Research Article

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Stable super-hydrophobic and comfort PDMS-coated polyester fabric

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Abstract: Super-hydrophobic fabrics have shown great potential during the last decade owing to their novel functions and enormous potential for diver's applications. Surface textures and low surface energy coatings are the keys to high water repellency. However, the toxicity of nanomaterials, long perfluorinated side-chain polymers, and the fragile of micro/nano-texture lead to the super-hydrophobic surfaces are confined to small-scale uses. Thus, in this article, a stable polydimethylsiloxane (PDMS)-coated super-hydrophobic poly(ethylene terephthalate) (PET) fabric (PDMS-g-PET) is manufactured via dip-plasma crosslinking without changing the wearing comfort. Benefiting from the special wrinkled structure of PDMS film, the coating is durable enough against physical abrasion and repeated washing damage, which is suffered from 100 cycles of washing or 500 abrasion cycles, and the water contact angle is still above 150°. This study promotes the way for the development of environmentally friendly, safe, and cost-efficient for designing durable superhydrophobic coatings for various practical applications.

Keywords: super-hydrophobic, plasma crosslinking, washing durability, abrasion resistance, wearability

1 Introduction

Super-hydrophobic surface commonly existed in the surface of bios, well-known as lotus leaves, butterfly wings,

and duck feathers (1), and was highly attracted by academia and industry for decades due to its tremendous potential applications of water repellency (2,3), anti-icing (4), self-cleaning (5), and anti-drag under water (6) in the industry of textile, coating, and aviation. Inspired from biostructure, various artificial super-hydrophobic surfaces were successfully produced by constructing nano/micro roughness from top-to-down or down-to-top methods and low surface tension surface from *in situ* process (7) and chemical postmodification (8,9). However, the insufficient mechanical robustness of artificial super-hydrophobic surfaces cannot bear the practical application. To overcome this problem, several strategies were provided by researches. High strength or hardness metal or ceramic nano/micro-structures were produced by chemical etching (10) or laser ablation (11) could increase the mechanical damage threshold. Thicker coating or block materials, which were entirely composed of super-hydrophobic nanoparticles (12) and slight binder (13), can also extend the abrasion lifetime because it provided many sacrificial layers. Moreover, biomimicking bios' regrowth when their microstructures are broken, low surface tension silane in microcapsules or micropores can remodify the destroyed structure to recover super-hydrophobicity (14). These strategies can certainly improve the robustness of super-hydrophobic coatings, membranes, or blocks; however, they cannot be applied on fabric because they cannot satisfy the basic demands of fabric, e.g., softness, permeability, washability, and skin contact safety.

Considering the comprehensive demands of water-repellent fabric, normally industry hydrophobic fabric was directly coated by hydrophobic polymer-like polydimethylsiloxane (PDMS) (5), poly(acrylic acid) (15), polyurethane (PU) (16), Ecoplus (Rudolf Gruop), and Zelan R3 (DuPont) with alkyl group (17) or perfluorinated side chain (18). Owing to the insufficient roughness, industry products cannot achieve very high-water repellency, which normally has a water contact angle (WCA) ~150° with a low sliding angle. Furthermore, high roughness obtained by adding or *in situ* growing nanoparticles (NPs) on fibers, e.g., silica NPs (19) and ZnO nanorods (20), could achieve super-hydrophobicity. Most reports

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showed that NP-coated fabric achieved robustness for hundreds of abrasion and washing cycles. However, these NPs were easily taken away from fibers by increased friction force due to the high roughness. Leaving aside the safety of detached nanoparticles on human skin, the rigid nanoparticles with polymer binder on fibers cannot provide enough mechanical robustness for practical applications. Furthermore, due to perfluorinated chains of eight carbons or more, have been shown to be persistent in the environment and bio-accumulate in living organisms (21), prohibition of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) in fabric cause the increasing interest of fluorine-free water-repellent agent. Thus, developing highly robust super-hydrophobic fabric with less change of fabric styles using fluorine-free materials is a great challenge.

PDMS with identical terminating methyl groups is elastic silicon with good biocompatibility and environmentally friendly and possesses excellent strength, wear resistance, and low surface energy (20–25 mN/m), which turned it into a good hydrophobic material (22,23). Utilizing patterning approaches, nano/micro PDMS pillar arrays, micro-winkles, and hierarchical structures like pillars on wrinkles are fabricated to form super-hydrophobic surfaces (24). Here, we developed PDMS wrinkles on fibers by radiofrequency capacitance-coupled plasma (RF-CCP) treatment of PDMS precursor-coated fabric. After plasma treatment, the fabric has extremely good super-hydrophobicity without any fabric style change, while traditional silicon treated fabric is sticky. Due to the wrinkle structures, plasma-treated PDMS-coated fabric has high mechanical robustness for more than 500 abrasion cycles and 100 washing cycles.

