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ORIGINAL RESEARCH

Modification of cotton fabrics in inductively coupled plasma

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Abstract Textile materials are widely used in various fields of industry and life. Modification of the properties of textiles can lead to an expansion of the possibilities of using a particular material. In this work, the cotton fabric was modified in inductively coupled plasma in an octafluorocyclobutane gas C_4F_8 environment. It turned out that such treatment leads to hydrophobization of the cotton fabric surface through the formation of a Teflon-like film. The composition of the film was determined using FTIR and XPS analyses. Processing at powers from 750 to 1250 W for 10 min results in discoloration and destruction of the material, which is observed visually. SEM images are shown local etching of the fibers. A shorter treatment from 0.5 to 3 min at a power of 1250 W does not lead to such consequences. The dependences of hygroscopic properties such as water absorption, capillary absorption, moisture content and contact angle on RF power applied to the gas discharge and process time are determined. The maximum obtained the contact angle is 120°–123°. The thicknesses of the resulting films range from 45 to 1032 nm. Water absorption, capillary absorption and moisture content of the cotton fabric after processing reach their minimum values and practically do not change.

Keywords Plasma modification · Textile materials · Hydrophobicity · Teflon films · Hygroscopic properties

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Introduction

Textile materials with hydrophobic and superhydrophobic films on the surface are widely used in various industries (Shah et al. 2022). Hydrophobic and superhydrophobic surfaces on textile materials are obtained in various ways, which can be divided into liquid and dry (Wei et al. 2020; Cheng et al. 2022). Liquid methods include the sol–gel method, coating by immersion in a solution, impregnation in emulsions and waxes, and electrospinning can also be attributed (Mazzon et al. 2019; Espanhol-Soares et al. 2020; Przybylak et al. 2020; Zhou et al. 2021). Dry methods for obtaining hydrophobic surfaces include atomic layer deposition (ALD), chemical vapor deposition (CVD), including plasma enhanced chemical vapor deposition (PECVD), as well as radio frequency (RF) plasma deposition, etc. (Chen et al.

2016; Xu et al. 2019; Şimşek and Karaman 2020; Eslami et al. 2021). Also an alternative to the liquid method is the electron beam irradiation method (Jiang et al. 2016). Often a combination of methods is used, for example, first impregnation in a solution, and then plasma modification is carried out, or, conversely, to create the necessary roughness, they are processed in plasma, and then immersed in solutions.

The essence of the methods consists of forming thin hydrophobic layers on the surface of the material (Sohbatzadeh et al. 2019) or inoculation of various functional groups that change the surface properties dramatically (Gorjanc et al. 2010).

In liquid methods, to impart hydrophobic properties to textile materials, solutions containing silicon, titanium, aluminum, copper, etc. ions are used (Khattab et al. 2019; Anjum et al. 2020; Rashid et al. 2021; Endiiarova et al. 2022; Prabhakar et al. 2022). Surface treatment with liquid solutions and emulsions is very effective in imparting hydrophobicity to various textile materials. This treatment makes it possible to obtain surfaces with a contact angle of more than 150°, such as in the work Wei et al. (2020). In Bu et al. (2019), Abeywardena et al. (2021), Khan et al. (2021), Thennakoon et al. (2022) the research on the production of hydrophobic and superhydrophobic coatings by wet chemistry is presented.

One of the used water-repellent agents when using dry methods are fluorine-containing gases or mixtures. Thus, Teflon-like films are obtained on the surfaces. Polytetrafluoroethylene (PTFE, Teflon) is obtained on many materials in the form of a film using various methods (Bodas et al. 2005; Satyaprasad et al. 2007, 2010; Quade et al. 2010; Satulu et al. 2019). Addition of Teflon coatings to cellulose and textile fibers makes the surface hydrophobic and oleophobic. In (Dimitrakellis et al. 2017) the process was carried out in two stages: selective etching in helium–oxygen plasma at atmospheric pressure, and then the deposition of Teflon coatings in RF plasma (13.56 MHz). C_4F_8 was used as the working gas. The RF power was fixed at 900 W, the pressure was 5 Pa, the C_4F_8 flow rate was 50 cm^3min^{-1}, and the substrate temperature was constant at 0 °C. The deposition rate was ~ 35 nm min^{-1}. The samples were processed for 45 s to deposit an ultrathin film about 25 nm thick, the contact angle achieved was over 150°. It is worth noting that other methods, such as electron beam irradiation, can be used to modify the surface before film deposition, but such treatment can be detrimental to the fibers (Abou Elmaaty et al. 2022; Thite et al. 2023). Irradiation allows for chemical modification, such as depolymerization, oxidation, etc. For example, in the work Henniges et al. (2013) cellulose was modified by irradiating the pulp with an electron beam. This treatment led to degradation of the material. It becomes obvious that there is not much oxidation of the cellulose backbone occurring, but rather an increase of the oxidized functionalities caused by newly formed REG. In the article Denes et al. (1999) as substrates, in addition to paper, aluminum foil, silicon, and stainless steel discs were used. Vapors of dodecafluorocyclohexane and octadecafluorodecalin were used as the working gas. It turned out that the impact of the plasma in just 30 s is large enough to reach an extremely high value of the contact angle (120°). Longer processing time does not significantly affect the contact angle. In Ťahel et al. (2004), Kloc et al. (2006) Teflon coatings were obtained on filter paper from a mixture of C_4F_8 with nitrogen. The contact angle of the water with the paper surface increased from 46.3° to about 144°. In the article Wang et al. (2022) special composite layers of Teflon and silver were received. Commercially available polyester fabrics with a smooth polyurethane coating were used as substrates. Silver and PTFE layers were obtained by magnetron sputtering, the thickness of the resulting layers was 50 nm. In the article Samanta et al. (2021) hydrophobic functionalization of cellulose tissue (viscose) was carried out using helium/tetrafluoroethane plasma. Superhydrophobic surfaces were obtained with a contact angle of about 153°. The fabric remained hydrophobic after 25 washes.

There are few works on obtaining Teflon-like hydrophobic films in ICP, so the aim of the work is to obtain Teflon-like films on textile materials in order to hydrophobize the surface in ICP and to study the dependence of the quality of coatings on the characteristics of the discharge.

Materials and methods

Materials

The textile samples were squares of bleached cotton fabric with a density of 142 g m^{-2}, the warp and weft fibers are alternately interconnected in a 1:1 pattern.

