Northern dawn), Aurora Australis, Van Allen Belt (radiation zones), the Sun, stars, Earth's ozone layer, ionosphere*, etc. However, natural plasma cannot be used at the current stage of technical development. For use in research, engineering and industry, plasma needs to be produced, which is usually done by electrical gas discharge.

The composition of plasma depends on the gas used in the formation of the plasma and on the chemical reactions in the formation of the plasma, forming free electrons, ions, molecules, atoms, UV photons, metastable particles, radicals, i.e. excited neutral and charged particles that participate in these reactions. Of course, such particles during plasma treatment can cause modification of the substrate surface [1, 2].

It is known that materials are composed of atoms and molecules and that with increasing temperature they change state from solid to liquid and further to gaseous state, which is schematically shown in Fig.1. Simply put, it can be said that as the temperature of solid materials increases, there is a more intense movement of parts of molecules, so that at some point they begin to leave their place determined by potential energy, the basic structure is destroyed and the transition to liquid state occurs. By further heating the material, the kinetic energy of the molecules becomes higher than their potential energy and they become freer, leading to separation into atoms and collisions with each other, and transition to a gaseous state. As the temperature rises to more than a few thousand degrees, the collisions of atoms become more intense, so electrons are released from the atomic structure, resulting in the formation of charge-carrying particles - negatively charged electrons and positively charged ions, i.e. the destruction of molecules creates positive and negative atomic groups. Electrically charged particles in motion create electric and magnetic fields, which provide the energy needed for further ionization of atoms, i.e. a state of almost completely ionized matter - plasma. In general, it can be said that plasma is formed by bringing energy to gas [2].

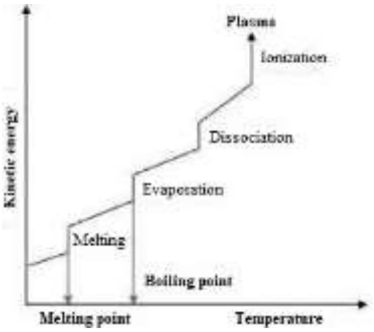


Figure 1. Schematic representation of the transition of the state of matter [2]

2.2. Plasma shapes and types

Plasma shapes and types are numerous and plasmas can be classified according to several criteria. Plasmas occurring in nature differ primarily in density (an order of magnitude of more than 10 exponents), and accordingly the differences in physical properties are extreme as well. A key parameter for distinguishing technical plasmas is the pressure of the neutral gas in which the ionized particles move relative to atmospheric pressure. According to this criterion, plasmas are classified into *low-pressure*, *high-pressure* and *atmospheric pressure* plasmas (*atmospheric plasmas*).

According to the temperature at which they are applied, plasmas are divided into *cold* and *hot plasmas*, Fig.2.

Low-pressure plasma is an example of cold or low-temperature plasma, which is also an example of non-equilibrium plasma, which means that electrons in plasma have a much higher temperature than the temperature of heavier ion particles in plasma. With appropriate external gas excitation in such a plasma, it is possible to achieve conditions in which the gas temperature is close to room temperature, while the effective temperature and kinetic energy of the electron are much higher and large enough to maintain plasma and initiate plasma-chemical reactions [4]. The temperature of the electrons T_e is of the order of magnitude 10^4 to 10^5K, while the temperature of heavy particles of ions and other particles of molecules, atoms and free radicals is close to room temperature, so that the gas hardly heats up and maintains room temperature. Therefore, such plasma is particularly suitable for the treatment of thermally sensitive materials such as most textile materials.

In *equilibrium plasma*s there is a thermal equilibrium, i.e. the temperature of electrons and heavier ion particles is equal and usually high, and the kinetic energy of all particles is high. In a surface treatment application, such plasmas would be significantly more efficient in the production of radicals and the flow of ions coming to the surface of the material. However,

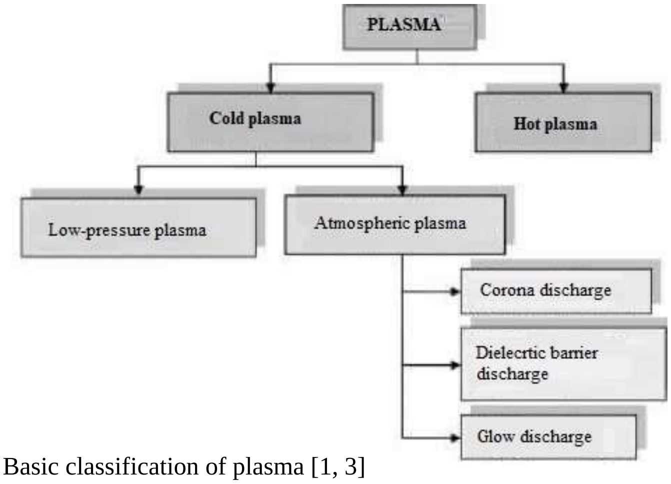
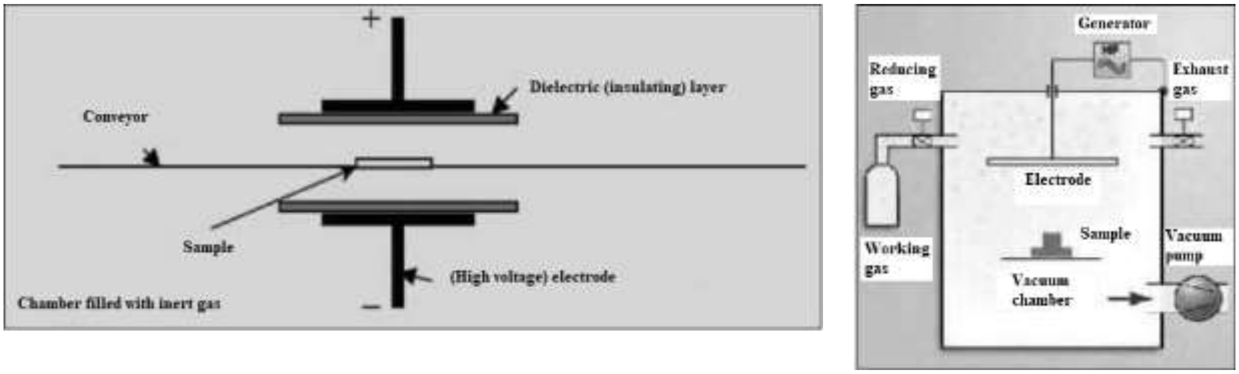


Figure 2 Basic classification of plasma [1, 3]

since they develop a high temperature due to the high degree of ionization, it is very difficult to control the energy of particles and thus optimize the production conditions of specific radicals, and it is impossible to process thermally unstable materials [4]. Such hot plasma is not suitable for textile applications, but is used in welding, thermal spraying, metal cutting and as a method for determining metals in water analysis.

Cold plasma, both *low-pressure plasma* and *atmospheric plasma*, are suitable for textile applications. Low-pressure plasmas are obtained in a diluted gas whose pressure is significantly lower than atmospheric pressure, while atmospheric plasma is produced at normal atmospheric pressure. Therefore, a vacuum chamber or a vacuum pump is not required to obtain atmospheric plasma, while such equipment is necessary in the production of low-pressure plasma. Fig. 3 schematically shows these two basic plasma systems. Both systems have a wide range of applications and can be used to modify the surface properties of organic and inorganic substrates (polymers, paper, wood, foil, foam, nonwoven and woven textiles), whether used in pre-treatment of materials or treatment in combination with various other means (solid inorganic and organic particles) [3,7].



a) atmospheric plasma (Dielectric barrier discharge) *b) low-pressure plasma*

Figure 3. Schematic diagram of the plasma system [7]

Plasma at atmospheric pressure can be obtained in several ways. The first method is *corona discharge*, in which the electric discharge is caused by an electric field of high frequency or voltage, and the surface of the treated substrate is exposed to the direct action of corona. Corona treatment is the oldest and most commonly used technique of plasma surface treatment of materials, which acts at atmospheric pressure and with the surrounding air as a working gas. In principle, this system is also suitable for use in the textile industry (width, speed, temperature), but the type of plasma produced still cannot provide the range of requirements for functional properties required for modern textile materials. In addition, the small distance between the electrodes (up to about 1 mm) makes the system unsuitable for treating thicker materials, and it cannot even ensure sufficient uniformity of treatment [8], therefore its application is limited.

In addition to corona discharge, two other discharge methods are used to obtain atmospheric plasma. Those are: *dielectric barrier discharge (*DBD); *silent discharge (*SD), schematically shown in Figure 3a, as well as *glow discharge (*APGD).

Glow discharge at atmospheric pressure is characterized as a uniform, homogeneous and stable type of discharge, most commonly applied in gases such as helium (He) and argon (Ar), and sometimes nitrogen (N_2).

This method of discharge is an alternative uniform source of cold plasma, which also has an advantage over low-pressure plasma because it operates without a vacuum, i.e. at atmospheric pressure. All three methods of discharge at atmospheric pressure can be used for the treatment of textile materials [3-6].

