

Article

Superhydrophobic Fabrics with Mechanical Durability Prepared by a Two-Step Plasma Processing Method

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Abstract: Most studies on superhydrophobic fabrics focus on their realization using additive manufacturing (bottom-up) techniques. Here we present the direct modification of three different fabrics using a plasma-based method to obtain anti-adhesive and self-cleaning properties. A two-step plasma processing method is used: (a) for the creation of micro-nanoscale features on the fabric surface (plasma texturing step) and (b) the minimization of the fabric surface energy (by a short plasma deposition step of a very thin, low surface energy layer). The entire process takes only 14 min and all fabrics after processing exhibit high water static contact angles (WSCA > 150°), low contact angle hysteresis (CAH < 7°) and advantageous mechanical durability against hand-rumpling. The method is simple and generic, and it can be therefore expanded to other polymeric fabrics (i.e., acrylic) in addition to polyester, without any limitation rising from the weaving characteristics of the fabric or the starting nature of the material (i.e., hydrophobic or hydrophilic).

Keywords: plasma micro-nanotexturing; superhydrophobic fabrics; mechanical durability

1. Introduction

A lot of attention has been recently given on optimizing the properties of fabrics. It is clear that clothing is the most important industry for fabrics; however fabrics can also be used in other interesting applications (i.e., oil-water separation [1]). Polymeric fabrics and textiles with special functionalities should have durability against laundry wear, dry cleaning and other mechanical wear stresses [2–4], if they are intended to be used for clothing. For this reason, such durability tests have become very popular [5]. Functionalities which emerge from surface engineering, such as antibacterial [6,7] or non-staining properties [8] have attracted a lot of attention recently and many ideas have already been applied in polymeric fabrics [8,9].

Most of the superhydrophobic fabrics are prepared after coating with a superhydrophobic coating (additive manufacturing) [2] rather than treating the fabric itself. Such an example is the superhydrophobic PDMS/ODA-coated PET fabric presented by Xue et al. [10]. Similarly, robust superamphiphobic fabrics were prepared after coating with a solution comprising poly(vinylidene fluoride-co-hexafluoropropylene), fluoroalkyl silane, and a volatile solvent. This fabric exhibited contact angles of 162°, 156°, and 150° for water, olive oil, and silicone oil, respectively [11]. In another similar approach, a superhydrophobic cotton fabric was prepared by introducing a fluorinated acrylate monomer, 1H,1H,2H,2H-nonafluorohexyl-1-acrylate, onto cotton fabric under simultaneous radiation. This fabric was also found to be durable against 250 domestic laundry cycles [12]. Zhou et al. [13]

reported a fabric that retained its superhydrophobic properties after evaluation using a standard protocol (AATCC Test Method 61-2006 test No. 2A) and no significant changes in water contact angle, water sliding angle and topography were observed after 500 washing cycles. Zimmermann et al. [14] presented superhydrophobic textile fabrics with durability against immersion in water, whereas Zeng and coworkers [5] presented cotton fabrics coated with a mixture of SU-8 photoresist, a fluorinated alkylsilane and silica nanoparticles that exhibited superhydrophobic properties and durability against long immersion times in organic solvents, as well as acid and base solutions.

As stated above, another interesting aspect for the clothing industry is the creation of antibacterial clothes. Approaches to control the bacteria adhesion have been recently reviewed [15], and it is apparent that again additive techniques are very popular. For example, superhydrophobic and antibacterial cotton fabric was fabricated after sol-gel coating and exhibited reduction of bacteria reaching 100% [16], while others [17,18] incorporated silver nanoparticles to prepare fabrics with antibacterial properties against both Gram-positive (*Staphylococcus aureus*) and Gram-negative bacteria (*Escherichia coli*). Except clothing, fabrics can also be used in other applications. For example, in oil-water separation; polyester fabrics coated with a 20 wt % fluorodecyl POSS/x-PEGDA blend have been presented [19], while cotton fabrics coated with vapor phase deposition of aniline have been presented by Zhou et al. [1].

In all these studies, the fabrics were prepared either by synthesis or by additive manufacturing, therefore direct modification of the fabrics is rare in the literature. This work focuses on the direct modification approach and as received commercially available polyester (PES) fabrics are transformed to superhydrophobic using plasma processing independently of their starting nature (both hydrophilic and hydrophobic fabrics are transformed to superhydrophobic).

The presented fabrics are intended to be used for covering wind turbine blades to grant them self-cleaning properties and for this reason they were processed in large pieces. To our knowledge, except for some scattered newsletters reporting that General Electric is working on superhydrophobic coatings that will allow the turbine operation in sub-zero temperatures and some papers from Karmouch et al. [20,21] and others [22] this use has not been previously reported. We believe that this work can pave the road towards such applications. Previous studies report that blades lose 10% of their annual power production from icing and other debris [23], thus the adaption of superhydrophobic coatings or fabrics covering the turbine blades will be advantageous.