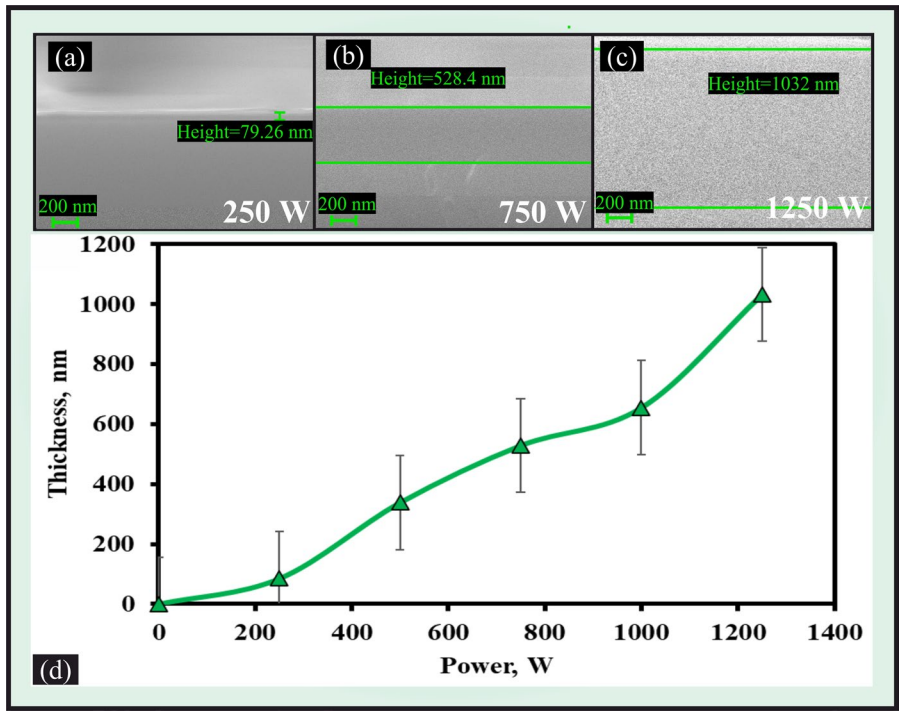

Fig. 1 SEM images of the Si with the resulting films depending on the RF power. **a** 250 W; **b** 750 W; **c** 1250 W; **d** plot of dependence the thickness on RF power

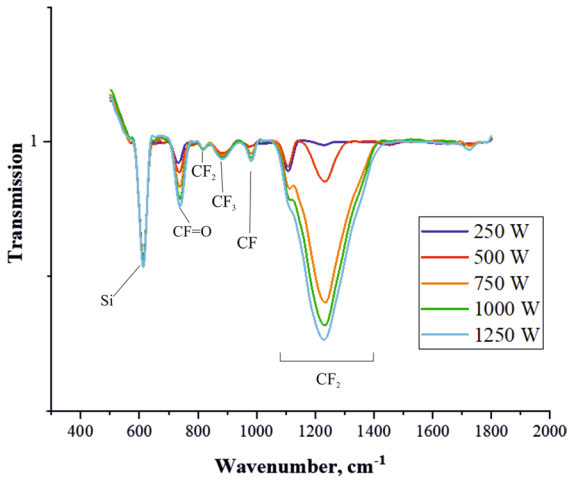

Fig. 2 FTIR-spectra of the resulting films depending on the RF power

Thread thickness was 100 µm. 1 cm^2 of material contains 20 warp and weft threads. Sample sizes were 10×10 mm and 10×150 mm. Also, single-crystal silicon substrates were used to control the chemical composition and thickness of the films.

Table 1 The absorption bands interpretation of the FTIR spectrum of Si with the resulting film

Analyze peak, cm^{-1}	Peak interpretation
610	Multiphonon absorption bands of single-crystal Si
725	Fluctuations of CF=O
819	Doublet stretching vibrations of the CF_2 bond
890	Deformation vibrations of the CF_3 bond
1105	CF stretching vibrations
1154	Stretching symmetric vibrations of CF_2 groups
1235	Stretching asymmetric vibrations of CF_2 groups

Methods

Preliminary preparation of the cotton fabric samples included treatment in a solution of caustic soda. A solution was prepared at a concentration of 240 g l^{-2}. The cotton fabric samples were placed in the solution for 3 min at room temperature, then washed in cold distilled water and dried without wringing.

The treatment was carried out in a low pressure inductively coupled plasma. The processing was carried out on an installation, a detailed description of which is given in the article Osipov et al. (2020).

Octafluorocyclobutane C_4F_8 (GOST TU 2412-128-05807960-96) was used as the agent gas. In the installation, the discharge chamber acts as an inductively coupled plasma generator and is located in the upper part of the reactor. There was also a second reaction chamber with an inspection hole, in which the samples were placed on the table. The generator frequency was 6.78 MHz. The GTVE-1000 generator was used as a source of RF electromagnetic power. The high frequency power generator (13.56 MHz) was used to create a negative self-bias on the substrate holder.

The measurement

The hygroscopic properties of cotton fabric were investigated after processing. The wettability was monitored by measuring the contact angle using the sitting drop method with a measurement error of 1.5% of the reading. Measurements were taken with the DSA 30 Kruss instrument.

The moisture content and the water absorption were determined according to standard testing (GOST 3816-81 or ISO 6741-1). The arithmetic mean of the results of two determinations was taken as the final test result, which was calculated with an error of not more than 0.01%. To determine the capillary absorption (standard test method DIN 53924) of textile materials, it was necessary to prepare 15 × 200 mm strips samples (3 pcs.).

The method of X-ray photoelectron spectroscopy (SPECS HAS 3500) was used to study the surface composition of textile materials. The survey spectra were recorded with a step of 1 eV, the spectra of individual lines with a step of 0–1 eV. We used Mg K_α radiation (1253 eV). The X-ray energy line width without monochromatization was about 0.5 eV.

The surface morphology of the samples and the thickness of films were studied using a SUPRA 55VP-25–78 scanning electron microscope with a resolution capability of 5–10 nm.

The FTIR spectra were obtained on an FSM 1201 IR-Fourier spectrometer. The spectra were taken in the absorption mode, the number of points was 20, the wavenumber range was 400–1800 cm^{-1}.

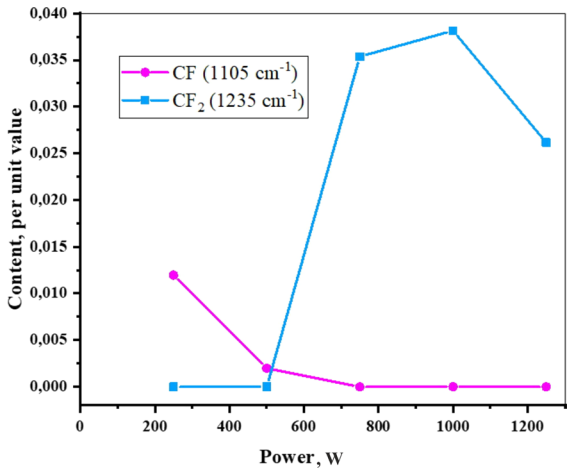

Fig. 3 The plot of the dependence of content of the components CF and CF_2 in the resulting films on the RF power

Results and discussions

Due to the critical role of RF power in textile processing, a series was first conducted to study the effect of power on hygroscopic properties. Then the influence of a process time on the resulting films was studied. The thickness and composition of the resulting films were estimated from Si substrate, which was used as a satellite sample.