The low-pressure plasma system consists of a vacuum chamber (Fig. 3b) in which the electrode and supports are located depending on the type of sample, since it is a discontinuous treatment of the material (sample size is determined by the size of the supports) [2,7]. It is described as a controlled and reproducible plasma treatment technique which, unlike atmospheric plasma, requires a vacuum pump to achieve low pressures ranging from 0.01 to 1 mbar. Excitation by an electromagnetic field at low pressure inside a vacuum chamber causes the acceleration of free electrons and when their kinetic energy is large enough to carry out plasma reactions such as ionization, fragmentation and excitation of matter, plasma is formed. This condition is noticed by the appearance of light (from light blue to purple). Atoms and molecules are ionized, excited and fragmented, and form a highly reactive gas mixture that physicochemically reacts with the sample during the treatment of the material, resulting in changes in the surface properties of the treated material.

The types of changes and the obtained effects primarily depend on the process gas and its physicochemical properties, but also on the characteristics of the substrate and the treatment conditions.

2.3. Reactions and plasma action mechanism

In order for plasma to form, it is necessary to ensure the transfer of energy from an external source, where the basic collision between electrons can be described by the reactions shown in Table 1 [2].

Table l. Plasma formation reactions

Excitation:	$A + e -> A^{*} + e$
Ionization:	$A + e -> A^{+} + 2e$
Dissociation:	$M + e -> M\bullet + \bullet M' + e$
Dissociative ionization:	$M + e -> M\bullet + \bullet M'^{+} + 2e$ $M + e -> M\bullet + \bullet M'$
Recombination:	$M^{+}+e+S -> M+S$
Dissociative recombination:	$M^{+}+e -> M\bullet + \bullet M'$
Recombination as a consequence of photon radiation:	$A^{+} + e -> A + hv$

Plasma affects the surface of the substrate chemically and physically, with the reactions between the plasma and the surface depending on the type of gas used and its chemical properties. Textile materials subjected to such treatments undergo chemical and physical transformations related to chemical changes in the surface layer, changes in the structure of the surface layer and changes in the physical properties of the surface layer. Plasma creates a high density of free radicals during the dissociation of molecules during electron collisions and photochemical processes. This causes the destruction of chemical bonds in the polymer surface of the fiber resulting in the formation of new chemical entities. The action of plasma on the surface of fibers and polymers results in the creation of new functional groups such as –OH, –C=O, –COOH, which affect the improvement of the wettability of fabrics (hydrophilic effect), and can be active centers for graft polymerization of different molecules [3].

Therefore, plasma is primarily used for surface treatment of materials, because its action modifies only the surface properties to a layer thickness of only a few tens of nanometers, leaving the basic properties of the material almost unchanged. In this way, selective modification of fiber properties is achieved, e.g. it can affect the ability to wet and dye, adhesion characteristics, etc., which can hardly be achieved by classical chemical processes without affecting the basic properties of such processed fibers [4]. In general, the action of plasma on the surface of a textile material can be roughly described through four groups of processes, Fig.4. [3,7]:

- Plasma cleaning of the surface,
- Plasma ablation or etching of the surface,
- Surface activation, plasma modification,
- Deposition, plasma polymerization.

Fig. 4 schematically shows these four types of actions and their effects in comparison with the untreated surface of the material.

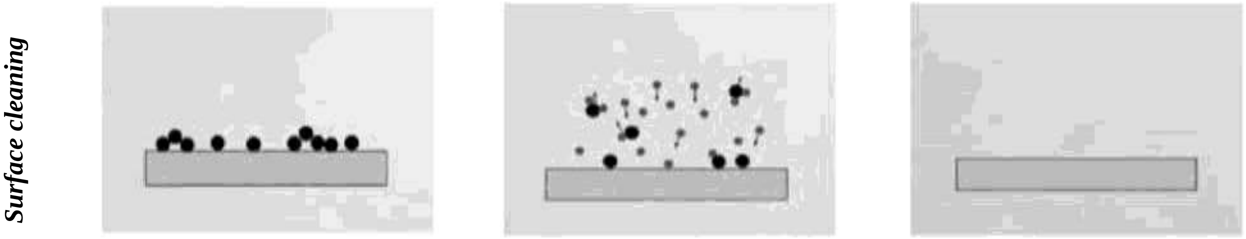
Plasma surface cleaning and ablation or etching are most often used in electronics, semiconductor industry, optical industry and in metal and ceramic cleaning [1,7].

The mechanism of plasma action is in the removal of a thin organic layer from the treated surfaces. The action of plasma during cleaning and ablation or etching of the surface of polymeric materials leads to the breaking of covalent bonds in the polymer chain. Such surface treatment with plasma in the field of textiles is used for sterilization and destarching of textile products, especially by using atmospheric plasma. The application of atmospheric plasma is also possible in the wastewater treatment processes of the textile industry.

The effects of plasma in terms of surface activation and modification are particularly interesting and find application in medical technology, the automotive industry and the plastics processing industry, as well as in the field of textiles [1,7]. In doing so, reactions occur between chemical groups on the surface of the substrate and chemical particles in the plasma, whereby new functional groups are formed on the surface of the substrate. With this method of plasma application in the field of textiles, it is possible to achieve an improvement in the wettability, i.e. hydrophilicity, but also hydrophobicity of textile materials.

The use of plasma in the modification of materials by applying organic and inorganic particles to the surface of the material, i.e. by polymerization of the appropriate agent in the surface layer with the participation of plasma, is becoming increasingly important. In this way, for example, permanent improvements in resistance to scratching, fogging, corrosion, soiling, and improvements in hydrophobicity, biocompatibility, adhesion during layering, protective layering, etc. can be achieved. This type of plasma application is particularly present in medical technology and the metal industry, but in textile technology as well [1,7].

Plasma polymerization [8] enables the application of thin layers of various agents (such as fluorocarbons, hydrocarbons, organosilicones, ceramic coatings, etc.) on all types of textile substrates. This requires the use of a monomeric gas or vapor, which contains atoms such as carbon, silicon, sulfur in the working gas. In the field of textiles, the application of plasma polymerization is used for surface modification in order to obtain textiles resistant to combustion, to achieve antimicrobial properties, and as a dry and environmentally friendly technology shows certain advantages over the corresponding wet treatment.



Untreated contaminated material surface

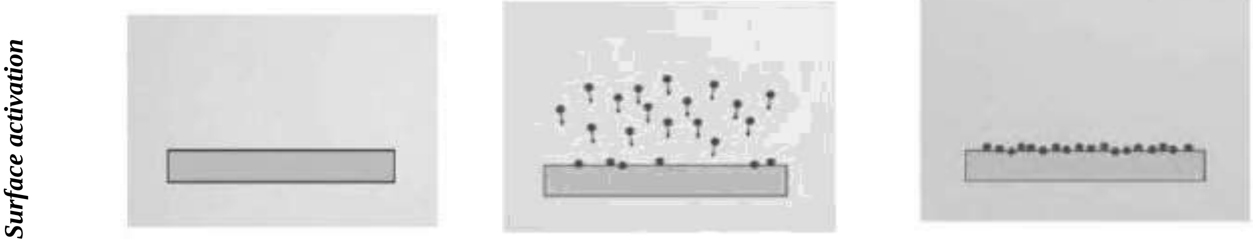
Plasma treatment (e.g. O_2)

Material surface after plasma treatment

Material surface coated with silicone and a layer of e.g. chromium

Plasma treatment

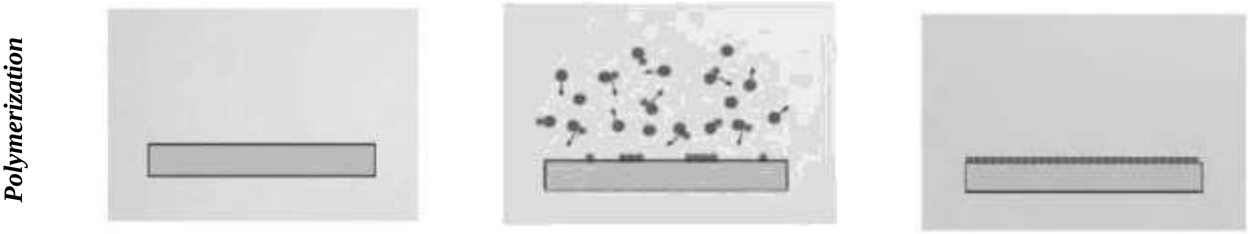
Structured surface after treatment



Material surface before treatment

Plasma treatment (e.g. O_2)

Activated surface after treatment

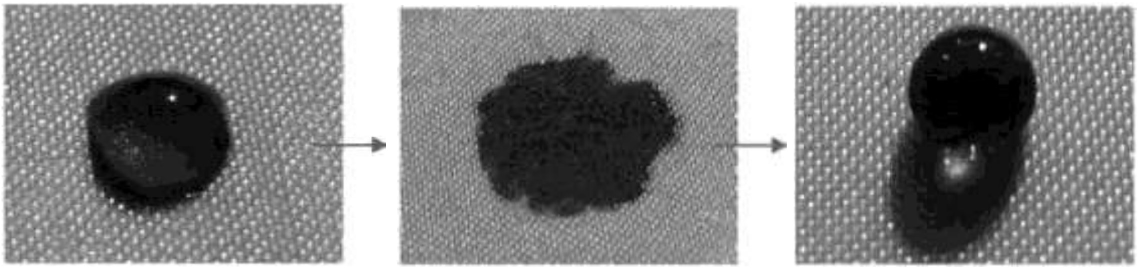


Material surface before treatment

Plasma treatment (e.g. CF_4)

Polymer layer uniformly applied to the material surface

Figure 4 Schematic diagram of plasma action on substrate surface [7]



a) Unprocessed surface

b) Hydrophilic

c) Hydrophobic

Figure 5 Effects obtained by plasma action on the material surface [7]

Such treatments can be efficiently performed with both atmospheric and low-pressure plasma, but low-pressure plasma with a closed reactor is still most commonly used [1]. Appropriate reactors for continuous treatment of fabrics and fibers are required for the implementation of plasma treatment, which should certainly be counted as an investment.