The same fabrics can find additional applications (i.e., oil-water separation, antibacterial fabrics, etc.). For the modification of the fabrics we use a two-step process: First, an oxygen plasma etching step is used to create random micro-nanoscale features on the fabric surface and then a short plasma deposition step using C_4F_8 is used to alter the surface chemistry from hydrophilic to hydrophobic. Similar methods (highly anisotropic etching of polymers) have been used in the past for the modification of other polymeric substrates [24]. In addition, polymeric micro-nanotextured surfaces have been shown to exhibit antibacterial properties for a wide range of bacteria concentrations) [7]. Here we use a slightly different method in a reactive ion etching reactor for the transformation of large fabric pieces (10 cm × 20 cm) to superhydrophobic, highlighting the ability to produce large area surfaces with consistent superhydrophobic properties. A first durability evaluation of the fabrics has been performed by hand-rumpling the processed, superhydrophobic fabrics and the results suggest that durability can be enhanced by carefully choosing the fabric type and properties as well as by tuning the plasma processing conditions.

2. Experimental Section

2.1. Materials

The fabrics used in this work were provided by Felix Schaller (Graefing, Germany, <http://www.felixschaller.com>). Three fabrics with different patterns made from polyester (PES) were transformed to superhydrophobic. Some of the fabrics were initially coated and thus had different wetting properties.

Fabric A (fiber diameter 11 μm) is a dual layered 100% PES that is coated with FC (Fluor Carbon) and has high hydrophobic properties, no air permeability and is stretchable. Fabric B (fiber diameter 13.5 μm) is 100% PES, with medium filter function (air permeability) and hydrophobic properties since it is coated with TEFLON[®]. Finally, fabric C (fiber diameter 9 μm) is a non-stretchable fabric consisting of PES 99% + Carbon 1%, (carbon fiber grid) and in contrast to fabrics A and B it is not coated with a hydrophobic coating. The samples were cut in large pieces of 10 cm \times 20 cm and were processed in a Reactive Ion Etcher to demonstrate the ability to fabricate large area superhydrophobic fabrics. Figure 1 shows SEM images of the fabrics weaving characteristics. Additional information about the fabric's properties can be found in the supporting information file.

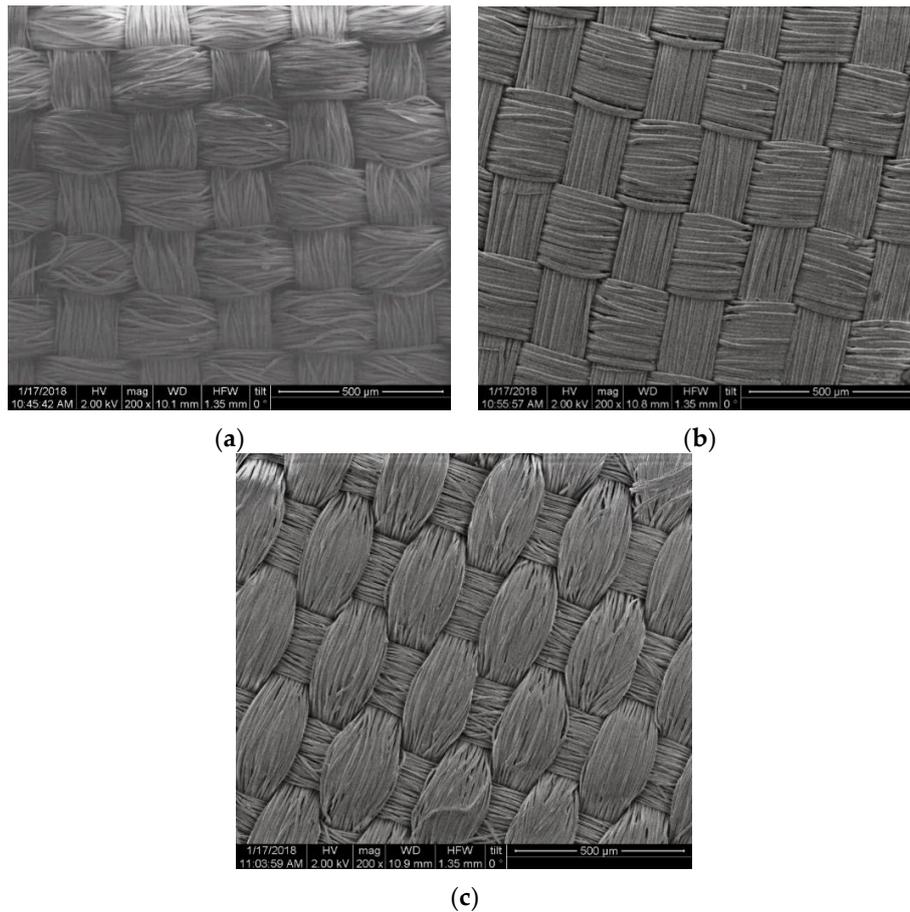


Figure 1. Top down SEM images of the fabrics surface before the plasma micro-nanotexturing: (a) Fabric A; (b) Fabric B; (c) Fabric C.