Influence of applied power

The first series of experiments was aimed at studying the properties of the resulting hydrophobic films depending on the RF power applied to the gas discharge. After processing, the cotton fabric samples were used to measure the moisture content, the water absorption, the capillary absorbency, and the contact angle. The value of the hygroscopic properties of initial cotton fabric was as follows: the contact angle was 0°, the capillary absorption was 4 mm, the water absorption was 24.59%, and the moisture content was 2.7%. The experiments were carried out at a fixed pressure of 2 Pa, a flow rate of C_4F_8 24.5 sccm, a process time of 10 min, a bias voltage of −12 V, and the RF power varied from 250 to 1500 W, the distance from the plasma initiation zone to the substrate holder is 150 mm. The thickness of the resulting films was evaluated by SEM, the images are shown in Fig. 1.

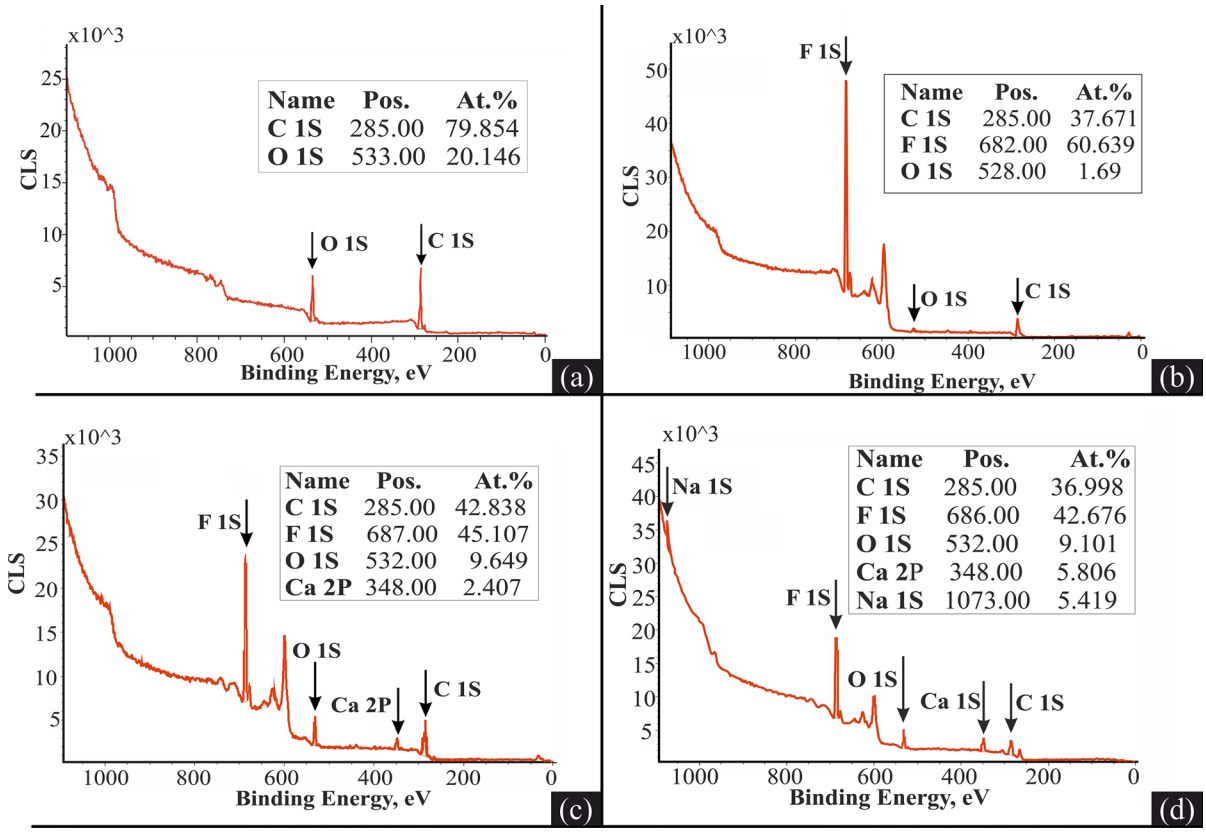

Fig. 4 XPS spectra of cotton fabric samples depending on the RF power. **a** Initial cotton fabric; **b** 250 W; **c** 750 W; **d** 1250 W

Based on the obtained SEM images, it can be concluded that an increase in the RF power leads to an increase in the thickness of the resulting film. We can say that a linear dependence is observed, the maximum film thickness obtained was 1032 nm at an RF power of 1250 W. The thickness of the resulting films was used to calculate the content of chemical components obtained from the FTIR spectra that make up the film. The obtained FTIR spectra of Si with a film are shown in Fig. 2.

When conducting a study of samples with the resulting film on an IR-Fourier spectrometer, spectra that include the absorption bands presented in Table 1 were obtained.

The absorption bands interpretation of the FTIR spectrum was carried out with the help of articles Denes et al. (1999), Andersen et al. (2003), Satulu et al. (2019). The obtained FTIR spectra show absorption bands inherent in the silicon substrate (610 and 1105 cm^{-1}), as well as absorption bands of fluorine-containing components. It should be noted that the fluorine-containing component is CF_2, indicating that the film has the nature of Teflon and is Teflon-like. In the obtained spectra, depending on the increase in the RF power applied to the gas discharge, a change in the intensity of the absorption bands 1105 and 1235 cm^{-1}. To assess the changes in the relative content of the components in the obtained films, the integration of each peak was carried out and the relative content of the components was determined. The content of the components was calculated by the ratio of the integral of the absorption band function to the thickness of the resulting film. The dependence of the relative content of the components in the films is shown in Fig. 3.

Thus, based on the dependencies obtained, it can be argued that an increase in power leads to an increasing part of the silicon surface being "overlapped" by a CF_2 component, but the CF bonds are