3. Parameters essential for plasma treatment efficiency

In order to carry out targeted modification of properties by plasma treatment and achieve the desired effects, it is necessary to carefully determine and optimize treatment parameters such as: gas type, gas flow, pressure regulated by a vacuum pump (in the case of low-pressure plasma), operating frequency of the device, treatment duration and distance between the electrode and the substrate surface. To illustrate the significance of individual parameters, Table 2 lists the different effects achieved by plasma depending on the operating frequency range at which the substrate modification is performed.

Table 2 Plasma system reactor operating frequencies and effects achieved [3,7]

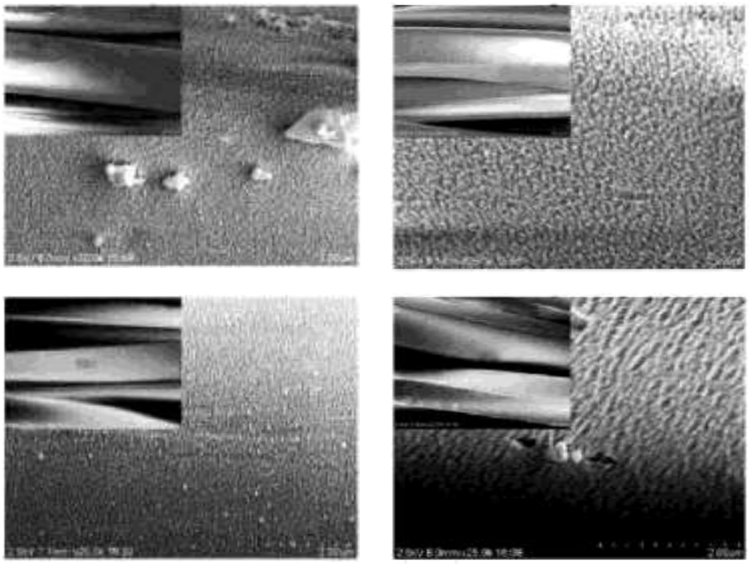
Operating frequency	Operating frequency range	Effect
10-50 kHz	Corona discharge	Surface activation and modification
50-450 kHz	Low frequency (lf) range	Surface activation, lower degree of layering in the polymerization process
13.56 or 27.12 MHz	Radiofrequency (rf) range	Surface activation, high degree of layering in the polymerization process
915 MHz or 2.45 GHz	Microwave plasma (mw) range	Surface micro-etching, polymerization

3.1. Influence of gas type

As already mentioned, due to the high reactivity of ionized particles and radical entities, plasma affects the surface of the treated substrate chemically and physically, thus changing many characteristics of its surface. The types of reactions between plasma and surface depend on the type of gas used and its physicochemical properties. To illustrate this relationship, Fig. 6 shows the effects of surface modification of a polyester substrate obtained by treatments with different gases, i.e. plasma types [9]. As an example, to illustrate the influence of gas type on the efficiency of low-temperature plasma treatment, the research of **Kan et al.** [10] which they conducted on woolen fibers and fabrics will be given. They used different gases: oxygen (O_2), nitrogen (N_2) and a mixture of gases composed of 25% hydrogen (H_2) and 75% nitrogen (N_2). Based on the obtained results, they concluded that the treatment with all types of plasma causes visible changes in the surface, i.e. the properties of the treated material. Changes in physicochemical properties are interesting, which was determined on the basis of testing the dyeing properties. Microscopic analysis revealed changes related to the surface of the wool fiber that becomes rougher and more grooved, which facilitates the diffusion of dye into the interior of the fiber. FTIR-ATR (Fourier Transform Infrared - Attenuated Total Reflectance) instrumental method was used to analyze the surface of a wool fiber, and it was determined that the type of gas plays a significant role in changing the surface chemistry. Thus, during nitrogen plasma treatment, NH_2 groups are formed on the fiber surface, which increases the dye absorption. On the other hand, the application of oxygen plasma leads to surface oxidation, which results in the breaking of cysteine bonds and the formation of cysteic acid, thus improving the hydrophilicity of the fiber surface, and also leading to improved dyeing ability. Hydrogen present in a mixture with nitrogen in one example of plasma has a strong reducing effect on the wool surface, which can lead to the formation of free carbon radicals during plasma treatment which then form C–C networks on the fiber surface reducing the ability to absorb dyes compared to e.g. treatment with oxygen. Similar findings were made by Spanish scientists [11] who studied the changes in wool fibers caused by the application of low-temperature plasma systems using different gases (air, N_2, water vapor and O_2). Additionally, they varied the frequency and treatment durations. Scientists from the University of Knoxville in their patent [12] describe the effects of treatment of hydrophobic fibers with atmospheric plasma using different types of gases: H_2, SO_2, N_2, He, CO_2, CF_4, HPMA (2-hydroxypropyl methacrylate), air and their combinations, with the purpose of finding the possibility of improving the dyeing ability. Treatment was performed at a frequency of 6.67 kHz and higher, which modifies the polymer surface by creating polar functional groups. Such treatment of polypropylene and aramid fibers achieves a better dyeing ability. The treated fibers were found to have a hydrophobic core, while the outer part of the fibers contained hydrophilic functional groups, which are the result of the action of the plasma's active substances with the polymer surface during treatment.

Based on the findings of this research, it can certainly be stated that the correct choice of working gas is an important factor in achieving the target properties. In conclusion, however, it is necessary to emphasize that the optimal properties can be achieved only with the correct selection of all parameters, while ensuring that there is no damage to the textile substrate.

Control (O_2) CO$_2$ plasma (0.1 mbar, 10 min)



Ar plasma (0.1 mbar, 10 min) Ion beam (1.3 10^{-3} mbar, 10 min)

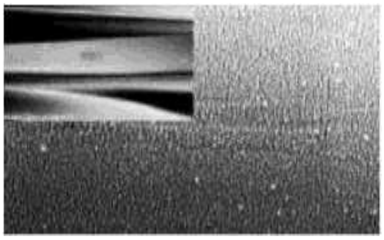
Figure 6 Influence of different types of gases (i.e. different types of plasma) on the surface of the substrate; recorded by SEM technique, at 2,000x magnification (smaller image) and at 25,000x magnification (larger image)

3.2. Influence of pressure and treatment duration

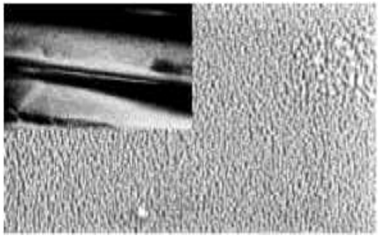
Parameters such as pressure and duration also affect the changes in the physicochemical properties of the textile substrate surface during plasma treatment.

In the low-pressure area (less than 1 mbar), the gas density is lower, the particles collide with each other less, and the surface modification of the textile material is carried out more efficiently with minimal losses. On the other hand, very low pressure causes a relatively low concentration of radicals per unit volume. Therefore, the importance of pressure optimization during plasma treatment should be emphasized in order to achieve satisfactory effects. In pressure ranges greater than 100 mbar (especially at atmospheric pressure and room temperature) due to higher gas density, there are more collisions between gaseous particles, which reduces the efficiency of radicals and thus their ability to bind to active sites within voluminous fabrics [13]. Molecules in air at atmospheric pressure collide with a collision frequency of 10^9 collisions per second, with a mean distance between collisions of about 10^{-8} m (10 nm), while at a pressure of 0.1 mbar the mean distance increases to a few millimeters [3]. In accordance with these settings, **Poll et al**. observed the effect of low-pressure plasma of operating frequency 20 kHz on the increase of hydrophilicity of cotton fabric surface (115 g/m^2). Low-pressure plasma treatments were performed at pressures from 0.6 to 8 mbar, as well as at pressures greater than 100 mbar, and the treatment duration varied from 50 to 800 s (about 13 min). Plasma efficiency was determined by measuring the hydrophilicity of the treated cotton fabric. They increased the number of fabric layers (up to four layers with a total thickness of 1 mm) to determine the depth to which the plasma was effective. Treatment duration is important in optimizing the treatment conditions of textile material. As the treatment duration increases, the active plasma radicals penetrate deeper and deeper into the structure of the material, increasing the hydrophilic effect. They concluded that during low-pressure plasma treatment, the optimal hydrophilicity was achieved at a pressure of 0.6 mbar and a duration of 700 s (about 12 min), even in the inner layers of the textile material [13]. In contrast, during atmospheric plasma treatment and with a significant increase in gas flow and treatment duration, there were changes in the characteristics of only the surface layer of cotton fabric [13]. Fig. 7 shows the influence of pressure on the intensity of surface roughness changes, and Fig. 8 shows the influence of treatment duration on the intensity of changes.