2 Materials and methods

The knitted polyester fabrics (100%, 120 g/m²) purchased from Miandu Textile Co., Ltd. (Zhejiang, China) were used as samples. The detergent 209 was purchased from Wangnilai Co., Ltd. (Guangzhou, China). PDMS, trimethylsiloxy terminated, M.W. 770 (CAS#9016-00-6) supplied by Alfa Aesar Tech. Co., Ltd. (Shanghai, China) was used as a monomer. The standard soapflake purchased from China textile institute of science and technology (Beijing, China). Ethanol (AR, ≥99.7%) and Argon gas were supplied by Changzhou Hongsheng Fine Detail Co., Ltd (Jiangsu, China) and Canghai industry Gas Co., Ltd. (Shanghai China), respectively.

2.1 Polyester samples preparation

First, the PET fabric was washed to remove any possible dust or chemical residues, which can probably affect the surface treatment. Second, it was immersed into 2 g/L detergent 209 and 2 g/L sodium carbonate with the bath ratio of 50:1 at a temperature of 40°C in the ultrasonic bath for 40 min. Then, it was washed repeatedly with deionized water and dried in an oven at a temperature of 70°C for 2 h.

2.2 Fabrication of super-hydrophobic PET fabrics

The scoured PET samples were dipped into a beaker containing 5 g/L PDMS monomer in the ultrasonic bath at a liquor ratio of 40:1 for 5 min. Subsequently, the samples were dried in an oven at the temperature of 70°C for 30 min to move the solution of ethanol. The dipped PET samples were then modified by the radiofrequency under the CCP mode, and the processing of plasma treatment for super-hydrophobic fabrics was the same to our previous article (25).

2.3 Characterization and simulations

Surface morphology was characterized by scanning electron microscope (SEM; Hitachi S-4800, Hitachi, Japan) and atom force microscope (AFM, 5500AFM-SPM, Aglient, America). Surface chemical compositions of samples were analyzed by X-ray photoelectron spectroscopy (XPS; Kratos AXIS UltraDLD, Shimadzu, Japan). The static WCA was measured on the DropMeter™ Professional A-200 instrument (Ningbo Haishu Meishi Detection Technology Company, China) with the volume of deionized water droplets were 5 µL according to GB/T 30693-2014, and sliding angles were measured at purpose-made stage with 10 µL deionized water. The washing and abrasion durability of samples was evaluated by the WCAs after several washes (according to AATCC 61-2006. 2A, SW-8, Mebon Instrument Co. Ltd., China) or abrasion (according to ISO 105-X12:2001, dry friction mode, Y571N, Nantong Hongda Instrument co., China) cycles, respectively. The wearing comfort of fabric is determined by the water vapor transmission (GB/T 12704.2-2009, YG601H, Ningbo Textile Instrument Factory,

Zhejiang China), air permeability (GB/T5453-1997, YG461E, Wenzhou Fangyuan Instrument Co. Ltd. Zhejiang, China), and cold-hot sensation (FZ/T01054.1-6-1999, model KES-F7-II Band, Tokyo, Japan). The Kawabata Evaluation System for Fabric (KES-F) hand value instrument, type of YG821 (Shanghai textile research institute, China) and thermo-sensation instrument, type of KES-F7-II Band (Total, Japan) were used to test the wearability of fabrics according to FZ/T01054.1-6-1999, and all the samples were conditioned for 48 h under atmospheric conditions of $20 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ relative humidity before tested.

Finite element simulation was conducted using Commercial software Abaqus/Explicit. For simplicity, a conventional bilayer model for generating wrinkling that consists of stiff film and compliant substrate was implemented. Both film and substrate were simulated as linear

elastic material and by four-node plane strain elements. The indenter was modeled as a discrete rigid body. To show the benefit of wrinkles, the maximum contact pressure during contact in both cases was set to be identical and the Mises stresses were compared.

3 Results and discussion

Figure 1a shows the FTIR spectrum of PET fabrics before and after plasma treatment. The enhancement of Si-CH₃ (C-CH₃) peaks in 780, 1,250, and 1,410/cm, and C-O-C (Si-O-Si) peaks in 1,200–1,000/cm was attributed to the crosslinking of PDMS on the surface of PET fabrics. After plasma treatment, PDMS film was successfully coated on the surface of PET fabrics.

Figure 1: (a) The ATR-FTIR spectrum of the samples before and after treatment. (b) The water repellency of PET samples and the corresponding electron temperature and electron density under different powers. (c) The image of water contact with PET (top) and PDMS-g-PET (below) fabrics surface.