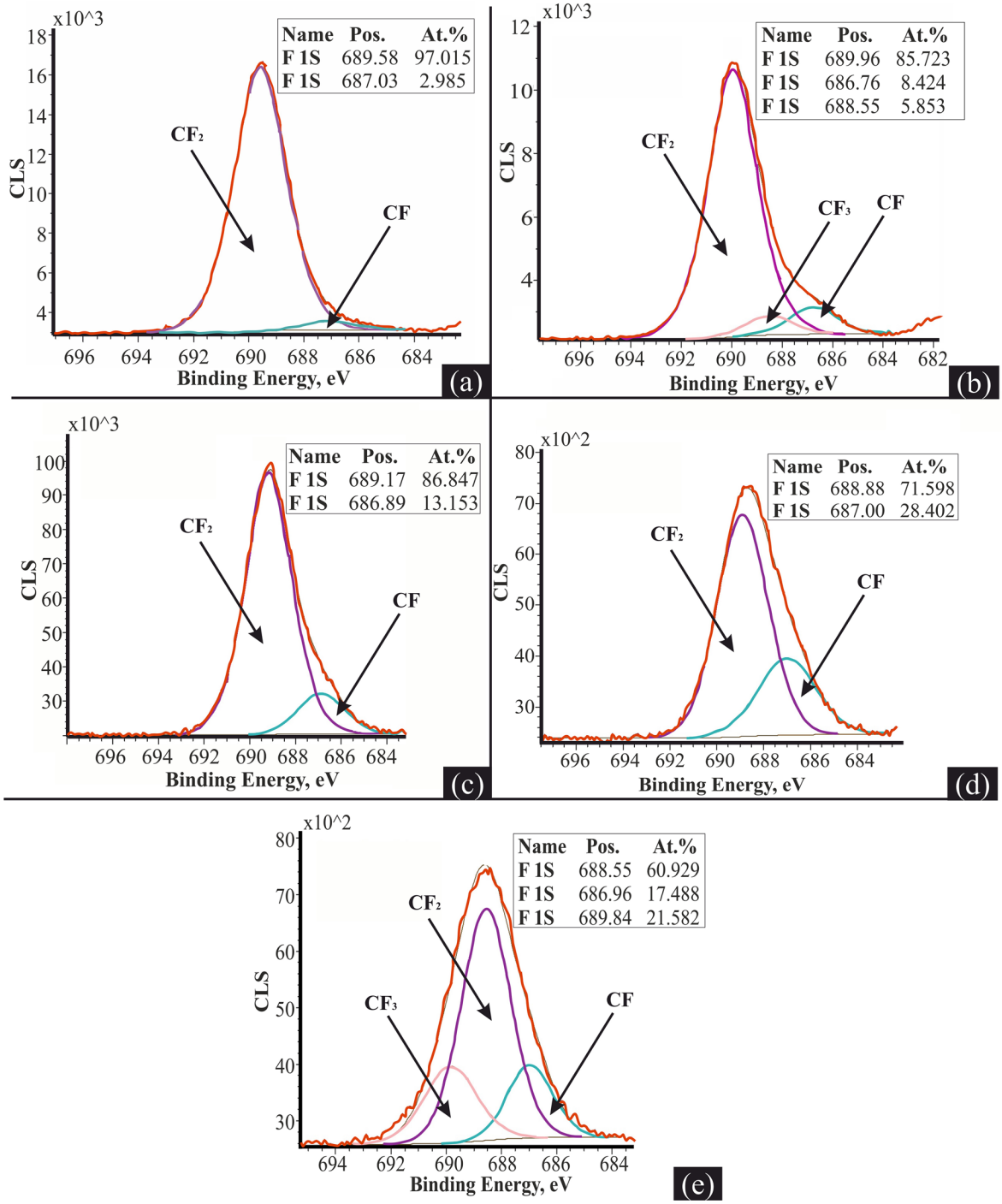

Fig. 5 Fluorine peak decomposition of cotton fabric after C_4F_8 ICP. **a** 250 W; **b** 500 W; **c** 750 W; **d** 1000 W; **e** 1250 W

still present, and after an increase in power above 500 W, CF bonds disappear altogether.

All samples of the cotton fabric with the resulting film were examined using X-ray photoelectron spectroscopy. The general spectra obtained from

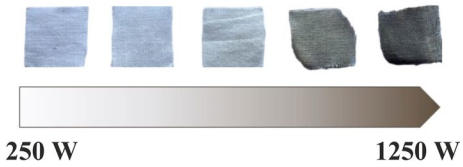

Fig. 6 External changes in the cotton fabric sample with an increase in RF power

some samples are shown in Fig. 4. Based on the spectra obtained, it can be concluded that each of the treated the cotton fabric samples contains carbon and oxygen components, which corresponds to the composition of the cotton fabric fiber, as well as the fluorine component, which indicates the presence of a fluorine-containing film on the surface, since this component is not observed in the spectra in the initial samples. Additionally, Fig. 4c–d shows the presence of a calcium component which is a contamination that got to the surface during the exposure of the samples to the atmosphere. A calcium component is observed on samples treated at 750–1250 W.

In Fig. 4d, among other things, the sodium component is observed in an amount of 5.4 at%. It is worth noting that the preparation of samples included their treatment in a solution of caustic soda, and sodium-containing components could linger in the fibers of the cotton fabric. The nitrogen component present in the spectrum can be neglected, since the atomic content is less than 1%, which is pollution.

To determine the bonds with fluorine, the peaks of fluorine were examined in more detail. The decomposition of fluorine peaks and determination of the correspondence of binding energies to certain