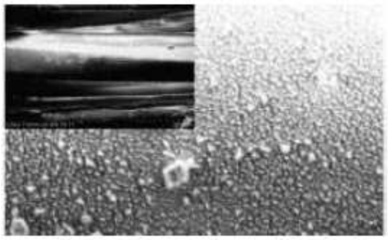
0.07 mbar RF O$_2$



0.1 mbar RF O$_2$



0.2 mbar RF O$_2$



0.4 mbar RF O$_2$

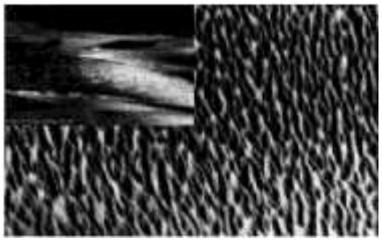
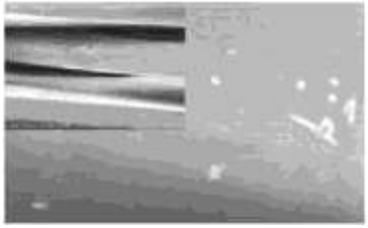
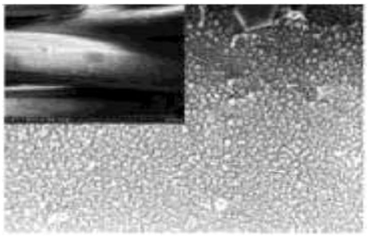


Figure 7 Influence of vacuum level on surface roughness; recorded by SEM technique, at 2,000x magnification (smaller image) and at 25,000x magnification (larger image) [9]

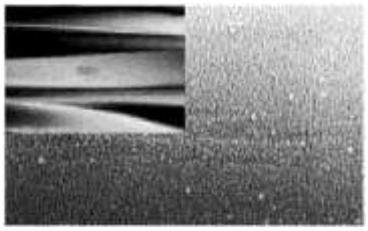
Untreated sample



5 min RF O_2



10 min RF O_2



20 min RF O_2

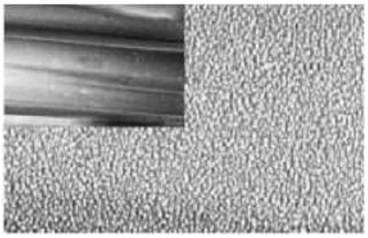


Figure 8 Influence of treatment duration on surface roughness; recorded by SEM technique, at 2000x magnification (smaller image) and at 25000x magnification (larger image) [9]

3.3. Influence of textile substrate properties

Reactions between the active plasma particles and the surface of the textile substrate (fiber) result in the formation of active sites on the fiber, capable of binding new molecules or atomic groups. Occupying active sites on the fiber surface prevents access of new active plasma particles to the surface, thus it becomes neutral and inactive for the binding of new active plasma particles, which during further treatment penetrate deeper into the interior of the textile structure. Therefore, the efficiency of plasma action also depends on a number of characteristics of the processed textile material. In the case of flat textiles (fabrics, knits, non-woven textiles), it is important to keep in mind their complex structure, which consists of numerous elements - from the type and fineness of fibers and yarns to the construction characteristics of flat textiles, its thickness, flat mass, density and texture openness, porosity, etc. Changes caused by plasma-chemical reactions take place not only on the visible surface, but also on the surface of the inner layer of the fabric that is thereby being modified, which is not the case, for example, when treating foils. It has been established that the penetration of plasma into the interior of the processed material depends on these numerous parameters of the textile structure and, for example, the loss of mass and the depth of surface ablation or etching also depend on them. Of course, the treatment duration and the pressure at which the treatment is carried out also play an important role, which was discussed in the previous points [13-15].

The influence of the substrate structure on the penetration of active particles into the interior of the woolen fabric was investigated by **C. Wang** and **Y. Qiu** [16] by applying a *plasma jet system* operating at atmospheric pressure. In this plasma system, the surface of the treated substrate is in direct contact with the nozzle through which the plasma exits. It was found that in the treatment of flat and non-porous substrates, the modification was limited to the outer surface only up to a few nanometers of thickness. On the other hand, when treating porous fabrics other than the outer surface, and depending on the porosity, the plasma can penetrate deeper into the surface layer and cause more intense changes in the form of ablation or etching, i.e. effects are achieved through two surface layers. Changes in surface morphology after plasma treatment were analyzed by an electron microscope (SEM analysis), during which damage to the shells along the fibers was found (shell tips were completely destroyed, and in some places the entire shell surface was destroyed). This indicates that the active plasma particles can penetrate the fabric through its pores and may cause damage to the inner part of the fabric if insufficient attention is paid to the treatment parameters (especially duration). Therefore, it should be emphasized that the optimization of process parameters is of great importance in order for the modification of both surfaces to be controlled, so that optimal properties can be achieved [13,16].

4. Existing knowledge on textile modification by plasma

Although the application of plasma in various fields of activity has been known for a long time, in the field of textile technology the development and commercialization of plasma technology has become increasingly important in the last 15 years, with the environmental aspect being one of the crucial factors. The following features can be highlighted, which nowadays make plasma treatment attractive for targeted modification of the properties of textile materials, and beyond:

- It is applicable to a variety of substrates, i.e. there is a wide choice of materials suitable for treatment,
- By treatment with optimal process parameters, the desired surface properties of the processed material are achieved without significant changes in their basic characteristics,
- It is also applicable to polymers in which the modification of surface properties is difficult or impossible by applying chemical processes in the wet state,
- Processes are carried out in dry, closed and safe systems,
- It is very environmentally friendly.

Plasma treatment is particularly suitable for the modification of polymeric materials, i.e. all types of textile materials, polymeric membranes, foils, composites, laminates, etc., or materials in which surface modification can result in a lasting improvement in properties [1].

The application of plasma has an increasingly important role in many other areas of activity, such as: in the automotive industry, in medicine (catheters, contact lenses, biocompatible implants, prostheses, sterilization of medical equipment), in electronics (in the manufacture of microelectronic elements, semiconductors, sensors, etc.), in the manufacture of solar cells, large plasma screens, in the plastics industry (all types of plastics) and elastomeric materials industry, in the metal industry (technological processes of welding) it is used as a light source, etc. [7,16].

One of the particularly interesting applications of plasma in recent times is the *layering of organic polymers with metal*, whereby the metal layer adheres firmly to the surface of the polymer by adhesive forces, which is achieved by pre-treatment of the polymer with plasma. In this case, O_2 is most often used as a gas source (plasma), and as a characteristic example, the layering of thermoplastic copolymer based on acrylonitrile-butadiene-styrene (ABS) with metallic copper by evaporation can be noted [17]. The application of plasma can also be useful in the production of composites, i.e. laminates, in which good adhesion between the fibers and the matrix, i.e. layers, depends primarily on their surface characteristics and mutual physicochemical bonding. A prerequisite for good adhesion between the fibers and the matrix is the residual surface energy of the fibers, which must be greater than or equal to the surface energy of the matrix [17]. This is exactly what can be achieved by plasma treatment, with four main effects, previously explained in Chapter 2.3, Fig.4. In addition to the physicochemical modification of the surface structure, the transverse chemical bonding of molecules caused by plasma reactions also contributes to the improvement of the permanent connection of components. All these processes, alone or in a synergistic combination, cause better adhesion and a stronger connection of complex structure elements [18].

In addition to these areas, plasma has applications in biology, membrane technology and environmental technology (waste materials elimination and air purification), etc. [17]. Recently, [19] has been published on the application of plasma in the field of microbiology, where a rapid and effective method of removing aflatoxins produced from the mold *Aspergillus parasiticus* has been developed, applied and tested on several types of nuts used in the diet.

4.1. Applied plasma types and treatment techniques

Based on a large number of published scientific and professional papers and technical information, it can be concluded that research on the possibility of applying plasma in the field of textiles is very current and represented, especially in the field of finishing. It is indicated that the application of plasma can be very effective in destarching, in achieving increased hydrophilicity and hydrophobicity, oil repellency, to reduce shrinkage caused by felting, increase the dyeing and printing ability, increase resistance to burning, improve antimicrobial properties and adhesion, for sterilization, improvement of antistatic properties, gloss regulation, UV protective properties, etc. [1, 13, 17, 18, 20-24]. Of course, more pronounced multifunctional effects are achieved if plasma treatment is combined with various other agents, primarily different organic and inorganic particles of micro and nano dimensions. Table 3 lists examples of plasma application for the purpose of modifying the properties of textile materials that have been written about in the scientific and professional literature in the last ten years. Numerous literature data were tried to be systematized according to physicochemical agents used for surface treatment of textiles made of various types of fibers. The achieved effects are related to certain types of physicochemical agents and types of fibers on which the research was conducted. It can be determined that research in this area is very current and numerous, but it is difficult to conclude how much of it has actually been applied in practice and commercialized. As previously pointed out, the treatment of textile materials is carried out exclusively with cold plasma, primarily low-pressure, but also atmospheric plasma, Fig. 3.