As we know, different setting values of radiofrequency (RF) power result in the change of electron temperature and electron density in the reaction chamber, and they play a leading role in modifying the material surface (26). Therefore, we used a double Langmuir probe to investigate the electron temperature and electron density in the reaction chamber during the discharge process, and the result is shown in Figure 1b. It was obvious that the electron temperature and electron density changed in the opposite trend. When the RF power was less than 125 W, the electron density was low, so we implied that the electron temperature was the major factor to influence the modification, and the WCA of the samples increased when electron temperature went down, and the water repellency of samples was observed to the best of WCA and SA up to 152.2° and 8.4° , respectively, at the RF power of 100 W. However, when the RF power was less than 50 W, the discharge process was unstable, and the samples could not exhibit high WCA. When the power was between 125 and 250 W, the electron density played the dominant role. The decrease of water repellency of the samples with the increasing electron density due to the increase of collision of active particles leads to the enhancement of plasma etching and may destroy the structural integrity of the PDMS drafted film. At the power of 250 W,

the WCA and SA were just 143.1° and 38.3° , respectively. Finally, when the RF power was higher than 250 W, the number of high-energy particles was reduced, and thus, fewer chemical bonds were broken.

As shown in Figure 1c and Video in Supplementary Material, PDMS-g-PET fabrics, which were treated at the power of 100 W, exhibited noticed different wetting behavior to PET fabric. After contact with water, the surface of PET fabrics was totally wet, while the PDMS-g-PET fabric stayed completely dry. This result indicated that PET fabrics got excellent repellency of water after PDMS was coated properly.

The SEM and AFM measurements were introduced to determine how the sample surface morphology changed before and after modification with different RF powers. As shown in Figure 2a and d, the PET fibers were smooth and free of defects, which accorded with the features of synthetic fibers. In contrast, PDMS-g-PET fabrics (Figure 2b–f) showed some differences. After treated by the power of 100 W (Figure 2b and e), a thin uniform film with some regular wrinkles film has appeared on the surface of PET fibers. This low surface energy film covered all the fibers on the surface of the sample, so it could exhibit super-hydrophobic properties. Nevertheless, the sample that was treated by 250 W (Figure 2c and f) did not have a

Figure 2: The SEM and AFM images and XPS survey spectra of (a, d, and g) PET and PDMS-g-PET fabrics with different RF power of (b, e, and h) 100 W, (c, f, and i) 250 W, respectively.

wrinkled structure on its surface and even exhibited some textures, which was caused by plasma etching. Therefore, the water repellency of this sample was much lower than the one treated by 100 W as shown in Figure 1a.

The chemical composition of the PET fabrics was analyzed by XPS and FTIR. As shown in Figure 2g, the pristine PET fabric was mainly composed of carbon, oxygen, and hydrogen. However, in Figure 2h and i, four peaks appeared at 100.8 eV (Si 2p), 150.2 eV (Si 2s), 284.3 eV (C 1s), and 531.1 eV (O 1s). Besides, the peaks of C–C (284.6 eV), C–O (286.4 eV), and C=O (288.8 eV) (21) bonds were also reduced to some extent. It may be because of the breakup of C–Si and C–O bonds in PDMS molecular chain by excited particles, and then chemical bond was formed with active sites on polyester fabric surface, and so there was silicon and less C–C, C–O, and C=O bonds existed on the PDMS-modified fabric. All these indicated that PDMS was successfully drafted onto the surface of PET fabric. Furthermore,

according to Figure 2g–i, the RF power had an influence on the element content and the percent peak area of C1s core level. Due to the drafting of the thin uniform PDMS film, C–Si component increased and other components decreased a lot at the power of 100 W. But when the power was at 250 W, the C–C, C–O, and C=O contents were all higher than treated by 100 W. It might be because that the high-energy particles were activated and destroyed the PDMS film and the polyester surface, leading to the increase of C–C, C–O, and C=O contents.

Figure 3a shows the washing durability of the PET samples before and after modification. The WCA value of untreated PET fabric and PDMS-*g*-PET fabrics, which was treated by RF power of 100 W, was changed from 120.1° and 153.2° to 87.5° and 150.6°, respectively, after 100 washing cycles. In addition, when the RF power was at 250 W, the WCA value changed from 141.3° to 127.12° at a steady rate. The abrasion durability of PET fabrics was also measured and the indenter with loading pressure of

Figure 3: (a) Washing durability and (b) abrasion stability of PET and PDMS-*g*-PET fabrics with different RF powers; surface morphology of PDMS-*g*-PET fabrics after different abrasion cycles with RF power of (c) 100 W and (d) 250 W.