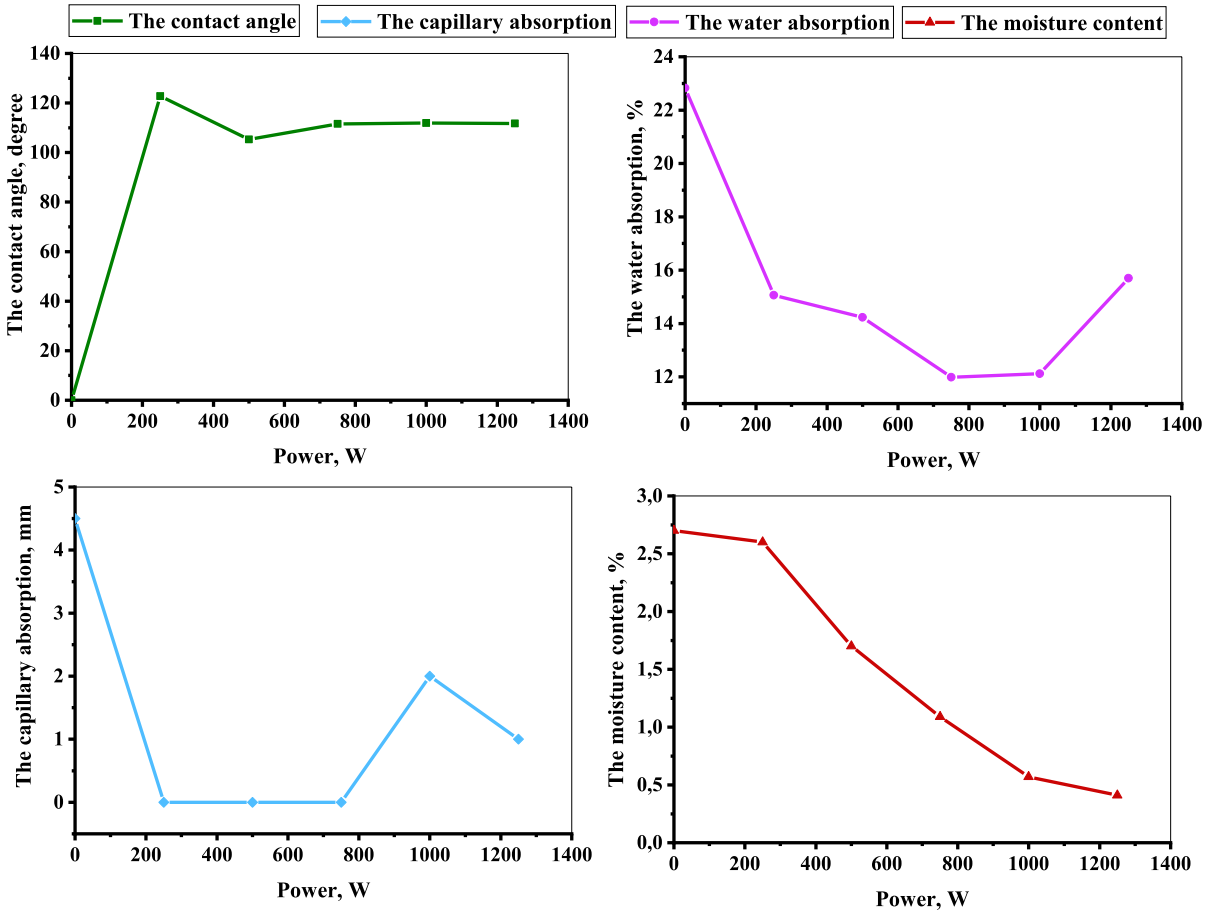

Fig. 7 Dependencies of hygroscopic properties on the RF power

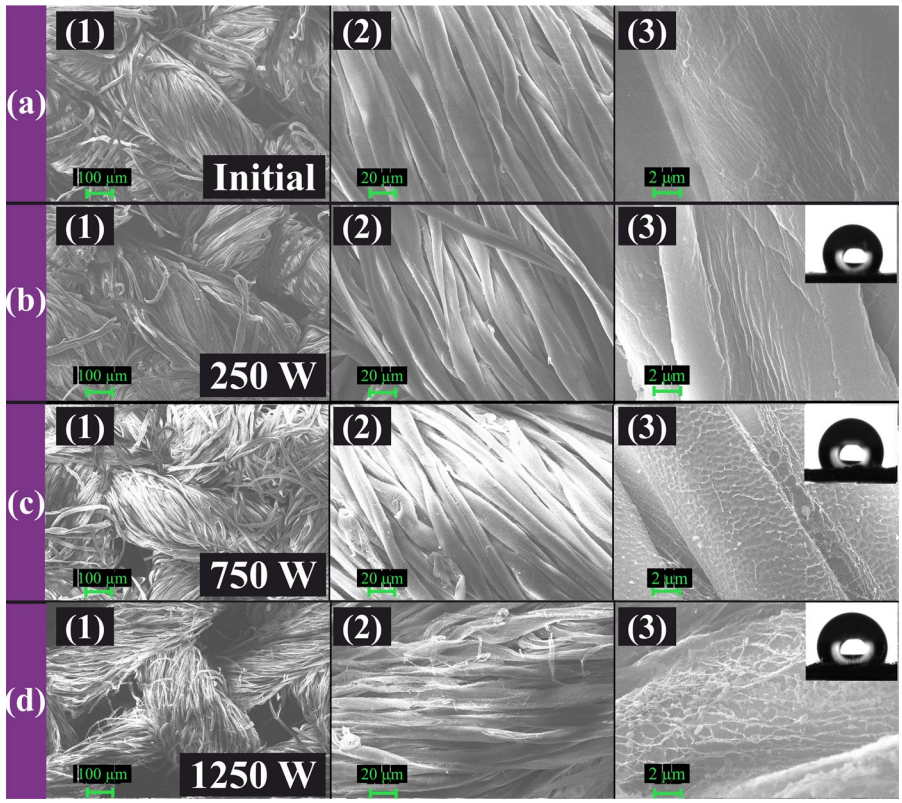

Fig. 8 SEM images of cotton fabric processed under at different RF power

components was carried out. Decompositions of the fluorine peaks for each sample are shown in Fig. 5.

Based on the decomposition of the fluorine peaks of each of the samples, it should be noted that several components were observed in the obtained films. The cotton fabric sample processed at 250 W contains mainly CF_2 components in the composition of the resulting film in an amount of about 97%, the remaining 3% correspond to the CF component. In the sample treated at 500 W, an additional CF_3 component was observed, the content of which is approximately 5.8%. Also, the CF_3 component was observed in the experiment with a power of 1250 W; in the other experiments, this component was absent. However, it is important to note that the XPS study is complicated by the fact that the tissue is translucent, and the substrate contributes to the lines of oxygen, carbon and fluorine, so undetectable lines may appear. The highest content in the obtained films is observed for the CF_2 component, the presence of other fluorine-containing components can indicate a different degree of decomposition of the C_4F_8 gas and the formation of various radicals in the plasma. It should be noted that for more correct data on the composition of the obtained films, it is better to carry out XPS analysis of silicon substrates rather than textile samples. To determine the homogeneity of the obtained films, XPS studies were carried out at two different scanning angles: 20° and 90°. There is practically no difference in the spectra obtained, from which it can be concluded that the resulting films are homogeneous, at least to a depth of 100–150 Å, since it is precisely this thickness that is studied during XPS.

It should be noted that carrying out processes at powers above 1250 W for cotton fabric is irrational, since an increase in power has led to a change in color and destruction of the integrity of the fibers (Fig. 6). The color of samples processed at powers of 250–500 W does not change, but a further increase in power already leads to a change. This may be due to the etching of the sample surface during the process, as well as its possible heating.

Dependencies of the contact angle, water absorption, moisture content and capillary absorption of the samples on RF power applied to the gas discharge are shown in Fig. 7.