4.2. Overview of research on the application of plasma in the field of fiber and yarn modification

As visible from the published literature, the action of plasma is investigated on various types of fibers. Scientists have shown a special interest in **wool**, which is understandable since it is a fiber with a characteristic scaly surface and distinctive morphology. It is therefore not surprising that wool is a common and suitable substrate for such research. Some research related to the treatment of wool with low-temperature plasma is presented in Chapter 3.2. as part of the consideration of the influence of gas type on the effects of plasma treatment. In general, it can be argued that in the study of wool as a textile substrate, in the foreground are modifications that lead to an increase in surface hydrophilicity, both due to physical and chemical changes on the fiber surface [4, 5, 25]. The most pronounced changes in wool occur in the epicuticular layer and partly in the exocuticle layer. Removal of the lipid F-layer facilitates the diffusion of water molecules and dyes into the fiber, which significantly increases the ability to wet and the ability to dye and print [10]. Plasma acts on the surface of the fiber by creating micro-grooves and cracks in the shells, which greatly changes the appearance of the processed fiber, as well as its morphology. Despite the increased irregularities and unevenness of the fiber surface, the frictional characteristics of the surface (differential friction coefficient) are reduced, and based on the established reduction of shrinkage caused by felting, a general positive effect on the reduction

Table 3 Overview of the possibilities of plasma application in the field

Gas	Purpose	Material	Property
O_2	Polymer modification Surface oxidation Surface modification	Cotton, cel. regenerates, CLY, silk PA, PE, PTFE Wool PES, PP, PAN	Dyeing properties, hydrophilicity, bleaching, antistatic properties; reduction of shrinkage due to felting, ability to wet and adhesion, increase of porosity and surface energy
H_2	Oxidized layer reduction, reduction effect	Wool, PAN	Reduction of absorption by forming a C-C network on the wool surface
N_2	Surface modification	PES, wool, PAN	Adhesion improvement Dye absorption improvement
Ar, He, Ne,	Surface activation and dewaxing Activation and polymerization Surface modification	PA PAN CV, CLY, wool	Increase of the depth of dyeing Improvement of fire resistance Improvement of sorption properties
N_2O, CO_2, NH_3, SO_2	Surface activation and modification	PA, PE, PP, PET, PTFE, wool, PAN	Hydrophilicity (wettability) Reduction of wetting angle
Hydrocarbons: ethylene (C_2H_4), ethane, acetylene, methane	Polymerization Surface modification	PES Wool	Hydrophobicity The dyeing intensity is increased
CF_4 (tetrafluoromethane) SF_4, SF_6 (sulfur hexafluoride)	Surface ablation and etching Surface modification	SiO_2, PA PP, PES, silk All types of textiles	Obtaining hydrophobicity, oleophobicity Improving dyeing The feel of the fabric (softer) and shine
Fluorocarbons: C_2F_4, $C2F_6$	Surface modification, polymerization	Cotton, PES	Hydrophobicity
Si-organic reagents, HMDSO (hexamethyl disiloxane), SiC_{14}	Polymerization	CV, PES cotton/PES wool	Antistatic properties Improvement of dyeing UV protection, dimensional stability
Phosphorus with monomers	Polymerization	PAN, CV, cotton	Rire resistance
Air	Surface modification Surface activation and modification	Wool PES	Dyeing ability, hydrophilicity Reduction of the surface resistance Humidity content, electrical conductivity
Steam	Surface modification	Wool, cotton	Surface hydrophilicity
Monomers (acrylonitrile and acrylamide)	Surface grafting, surface ablation and etching	PES	Hydrophilicity, reduction of the surface resistance, increased conductivity

of the propensity to felting can be assumed [26, 27]. Similar was also established by **R. Molina et al.** [11] in investigating the influence of the operating frequency of a plasma system on the efficiency of wool fiber treatment for the purpose of reducing felting.

The effects of plasma treatment have also been investigated on **viscose fibers** [28]. The aim was to increase the absorbency and water retention capacity of nonwovens made of viscose fibers intended for hygienic and medical purposes. After optimizing the parameters of low-pressure plasma treatment, positive results were achieved. Using electron microscopy, FTIR analysis, testing the ability to absorb and retain water, it was found that the desired effect was due to physical changes in the morphology of the fiber surface visible as surface ablation or etching, but also due to the formation of polar chemical groups responsible for increasing water binding. **H.A. Karahan** [29] studied whether the possibility of dyeing **cotton** with acid dyes can be achieved by atmospheric plasma treatment. Acid dyes have an anionic character and are used to dye fibers such as wool, silk and polyamide, in which cationic groups can be formed under certain conditions. Cotton can be said to have a predominantly anionic character and it is not common to dye it with acid dyes. By activating the surface of the cotton material with plasma (air, argon), it is possible to graft molecules of cationic character, which gives the fibers affinity for acid dyes. In general, it has been shown that the application of plasma technology is very suitable for pre-treatment of materials before grafting various monomeric and even oligomeric molecules into the basic polymeric structure of fibers. In addition to the reasons related to efficient physicochemical processes in such treatments, the interest in the application of plasma technology is also contributed by favorable indicators from the ecological and economic point of view.

Plasma treatment is also of great interest in the field of **composite materials**, primarily as a way to improve the adhesion strength between the fibers (reinforcers) and the polymer matrices. It is known that the high properties of composites do not depend only on the physical properties of individual components (fiber and matrix) but that this boundary area between the components plays an important role, primarily the size of the adhesion forces and the compatibility of the components. Adhesion can certainly be affected by modifying the surface characteristics of the fiber in which plasma can play a useful and important role. In the research of **V. Chirila et al.** [30] changes in the mechanical properties of the polymer composite depending on the amount of carbon nanofibers in the polypropylene matrix were monitored. As the strength of the composite depends on the strength of the adhesion between the fiber and the matrix, they tried to improve it by treating the fibers with oxygen plasma. The oxygen treatment was performed in a microwave reactor with a power of 80-120 W, at a pressure of 0.4 mbar, with a gas flow of 80 cm^3/min and a treatment duration of up to 7 minutes. It was found that a composite with a 5% content of carbon nanofibers previously treated with plasma has about 11% higher tensile strength compared to a composite containing the same amount of untreated carbon nanofibers. Moreover, this strength also exceeds the strength of the composite, which contains twice the proportion of untreated fibers (10%). This has shown that the treatment of fibers with oxygen plasma can be a good alternative to conventional oxidative treatment. In doing so, surface modification can increase the density of functional groups (carboxyl, carbonyl and hydroxyl) on the fiber surface and/or the matrix surface, which can also improve the bond strength between two components. It should be noted that insufficiently controlled plasma treatment, due to the high reactivity of ionic and radical entities, can lead to damage and unwanted consequences on the material. Therefore, it is necessary to always optimize the treatment conditions and adapt the type of plasma device to the treated substrate in order to avoid possible negative impact on the properties of the treated material [31].

Studies conducted on **cotton yarn** have shown that treatment of yarn with low-pressure plasma can cause a significant increase in breaking force and a decrease in elongation at break, and the magnitude of the change is significantly affected by the treatment duration [32]. It was previously mentioned that intensive plasma treatment can damage the fiber at the molecular level, but also in the form of mass loss [33], which indicates the importance of optimizing the process parameters to avoid a negative effect. A positive effect of plasma treatment on mechanical properties was also found in high-strength fibers. Studies conducted on **Kevlar monofilament** indicate an increase in tensile strength after treatment with atmospheric plasma with He and He/O_2 mixture [34]. Similar effects of improving the mechanical properties were achieved in the treatment of **Specter** fibers (HMWPE) [35,36], despite the fact that there was a significant change in the morphology and ablation or etching of the surface of the treated filaments. Unlike Specter, plasma treatment on Kevlar causes almost no changes in surface morphology. The increase in tensile strength is assumed to be due to cross-linking. Another reason could be in the helium used for the treatment, which is known for its efficient cross-linking of polymers [37].

The application of atmospheric plasma of a mixture of gases He/air and He/O_2/air in the destarching of cotton yarn was investigated by **Z. Cai et al.** [38]. The starch was based on polyvinyl alcohol (PVA). Based on the test results, it was determined that better destarching efficiency is achieved with a He/O_2/air mixture, whereby the PVA film is removed from the cotton surface, and the starch is removed from the fibers already by washing in cold water. The tensile properties did not show statistically significant changes after the treatment.

Scientists from the Faculty of Chemical and Food Technology, Department of Plastics and Leather in Bratislava (Slovakia) studied various types of surface modifications of **polyester cord** [39] by low-temperature plasma at atmospheric pressure. They carried out the modification in two ways. In one series of studies, they performed cord surface activation by plasma treatment with a mixture of nitrogen and air, and in another series, they used a mixture of nitrogen and butadiene to perform plasma polymerization. Using the standard technique of surface morphology analysis (SEM, AFM) and chemical elemental analysis (XPS), they found that there were changes in surface characteristics (surface roughness caused by nitrogen action), but also chemical interactions related to the surface that are responsible for improved adhesion between the treated PES cord and the polymer matrix. To evaluate the effect of plasma treatment on the adhesive strength between cord and rubber, they compared it with the adhesive strength between resorcinol formaldehyde latex (RFL) and PES cord treated by conventional chemical treatment in the rubber industry. They concluded that the use of plasma can simplify the surface modification of the PES cord and increase adhesion to the rubber, without adversely affecting the environment.