44.8 kPa, which was typically used to evaluate fabrics for thin thickness garment usages (27). As shown in Figure 3b, PDMS-g-PET fabrics had better abrasion stability than the pristine one. The treated super-hydrophobic PET fabrics at 100 W got the best durability, and the WCA was still being 151.1° after 500 abrasion cycles, while the WCA of the sample treated at 250 W was just 113.3° . The SEM image with different abrasion cycles (Figure 3c and d) demonstrated that PDMS was peeled off from the fiber after 100 cycles for PDMS-g-PET fabrics with a power of 250 W. While it was found that the wrinkles were relaxed after 300 cycles, and only the short PDMS strips are taken off from the fiber after 500 cycles for PDMS-g-PET fabrics with the power of 100 W. It was supposed that the destruction of the structure and integrity of PDMS film was the main reason for the decrease of water repellency.

To further investigate the contribution of wrinkled PDMS coating during abrasion, the failure process was simulated by a finite element method. First, the damage of the surface film was divided into two steps: reversible wrinkle relaxation and irreversible coating damage (the schematic in Figure 4a) based on the observed SEM images. Figure 4b and c represents the effect of the wrinkle structure on the stress distribution of PDMS films. It is shown that wrinkles can disperse the force caused by the indenter, while flat films have stress concentration both at the surface and the interface by the shearing force (Figure 4c). This is consistent with the SEM images, which indicated the failure happened at the interface for PDMS-g-PET fabrics with the power of 250 W, while

the failure happened at the top surface for the fabric with PDMS-g-PET fabrics with the power of 100 W.

As presented in Table 1, the hand value (HV) for fabric softness changed slightly (0.183%) after plasma treatment. For stiffness and smoothness, the HV ranged from 5.350 to 5.459 and from 48.448 to 50.450, respectively. Yim and Kan (28) had pointed out that weight and thickness of warp-knitted fabrics did highly correlated with stiffness, and heavier and thicker fabric was stiffer consequently. In contrast to the aforementioned changes of fabric properties, the maximum transient flow $q_{\max}$ of treated PET fabric was decreased to 0.10 (−4.762%), it means that at the moment of contact between the fabric and the skin, the heat of the fabric leading away from the skin surface was reduced, and then, the comfort of the fabric was increased.

Besides the HV, the clothing comfort of costume textiles is also closely related to water vapor transmission and air permeability and is strongly influenced by the construction (such as pore size) and surface properties of fibers. The water vapor transmission and air permeability are considered the determining factors of the “breathability” of clothing textiles. For PET and PDMS-g-PET fabrics, the high “breathability” of pristine samples indicates the presence of numerous open holes and the moisture absorbability by fibers themselves, which assist the transmission and permeation of water vapor and air through the clothing as presented in Table 1. The slight increase of air permeability (2.29%) of treated samples might be caused by the etching of plasma and

Figure 4: (a) Failure process diagram of PDMS film surface; simulation of stress distribution and damage behavior of (b) plat film and (c) wrinkled PDMS film in abrasion test.

Table 1: The wearability of PET and PDMS-*g*-PET fabrics

Mechanical properties	Units	PET	PDMS- <i>g</i> -PET	Rate of change (%)	Performance improvement
Stiffness	—	5.350	5.459	2.032	↓
Fullness	—	30.908	30.965	0.183	↑
Smoothness	—	48.448	50.450	4.214	↑
Thermo-sensation	J/cm ² s	0.105	0.100	−47.619	↑
Air permeability	mm/s	1594.56	1631.06	2.29	↑
Water vapor transmission	g/m ² h	48.41	46.37	−4.21	↓

increased porosity of fabric. Conversely, the decline of water vapor transmission (−4.21%) of treated samples was caused by the formation of PDMS film, which reduced the conduction and adsorption of water vapor in the fabric. Depending on the wearability test results, we could conclude that plasma treatment had little effect on the wearability of the fabric. It is believed that plasma treatment only modifies several molecular layers (about 10–20 nm) of fabric surface, without changing the bulk properties of the fabric (29).

4 Conclusion

In this article, a stable PDMS-coated super-hydrophobic poly(ethylene terephthalate) (PET) fabric treated by argon combined capacitively coupled plasma (CCP) was fabricated, and the relationship between RF power and the properties of plasma polymerized PDMS film was also discussed. It was observed that the best hydrophobicity was obtained at the power of 100 W and the worst at the power of 250 W. PDMS films polymerized and drafted to the surface under such conditions resulted in hydrophobic surfaces with WCAs of 153.2° and 141.8°, respectively. The surface morphology and chemical composition tests proved that the 100 W modified PDMS-*g*-PET fabrics had a thin and uniform wrinkled PDMS film drafted to its surface, which equipped it with good water repellency property, washing, and abrasion durability. Besides, super-hydrophobic treatment had little effect on the wearability of PET fabric. All mentioned earlier, a simple, low-cost, and environmentally friendly fabricating technology was found to obtain a stable robust and comfort super-hydrophobic polyester fabric.

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Chengjiao Zhang: visualization, project administration, resources.

Conflict of interest: The authors state no conflict of interest.

Supplementary material: Video presenting the contact state of water flow with PET and PDMS-*g*-PET fabrics surface.

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