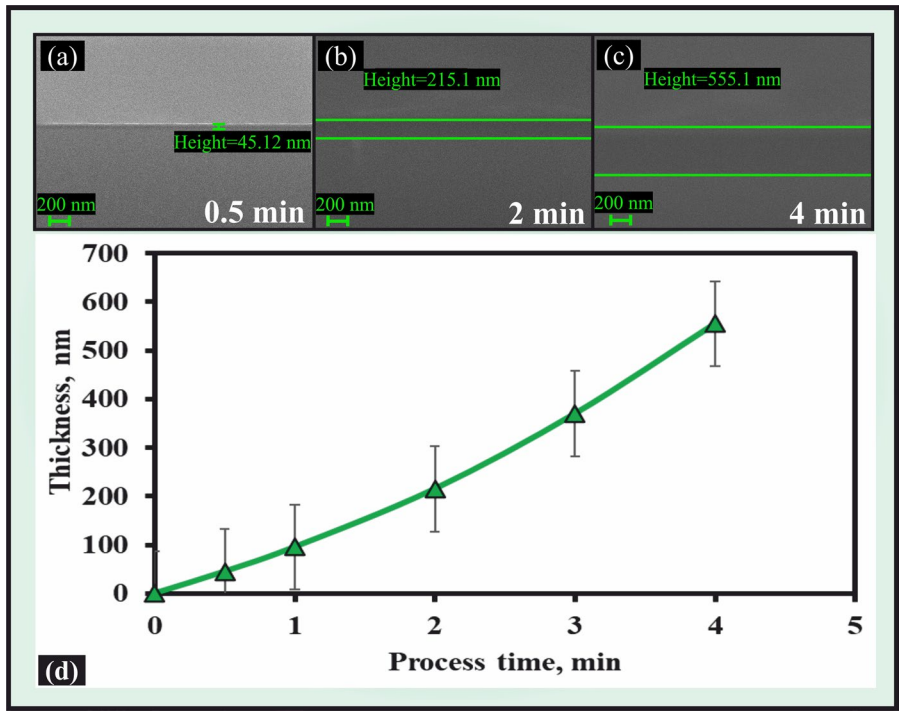

Fig. 9 SEM images of the Si with the resulting films depending on the process time. **a** 0.5 min; **b** 2 min; **c** 4 min; **d** plot of dependence the thickness on process time

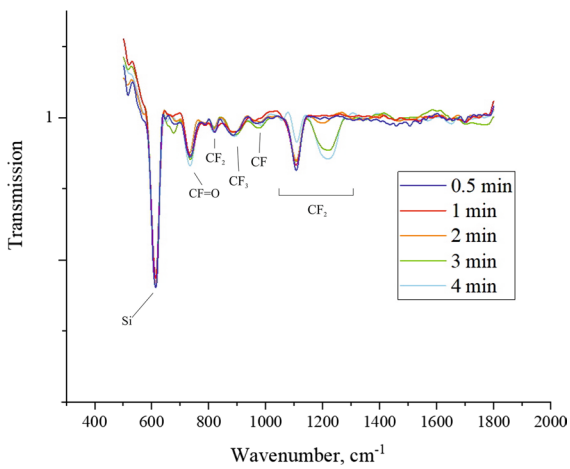

Fig. 10 FTIR-spectra of the resulting films depending on the process time

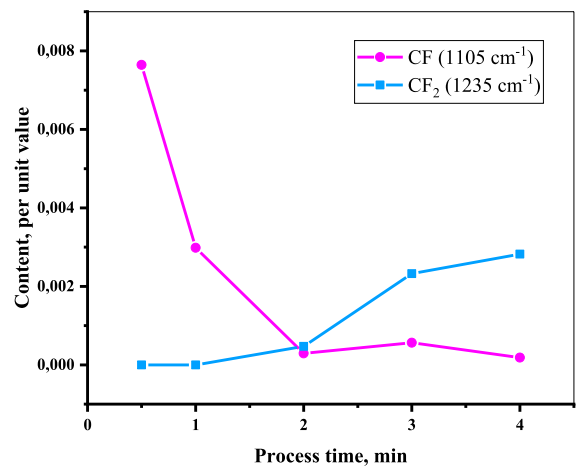

Fig. 11 The plot of the dependence of content of the components CF and CF_2 in the resulting films on the process time

The results obtained indicate that the creation of a hydrophobic surface is already observed starting from a power of 250 W. The maximum obtained contact angle is 123°, and a further increase in power does not lead to its growth, but only a decrease to 112° is observed which can indicate a change in the surface relief of the cotton fabric. It is worth noting that such parameters as capillary absorption and moisture content became completely minimal after treatment, and a change in power no longer led to any changes. Water absorption is reduced to 12% after treatment. Thus, a decrease in the hygroscopic characteristics of cotton fabric such as water absorption, moisture and capillary absorbency indicates the obtainment of a film that covers the existing pores and capillaries, thus preventing moisture from being absorbed into

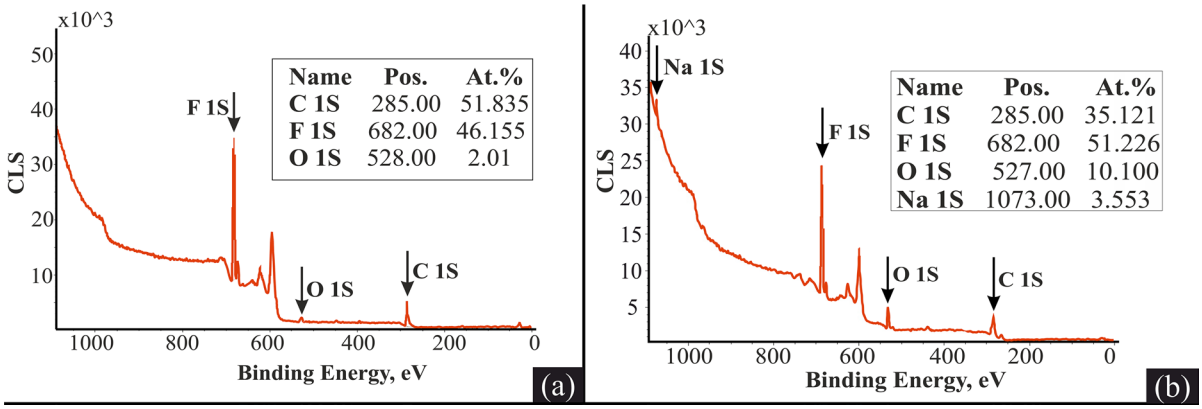

Fig. 12 XPS spectra of cotton fabric samples depending on the process time. **a** 0.5 min; **b** 4 min

the bulk of the material. To assess the surface morphology of the treated cotton, the samples were studied on SEM, the resulting images are shown in Fig. 8.

The obtained images from the scanning electron microscope (SEM) of the processed samples also indicate a violation of the integrity of the structure. In Fig. 8, the images are shown at various magnifications (under the numbers 1,2,3), which allows us to conclude both local etching of individual fibers and violation of the integrity of the fabric structure. At a power of 250–500 W, no noticeable destruction and etching of the samples is observed in the SEM images; starting from a power of 750 W, local etching of the fibers of the samples begins, which is clearly expressed in the form of a "network" pattern in Fig. 8c–d. In Fig. 8d2, thinning and breaking of the sample threads is observed. Thus, it can be concluded that at a power of 1250 W, the thickest film is observed, consisting mainly of the CF_2 component, while the surface is hydrophobic and moisture content, capillary absorption and water absorption are minimal, but it should be noted that with such a long treatment, the surface cotton fibers begin to be etched, which can significantly affect the strength characteristics of the fabric, so it is important to study the effect of processing time in cotton ICP at maximum power in order to obtain a thick and durable film and not destroy the surface of the material.