By the method of spraying particles in plasma, **D. Hegemann et al.** [40] carried out the layering of the yarn with silver particles and applied a very thin metal film to the surface of the yarn (fibers). This achieves bactericidal properties and at the same time increases the electrical conductivity, so the yarn can be used to make antibacterial and antistatic clothing (used in medicine and in intelligent clothing with built-in electrodes and sensors). The advantage of this treatment is that the processes of surface activation and layering are carried out simultaneously and in harmony, which achieves greater uniformity of the film and better adhesion of the metallized layer and fiber.

4.3. Overview of research on the application of plasma in the field of flat products modification

It is known that many important properties of polymeric materials (e.g. adhesion, friction, hydrophilicity, dyeing ability, etc.) are largely conditioned by the physical and chemical characteristics of their surface. Plasma treatment can be an effective and environmentally friendly way of targeted change in the surface characteristics of flat textile structures, which can reduce the use of environmentally unacceptable chemicals in finishing processes. Unlike wet physicochemical processes that penetrate deep into the interior of the fiber, reactions caused by plasma take place on the surface, at a depth of up to several tens of nm, and therefore plasma treatments can be recommended as favorable. If carried out in optimized and controlled conditions, as a rule, they do not cause unwanted changes in the basic properties of the material.

Research on the possibility of applying plasma in the field of modification of the properties of cotton materials in recent years is increasingly present, and the main areas of modification are: increasing hydrophilicity [13,41], dyeing ability [42], hydrophobicity [43,44], bleaching efficiency [45], fire resistance [44,46], etc. In this sense, **M.J. Tsafack and J. Levalois-Grützmacher** [47] have studied the influence of low-pressure microwave plasma treatment on the effectiveness of various anti-burn agents applied to the surface of **cotton and acrylic fabrics** [44, 46]. Based on the obtained findings, they developed a method in which surface activation and monomer grafting and polymerization reactions take place simultaneously in a plasma chamber (so-called plasma-induced graft polymerization, PIGP) [46,47]. It has been established that the enhancing action of plasma can be achieved in three ways: by activation, i.e. by modifying the surface structure of the fabric, then by polymerizing the selected agent on the surface of the material and by grafting the agent with plasma activation (so-called plasma-grafting).

The research was carried out on cotton fabrics with a flat mass of 120 and 210 g/m^2 with the use of acrylic monomers containing phosphorus (acrylate phosphates, phosphonates and phosphonamides), with a photoinitiator (PIGP method). Based on the spectra recorded by the NMR and IR (ATR) analysis, the polymerization of acrylic monomers was confirmed, and it took place within the microwave argon plasma, by a radical mechanism, most often initiated by radiation from the UV and visible part of the spectrum. Thermal characterization of polymers was performed by thermogravimetric analysis (TGA) and the thermal stability of the obtained polymers on cotton fabric was determined. In all treated samples, an LOI value above 25 was achieved, which confirmed the effectiveness of such treatments [46]. A year later, the same group of scientists investigated the possibility of obtaining multifunctional surfaces with a minimum number of treatments in order to obtain fire-resistant and water-resistant cotton material [44]. The treatments were performed with low-pressure microwave plasma using the PIGP method, according to [46], and subsequently treated with CF_4 plasma. By treating at a pressure of 0.66 mbar, gas flow of 36 cm^3/min, power of 300 W and duration of 5 min, they achieved fire and water resistance properties. At the same time, research of the possibility of increasing fire resistance was carried out on acrylic fiber fabrics (290-300 g/m^2), also with the use of four acrylic monomers containing phosphorus. The treatment was performed with microwave argon plasma with a frequency of 2.45 GHz under defined conditions: pressure of 0.4 mbar, gas flow of 125 cm^3/min, power of 100 W and treatment duration of 15 min. Based on the analysis, they concluded that the developed method is effective because the polymer is covalently bonded to the fabric surface, which ensures the durability and stability of the applied layer in regard to washing (**Mc Sherry** method) at higher temperatures as well, and thus better resistance to burning [47]. In the paper [48] it is pointed out that a large number of works on woolen fabrics relate to the study of surface characteristics of fibers in terms of improving surface wetting [49], surface energy of wool (Wilhelm's principle) [6], the impact on shrinkage [5, 26, 27, 50] and the improvement of dyeing properties [49], while the number of papers in the field of plasma influence on mechanical [51] and thermal properties [48] and on-air permeability [48] is smaller. Therefore, in his paper he gives results on the influence of low temperature O_2 plasma on these properties. By applying the KES and FAST systems, the tensile, shear, compression, surface and bending properties of the samples after treatment were analyzed, and the obtained changes were explained by the increased action of friction force inside the yarn and within the fibers that resulted from the treatment. Changes related to air permeability properties after treatment are probably related to changes in fabric thickness and surface morphology (rougher surface), and changes related to thermal properties can be related to air trapped in the spaces between yarns [48]. It is obvious that plasma certainly has a positive effect on the modification of wool properties, but it should be noted that in addition to such excellent results, the problem of poor wool feel that occurs just after plasma treatment and which is the subject of study of many scientists [4] has not yet been resolved. In the paper of **C. Canal et al.** [52] emphasis is placed on clarifying the ambiguities related to the role and significance of the lipid layer in wool on the shrinkage and usage properties of wool. By comparing the test results of untreated and plasma-treated wool samples, it is proven that plasma action can lead to oxidation and partial removal of the lipid layer, which directly affects the improvement of the hydrophilicity of the surface of wool fibers.

The application of plasma in wool finishing processes is a much more environmentally friendly process compared to conventional chlorination processes in which large amounts of chlorine go to wastewater. The possibilities of treatment in which enzymes and biopolymers are used in addition to plasma are also being investigated. **P. Erra et al.** [26] have been investigating the properties of wool after plasma treatment and additional treatment with biopolymer **chitosan.** Plasma treatment was performed with a microwave plasma with frequency of 2.45 GHz, with air and a pressure of 1 mbar, and at different durations up to 5 min. Based on the obtained results, it was found that this combined treatment reduces wool shrinkage whereby chitosan is adhered to the hydrophilic surface of the fiber, which is why the physical changes in the fiber surface are smaller compared to the change that occurs in plasma treatment without the addition of chitosan. This method of modification represents a new ecological procedure for treating wool against shrinkage.

The emphasis on modern textile treatment with the purpose of improving the functional properties by modifications exclusively at the level of the fiber surface is also placed by **D. Jocić et al.** [53]. By combining "dry" pretreatment (plasma) and "liquid" after-treatment with a biopolymer (to stabilize and improve the achieved effect), they developed an optimal method for obtaining wool resistant to shrinkage. As a substrate, they use **non-woven textiles** made of recycled wool with the intention of using such a highly absorbent material to remove heavy metals and dyes from the textile industry's wastewater. In addition to wool, research has been conducted on cotton by ozone treatment for the purpose of obtaining cotton of improved whiteness, but also interactive cotton material with a built-in layer of hydrogel (capable of reacting to changes in pH values with different degrees of swelling) [53].

M.S. Kim et al. [50] investigated the effect of O_2 plasma treatment on the dimensional and surface properties of woolen fabrics in combination with after-treatment with silicone polymers. Pre-treatment was carried out in a plasma system of frequency 13.56 MHz, pressure 1 mbar, with 99.9% pure O_2, of flow 50 cm^3/min, at various levels of power (50, 100 and 150 W) with treatment duration of 1 min. Upon completion of the pre-treatment, the samples were impregnated with two types of silicone polymers. Based on the analysis of the results, it was concluded that plasma pre-treatment (the effect of plasma action was restricted to the surface layer of depth of 1 to 10 μm) in combination with silicone polymers results in some smoothing of the flaky surface (silicone film) which is also reflected on the dimensional stability of woolen fabrics, less propensity to felting and less propensity to creasing (recovery angle increases). However, the bending stiffness increases even after the treatments, which the author explains by a stronger connection between the fibers within the yarn and the yarn with each other, which limits the fiber-fiber movement and increases the stiffness of the fabric. Testing of the mechanical properties showed that there is a decrease in the resistance to further tearing of plasma-treated samples, which then increases after treatment with silicone polymers, which the authors interpret by the formation of an elastic silicone mesh on the fiber surface. The breaking force does not change significantly (it decreases very little). Layering of woolen fabrics with organosilicones in order to achieve better dimensional stability of woolen fabrics has also been studied by *Italian scientists* [54] using atmospheric plasma. They carried out the treatment in two phases; surface oxidation in the first phase and layering with the organofunctional polymer hexamethyldisiloxane (HMDSO) in the second phase. Plasma oxidation of the surface reduces the number of disulfide bridges and reduces the hydrophobic character of wool. The sample thus activated is subjected to treatment with HMDSO in plasma to initiate a grafting reaction with plasma. SEM analysis has shown that the samples of woolen fabrics treated in this way have a less rough surface thanks to the layered polymer which gives a smoothing effect. This results in the hydrophobicity of the surface - the wetting angle of water increases up to a maximum of 180°.

Apart from wool, to a lesser extent the research covered textile materials from other types of fibers. **K.K. Wong et al.** [55,56] have been researching the possibilities of applying plasma in order to facilitate the separation of flax fibers from the core and in general the process of obtaining fibers and their processing into more complex products. Experiences related to flax processing processes are related to energy costs, requirements for special machines and problems related to wastewater and environmental pollution. Precisely for these reasons, there was a need to find the possibility of processing flax in a more acceptable way, and this is where the low-temperature plasma treatment has found its place. It has been shown that low-temperature O_2 and Ar plasma applied to linen fabrics causes physicochemical changes in the surface layer, which ultimately result in better sorption and dyeing properties and a softer feel. Such discoveries are very important for the revitalization of flax production and the expansion of the use of flax products. **M. Radetić et al.** [57] investigated the influence of low-temperature plasma pre-treatment in combination with enzymatic treatment on the dyeing properties of a fabric made of hemp fibers. Isothermal dyeing at a temperature of 60°C (so-called cold dyeing) with acid and direct dyes was performed.

Scientists from the Institute of Textiles and Clothing in Hong Kong [58] are studying the effects of low-temperature plasma to improve the dyeing ability of **Tencel fabric** and obtain a value-added product to penetrate new markets. Treatment was carried out with O_2 and Ar plasma, with gas flow of 10 cm^3/min, pressure of 0.1 mbar, power 100 W and treatment duration of 5, 10, 20, 30 and 60 minutes. Based on the results of extensive tests, they concluded that a treatment duration of 5 min was sufficient to improve the dyeing ability, as determined by the process of depletion of dye from the bath. On the spectra recorded by the FTIR technique on a sample treated with plasma, more hydroxyl groups were determined compared to the untreated sample. However, as cellulose fibers already contain a large number of hydroxyl groups, it is not considered that the increase of hydroxyl groups during plasma treatment is not the main reason for the increase in the ability to receive dyes. The thesis is that a significant increase in the diffusion of dye molecules into the interior of the fiber is primarily a result of the increase in the total specific surface area of the fibers caused by the change in the surface structure during plasma treatment. This has also been confirmed by SEM images on which the visible effect of ablation or etching of the fabric surface is particularly pronounced in O_2 treatment (similar to the phenomenon that occurs on cotton). This effect was confirmed by a relative decrease in the mass of fabric samples after processing (O_2 - by 1.4%; Ar - by 3.4%) and an improvement in wettability (reduction of the wetting angle of fabrics treated with O_2 - by 21%, Ar - by 29%) compared to the untreated fabric sample.

It is well known that one of the disadvantages of man-made fiber products is their tendency to charge with static electricity caused by poor electrical conductivity, mainly associated with poor ability to absorb moisture. One way to address this problem is by plasma treatment and grafting of hydrophilic monomers onto the substrate surface. In this sense, **N.V. Bhat and Y.N. Benjamin** [41] are investigating two types of treatment for grafting fabrics using low-pressure plasma with radio waves frequency generated in the air and plasma with the addition of hydrophilic monomers (acrylonitrile and acrylamide). The effects of the treatments were investigated based on the measurement of the change in surface electrical resistance and the percentage of the equilibrium of moisture in the samples of cotton and polyester fabrics after treatment. The relative reduction in surface resistance on polyester fabric is greater than the one on the cotton sample. The decrease in electrical resistance is primarily due to the formation of polar chemical groups on the fiber surface. During plasma treatment on polyester material, as in relation to the initial state a relatively larger change of polar groups (larger number) is realized, the changes in the conductivity of this substrate are larger. It is known that cellulose has a large number of hydrophilic groups even without plasma treatment and does not show a tendency to charging with static electricity, but it is interesting that plasma treatment still achieves a certain additional reduction in surface resistance on this substrate. The advantages of using low-temperature plasma for the purpose of modifying textile products by thin film application were patented in 1999 by Russian scientists [59]. At the same time, English and Italian scientists were working to investigate the possibility of applying plasma with the aim of increasing the resistance of textiles to soiling [59, 60]. According to the researchers, plasma-treated fabrics with sulfur hexafluoride (SF_6) as a monomer have a softer feel, water repellency and oil repellency, and a brighter and more stable color is achieved. The treatment is carried out at room temperature, in a vacuum chamber at a pressure lower than atmospheric and in the range from 0.1 to 20 mbar. The electron density ranges from 10^8 to 10^{11} cm^{-3}, and the treatment duration should not exceed 15 min, in some cases 5 min is sufficient, as stated in the patent. Under these conditions and with this type of plasma, thermal degradation of the material being treated can be avoided.

A group of scientists [61] developed an ***in situ* plasma polymerization method** for embedding functional groups in the surface of polyester and polyamide fabrics using five alternative methods in which acrylic acid, water, air, argon and oxygen are used as agents. The aim is to improve the sorption characteristics, wetting and dyeing ability, as well as resistance to soiling, and by examining these properties before and after treatment, it was found that positive effects were achieved with all these properties. Canadian researchers [62] have developed a procedure that leads to a significant increase in the electrical conductivity of polyester fabrics and a decrease in the tendency to charging with static electricity. This is a cold plasma and hydropropyl-pyrrole fabric treatment in three steps: first the surface of the polyester fabric is activated with Ar plasma, then hydropropyl-pyrrole is grafted *in situ*, and pyrrole copolymerization with grafted hydropropyl-pyrrole is carried out. The potential application of the electrically conductive textile obtained in this way is in the filtration of flammable and explosive chemicals, in the production of light composites in construction, in the production of protective clothing, etc. Similar studies of the possibility of increasing electrical conductivity by applying low-pressure plasma and grafting conductive polymers polyaniline and polypyrrole have been conducted on PA 6 and PP fabrics [63,64].

In addition to researching how to achieve the best possible hydrophilicity of the surface, many scientists are working to find solutions to achieve greater hydrophobicity of textile surfaces, also by low-pressure and atmospheric plasma treatment [19, 65-67]. The best illustration of a hydrophobic surface is presented as the "lotus leaf effect" (patent of prof. W. Barthlotta, 1997) according to a natural phenomenon when a water drop comes in contact with the surface of a lotus leaf, Fig. 9. In doing so, the drop retains its shape due to the nanostructure of the leaf surface roughness (size non-uniformity measured in nano-dimensions), which is visible with an electron microscope. Such hydrophobicity results in a self-cleaning effect, in such a way that the drops, during their rolling down the leaf surface, take away the impurities, leaving the surface clean. It was this phenomenon that challenged scientists to achieve similar effects on textile materials by applying plasma and various agents based on fluorinated compounds (C_2F_6, C_2F_4) or silicone-based compounds (hexamethyldisiloxanes, polydimethylsiloxanes) to obtain highly comfortable, inexpensive, breathable hydrophobic surfaces resistant to washing. In general, it can be said that the principle of action of different hydrophobic treatments on different textile materials is the same, i.e. it leads to an increase in the wetting angle of water drops on the fiber surface. Treatments are carried out not only to reduce wetting, but to obtain the appropriate sliding angle important for the ability of water drops runoff from the sloping surface in a manner similar to that of the lotus leaf surface [65]. Research was performed using cold plasma, and morphological changes in the treated surface of PES fabric were examined using SEM and AFM (Atomic Force Microscope) techniques. The hydrophobicity property was determined by measuring the wetting angle using the DCA (direct contact angle) technique. Based on the results obtained by the AFM technique, it was found that the surface roughness increases as the treatment duration increases, which in the next phase of polymerization of the selected agent provides better surface hydrophobicity [19]. The plasma activation and surface preparation phase is very important. It is necessary to achieve the best possible oxidation of the surface, whereby increased hydrophilicity allows better interaction between the film and the substrate. A hydrophobic film on the fabric surface is created by polymerization of the fluoropolymer in the plasma after surface activation has been performed [65-67]. Scientists believe that the application of atmospheric plasma treatments will become a useful and successful method for the textile industry, because the successful modification of the textile surface can be achieved by using a small amount of chemicals. It is performed at atmospheric pressure and the treatment can be carried out continuously [67]. A significant step forward in the application of plasma was made by *Italian scientists* [68] which used SF_6 plasma to fluorinate the surface of polyester mesh and hexamethyldisiloxane (HMDSO, $C6H_{18}OSi_2$) for polymerization to create a hydrophobic surface. Such mesh is used in filters to separate oil and water in the separation technology, in a way that it absorbs oil but does not absorb drops of water, which can effectively stop the passage of unwanted fluid.