Influence of the process time

The second series of experiments is a change in the processing time of the cotton fabric in ICP with other technological parameters fixed. The pressure, bias voltage, RF power applied to the gas discharge and flow rate of C_4F_8, the distance from the plasma initiation zone to the substrate holder in the ongoing series of experiments were fixed and amounted to 2 Pa, − 12 V, 1250 W, 24.5 sccm, 150 mm respectively. The processing time ranged from 0.5 to 4 min. The thickness of the resulting films was evaluated by SEM, the images are shown in Fig. 9.

An increase in the process time leads to a linear increase in the thickness of the resulting films on the surface. The FTIR spectra of the obtained films are shown in Fig. 10.

The FTIR spectra (Fig. 10) show similar absorption bands, which are shown in Table 1. The spectra show that changes in intensities are observed only for the absorption bands at 1105 and 1225 cm^{-1}. Thus, the amounts of these components in the resulting films were considered. The dependence is shown in Fig. 11.

This dependence indicates that, just as with an increase in power, an increase in the content of the CF_2 bonds in the resulting films and a decrease in the content of the CF bonds are observed.

The cotton fabric samples were examined using XPS, the spectra of some samples shown in Fig. 12. It is worth noting that the spectrum contains components corresponding to the composition of the cotton fabric material (carbon and oxygen), as well as the fluorine component.

The fluorine component is observed on each sample, which indicates the presence of a film on the surface. To clarify the composition of the obtained films,

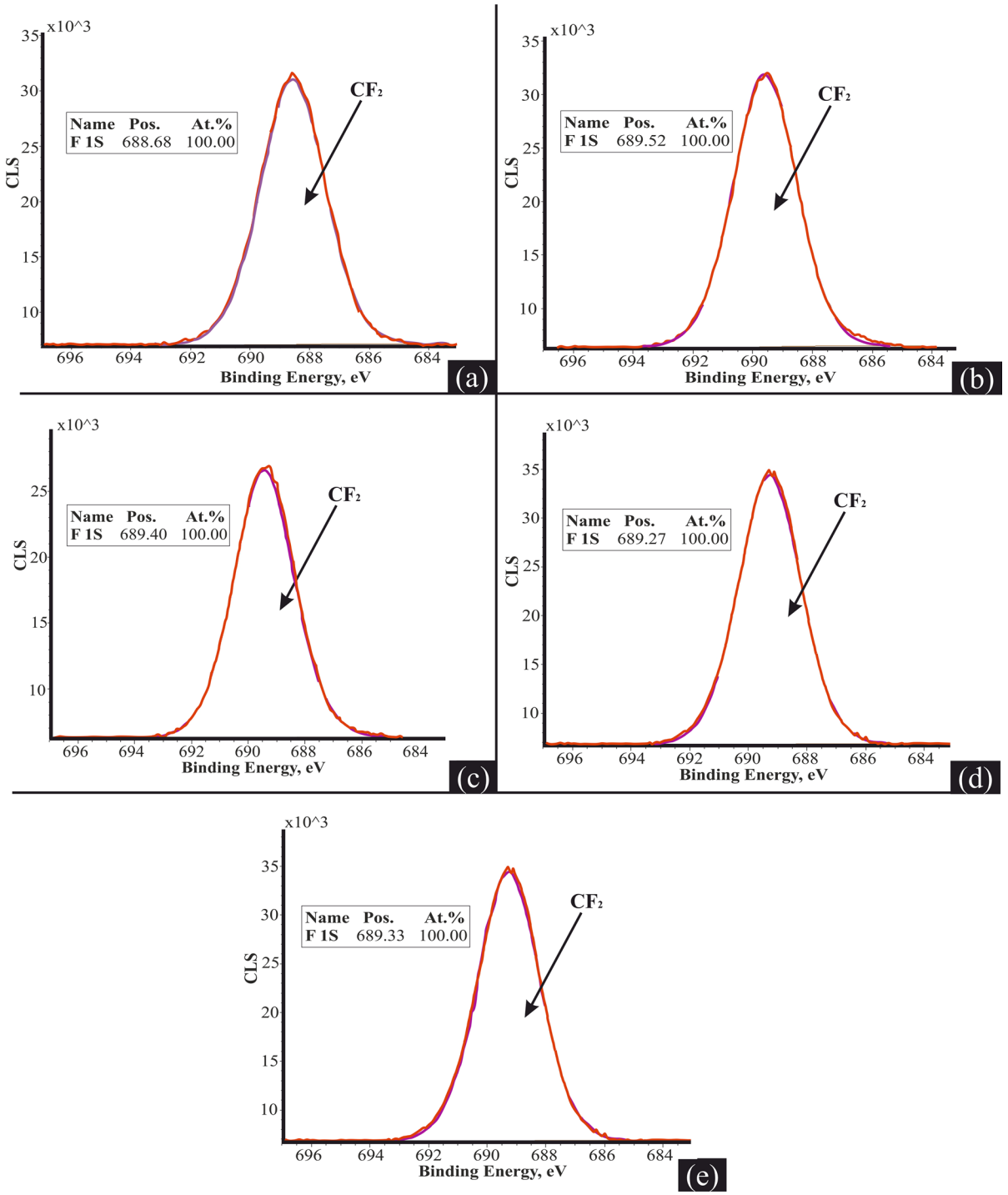

Fig. 13 Fluorine peak decomposition of cotton fabric after C_4F_8 ICP. **a** 0.5 min; **b** 1 min; **c** 2 min; **d** 3 min; **e** 4 min