E. Selli et al. [69] and **P. Chaivan et al.** [70] have been researching the possibilities of obtaining hydrophobic properties of silk fabric treated with environmentally friendly cold SF_6 plasma. They believe that the most commonly used plasma treatments with organic fluorides (CF_4, C_2F_6) give polymer films of low durability and therefore such treatments should be avoided in favor of treatments in which fluorine atoms bind to the fiber surface by plasma-initiated grafting [69]. The efficiency of hydrophobing and the durability of the effect is enhanced by the increase in crystallinity by the formation of new cross-links in the polymer structure during treatment. In addition to the previously mentioned polymerizations with siloxanes [25, 58, 71] and fluoropolymers [65-67], in order to achieve hydrophobicity, but also to improve adhesion properties, research on the layering of cotton and polyester fabrics with aluminum (Al) and copper (Cu) particles on a surface previously treated with Ar plasma was performed [43]. To determine the hydrophobicity of the surface, the drop test was carried out, according to which in all samples treated with plasma and metal particles the absorption time of the drop significantly increased compared to the untreated sample (untreated cotton: 5.8 s, cotton-*Cu*: 60

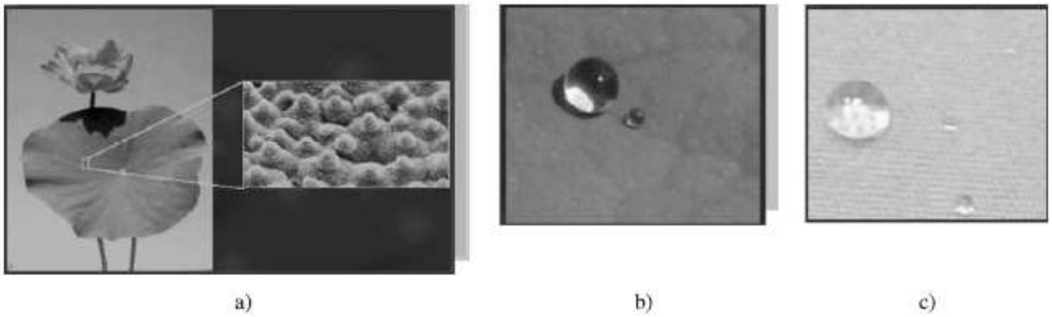

a) b) c)

Fig. 9 Lotus leaf effect: a) lotus leaf and SEM image of its nanostructured surface, b) hydrophobic surface of the lotus leaf and the self-cleaning effect, c) hydrophobic cotton fabric treated with plasma [65] min, cotton-*Al*: 20 min; untreated PES: 18.2 s, PES-*Cu*: 80 min, PES-*Al*: 39 min). The physicomechanical properties and the general appearance of the material do not change significantly, but copper causes a certain color. Therefore, the authors prefer the use of Al particles, although a somewhat weaker hydrophobic effects are achieved with them.

Researchers are showing great interest in modifications with silver (Ag) particles with which excellent antibacterial and antistatic effects can be achieved. In the previous section, it was pointed out that D. Hegemann et al. applied Ag particles by yarn metallization process. A large number of scientists are investigating the process of applying Ag particles using plasma, in order to obtain layers of antibacterial and multifunctional properties in one treatment. The application of particles can be carried out by chemical layering (plasma polymerization, PECVD process), [72] and physical spraying (PEVD process) [73]. The application of silver nanoparticles in antibacterial textile treatment is interesting because of the excellent antibacterial effects that are achieved and their stability. The possibility of applying corona treatment to activate the surface of PA fibers is also mentioned, which can contribute to the efficiency of binding of Ag nanoparticles from colloidal solution to the fabric surface, creating a composite system of antibacterial properties [74]. In addition to silver particles, antimicrobial layering of PES fabric was performed with ammonium chloride, where atmospheric plasma was used for the pre-treatment of fabric [75].

In the recent literature, there is more and more information on research into the possibility of applying plasma techniques to modify the properties of textile materials from various types of fibers - polyamide [76,77], polypropylene [77-80] and polyurethane [81, 82]. **J. Yip et al.** [76] have been investigating the influence of low-pressure plasma from non-polymerizing gases (O_2, Ar and CF_4) on changes in morphological characteristics, mechanical and thermal properties and air permeability in the treatment of PA6 fabrics. After treatment, based on SEM analysis, it was found that after short-term treatment (5 min) with oxygen and argon, the fiber became smoother than untreated fiber, and by extending the treatment duration to 30 min, the surface became wrinkled. On the other hand, CF_4 causes milder changes, and with a shorter treatment duration it tends to form a film, which agrees with the results of the research conducted by **T. Yasuda et al.** [33]. After a longer exposure to CF_4 plasma, the surface of the fiber was etched. **M.G. McCord et al.** [77] found in their study that the tensile strength of PA6.6 treated with He and He/O_2 mixture plasma increases, without changes in surface morphology. The effects of He and He/O_2 mixture using plasma at atmospheric pressure were investigated in [77] and [78] on PP fiber nonwovens. Extensive SEM surface analyzes showed significant changes in the surface area of PP, especially after treatment with a He/O_2 mixture, which was confirmed by elementary surface analysis (XPS method), which determined that the relative oxygen content in relation to carbon (O/C) increased to 29%. As PP has a very low value of free surface energy (about 20-25 mJ/m^2), and thus weak hydrophilic properties, modification of the surface of PP with low-temperature plasma is one of the promising solutions to improve hydrophilic properties [79]. Recent research has addressed the modification of PP fabrics for protective masks, so that polymerization with acrylic acid in O_2 plasma has been performed to increase water absorption capacity [80].

Polyurethanes have proven to be polymers suitable for use in biomedical purposes due to their excellent compatibility with the human body, so their use is becoming increasingly important today. They play a significant role in the manufacture of prostheses or implants (e.g. blood vessels, prostheses, joints, ligaments, surgical sutures), in the use of therapeutic aids (e.g. hemodialysis, blood oxygenation, catheters, blood bags, etc.), in systems with controlled release of drugs, microcapsules and for diagnostic analysis. Therefore, special attention is paid to the modification of the surface characteristics of thermoplastic polyurethane using (microwave) plasma for the purpose of surface activation. Such pretreatment allows the grafting-on of specific functional groups of acrylic acid to the surface of the polymer, which in a further stage leads to the hardening of collagen. In this the carboxylic groups from acrylic acid play an important role because they react with the amino groups of the protein, which affects the better fixation of collagen on the surface of the substrate. In addition to collagen, heparin is also used, which is known to prevent cell accumulation and thus contribute to increased blood compatibility. Plasma chemically alters the surface of polyurethane and increases its hydrophilicity, thereby reducing protein adsorption and cell adhesion to tissue walls (thrombus formation in blood vessels), making it interesting to study in the field of biomedicine [81, 82].

5. Conclusion

There is no doubt that research on the possibility of modifying the properties of textile fibers and materials by applying various types of plasma and plasma in combination with other physical and chemical agents is very current and numerous. Published papers show that respectable results have been achieved, but that the space for further development is wide, i.e. that there is still a number of unknowns that need to be explored. It can be assumed that plasma technology, primarily due to its environmental friendliness, will over time replace certain environmentally unsuitable finishing processes. These are mainly processes in which chemicals are used that can be harmful to health, participate in the pollution of wastewater and air and the generation of large amounts of solid waste, which could be avoided by the use of plasma. The disadvantage of plasma processing technology compared to traditional processes is the high investment costs.

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SUMMARY

Modification of Textile Properties Using Plasma

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An overview of the published investigations undertaken in the field of the target applications (modification) of the properties of textile materials using different types of plasma and the plasma in combination with other physical and chemical agents is provided. The emphasis lies on the properties which are the consequence of the characteristics of textile surface materials (hydrophilicity, hydrophobicity, antistatic property, electrical conductivity, multifunctionality). After explaining plasma phenomenology as a special state of matter, the possibilities of generating and obtaining plasma are described. The classification of plasmas according to different criteria is explained. The plasmas suitable for the treatment of textile materials are emphasized and their basic parameters relevant for the implementation of material treatments are explained. Numerous investigations described in many papers over the last ten years or so are shown according to the type of textile materials on which they were performed (fibers, yarns and fabrics).

Key words: textile materials, plasma, plasma treatment, textile surface, modification of properties, multifunctionality, ecological processes

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Modifizierung von Textileigenschaften durch Plasmaverwendung

Eine Übersicht über die veröffentlichten Untersuchungen, die im Bereich der gezielten Veränderungen (Modifizierung) der Eigenschaften von Textilmaterialien durch Verwendung unterschiedlicher Plasmatypen von Plasma und des Plasmas in der Verbindung mit anderen physikalischen und chemischen Mitteln vorgenommen werden, wird gegeben. Die Betonung liegt auf den Eigenschaften, die die Folge der Eigenschaften von Textil-oberflächenmaterialien (Hydrophilie, Hydrophobie, antistatische Eigenschaften, elektrische Leitfähigkeit, Multifunktionalität) sind. Nach der Erklärung der Plasmaphänomenologie als einem speziellen Zustand der Materie, werden Möglichkeiten zur Plasmabildung und -gewinnung beschrieben. Die Klassifizierung von Plasmen nach verschiedenen Kriterien wird erklärt. Die für die Behandlung von Textilmaterialien geeigneten Plasmas werden hervorgehoben, und ihre grundlegenden für die Durchführung von Material behandlungen relevanten Parameter werden erklärt. Zahlreiche, in vielen Arbeiten beschriebene Untersuchungen im Laufe der letzten zehn Jahre oder mehr, werden gemäß dem Typ von Textilmaterialien dargestellt, auf denen sie (Fasern, Game und Stoffe) durchgeführt wurden.