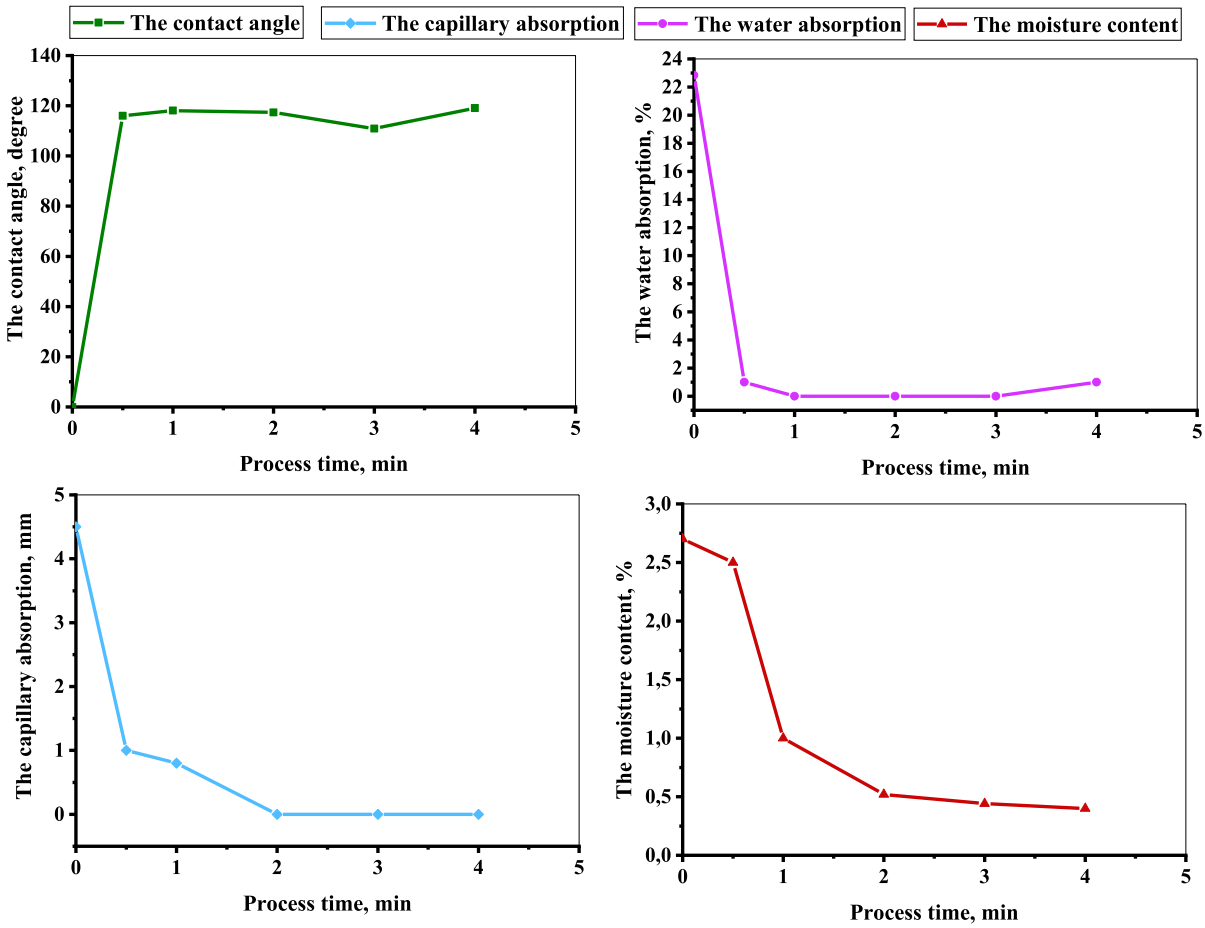

Fig. 14 Dependencies of hygroscopic properties on processing time

the fluorine peak was considered more carefully, and its decomposition into components was carried out (Fig. 13).

Based on the decomposition of the fluorine peak, it can be said that the resulting film mainly consists of CF_2 components, and therefore is Teflon-like. It is important to note that the CF and CF_3 components are missing during this treatment, which may be due to the decomposition of C_4F_8 in plasma mainly into CF_2 radicals. This indicates that under such modification conditions, a pure Teflon film is obtained without additional components. Dependencies of the contact angle, water absorption, moisture content and capillary absorption of the textile samples on the process time are shown in Fig. 14.

The results obtained indicate that the treatment of the cotton fabric in plasma even for 0.5 min leads to some hydrophobization of the surface, since a sharp increase in the contact angle is observed. We can talk about the formation of a hydrophobic film, since capillary absorption and water absorption also decrease and reach the value 0. The moisture content during such processing changes slightly, but a further increase in process time leads it to a minimum value and is 0.5%. The maximum obtained contact angle is 120º at a plasma treatment time of 4 min. The surface morphology of the treated samples was studied using SEM and the resulting images are shown in Fig. 15.

It should be noted that, in contrast to the treatment at 10 min (Fig. 8), the network structure of the fibers is observed only at 4 min of treatment. At process times from 0.5 to 3 min, no changes are observed on the surface of the fibers, which means that the surface is not etched, and such processing is not destructive.

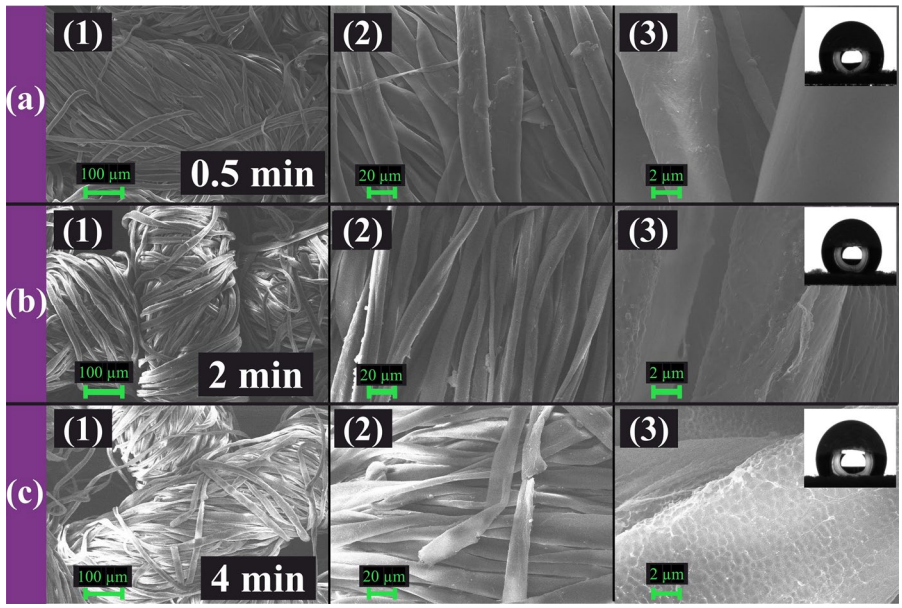

Fig. 15 SEM images of cotton fabric processed under at different process time

Thus, it can be concluded that with a RF power of 1250 W, non-destructive processing will be processing for 3 min, a further increase the process time leads to etching of the surface. With a three-minute treatment, a Teflon-like film with a thickness of 370 nm is obtained, with a wetting angle of about 120º and other hygroscopic properties have their minimum value.

Conclusions

The modification of the cotton fabric in ICP in a C_4F_8 gas environment leads to surface hydrophobization. The hydrophobization of the surface is provided by the formation of a Teflon-like film, which mainly consists of CF_2 groups, fixed using FTIR and XPS analyses. With an increase in RF power applied to the gas discharge, an increase in the thickness of the resulting film is observed, the maximum film thickness obtained is 1032 nm. The resulting contact angle of the treated cotton fabric is from 118 to 123º. Water absorption, capillary absorption and moisture content with increasing RF power are reduced to the lowest possible values. But it is worth noting that at RF power applied to the gas discharge from 750 to 1250 W at a processing time of 10 min, material destruction and a change in its color from white to brown are observed. SEM images are shown local etching of the fibers. Therefore, it can be made that the processing of the cotton fabric with such parameters is not applicable. Reducing a processing time avoids destruction of the processed material. In the SEM images, the etching of the cotton fabric fiber surface is observed only after processing for 4 min. It should be noted that modification at a power of 1250 W and a time of 0.5 to 4 min leads to the formation of a pure Teflon film on the surface of the cotton fabrics without impurities of other components.

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Declarations

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