The fabrics have dense weave and different warp and weft characteristics, SEM images reveal that fabric A stretchability is the result of looser weave observed in fabric A. The fabric weave affects wetting properties, because it influences the surface fraction. Nevertheless, the micro-nanotexture is the most critical factor determining wetting properties as we will show below, since the fiber diameter is similar in all the fabrics we processed.

2.2. Plasma Treatment

The fabrics were processed in a Reactive Ion Etcher (Alcatel Nextrel NE 330), which is designed for batch wafer processing and it can take samples with dimensions up to 20 cm. It uses the reactive ion etching mode to give good anisotropy, selectivity, and uniformity in etching. It offers the advantage of providing good reproducibility and large area processing. The surface modification method is a two-step process comprising:

- A plasma etching step to create micro-nanotexture at the following conditions: O₂ flow: 100 sccm, power: 400 W, pressure: 10 mT for 12 min. At these conditions, other polymers (cyclo-olefin polymer COP, polystyrene PS, etc.) are etched with an etch rate of 100–200 nm/min. Shorter etching duration have also been tested but the resulting topography was not sufficient for superhydrophobicity.
- A plasma hydrophobization step for 2 min using C₄F₈ (10 mT, 50 W, 16 sccm). The resulting low surface energy (hydrophobic) layer thickness is 20 nm on a flat surface, while on a rough surface will be noticeably thinner but it is expected to alter the surface chemistry from hydrophilic to hydrophobic. For this reason, we consider that the proposed method is not categorized as an additive manufacturing method (bottom-up). Additionally, during the etching step, material is removed and therefore the thickness of the fibers on the treated fabrics will be lower than on the untreated ones.

The topography created after the plasma treatment is a result of co-deposition of minute amounts of quartz sputtered from the quartz reactor substrate and the highly anisotropic etching conditions applied for the fabrics treatment. Sputtered quartz areas act as etch inhibitors which for oxygen plasma enables the formation of nanotexture, which can grow to micro-nanotexture depending on the etching time [17]. This plasma etching process, which simultaneously removes material and textures the surface according to the above described mechanism, is well established and extremely reproducible with variation lower than 10%. Depending on the application targeted, one or both sides of the fabric may be modified.

2.3. Characterization Methods

The wetting properties and the morphology of the fabrics have been evaluated before and after plasma processing to demonstrate the effect of the method proposed. Moreover, the mechanical durability of the fabrics has been probed using a qualitative assessment test of rumpling the fabrics several times. The morphology characterization was done on a FEI inset SEM.

The contact angle analysis was performed with GBX Digidrop system (GBX, France, http://www.gbxonline.com/DGD_-_DX.html). Static contact angles were measured using typically 5 µL liquid droplets in three different spots of the fabric to check the homogeneity of its surface properties. Dynamic measurements were done by measuring advancing and receding angles as the droplet volume was continuously increased and decreased, respectively. Then, contact angle hysteresis (CAH) was calculated as the difference between advancing and receding contact angle. Standard deviation is typically around 2°.

The thickness of the hydrophobic film after plasma deposition using C₄F₈ gas was measured on flat Si pieces using a spectroscopic ellipsometer model M2000F by Woollam Co. (Lincoln, NE, USA).

3. Results and Discussion

3.1. Fabrics Topography after the Plasma Micro-Nanotexturing

Oxygen plasma treatment using high anisotropic conditions was used to modify the fabrics, yielding to a random micro-nanotopography on the fabric fibers. Figure 2 shows that the fabrics surface after the plasma micro-nanotexturing step exhibit random topography with micron and sub-micron scale features (nano) on the fibers. This topography is similar to the topography observed on other polymeric materials after plasma etching [24]. We observe that in all fabrics regardless of their initial weaving, fabric material and coating characteristics micro-nanoscale topography was created. The extent of the topography is of course influenced by the weaving, fabric material, and coating characteristics, but the strength of the plasma micro-nanotexturing technology is that it allows for such topography to be created on the surface of any polymeric material. This micro-nano texture in combination with an ultrathin coating can render all fabric materials superhydrophobic, as will be seen below. As we will demonstrate in the next section, all fabrics were successfully transformed to

superhydrophobic (water contact angle $> 150^\circ$, hysteresis $< 10^\circ$). Thus, the feature dimensions and distribution in combination with the low surface energy are appropriate for superhydrophobicity. These micro-nanoscale features in combination with the fabric weave determine surface fraction, which is the ratio of the wet surface area divided by the projected one on the solid plane. We have shown that for water, the upper limit in surface fraction in order to achieve superhydrophobicity is 0.25 [25] and we assume that this is fulfilled in all fabrics tested. In addition, we have shown that surfaces with features like the ones on fabric A are extremely stable superhydrophobic surfaces [26]. A more careful look in Figure 2 reveals that the topography is thicker and higher in fabric A, and smaller in fabrics B and C. This results in a slightly higher water contact angle on fabric A. The topography differences can be attributed to: (a) the TEFLON[®] coating material existing in fabrics A and B which can result in different etching rates between different areas and the (b) fabric weaving characteristics which influence the surface fraction.

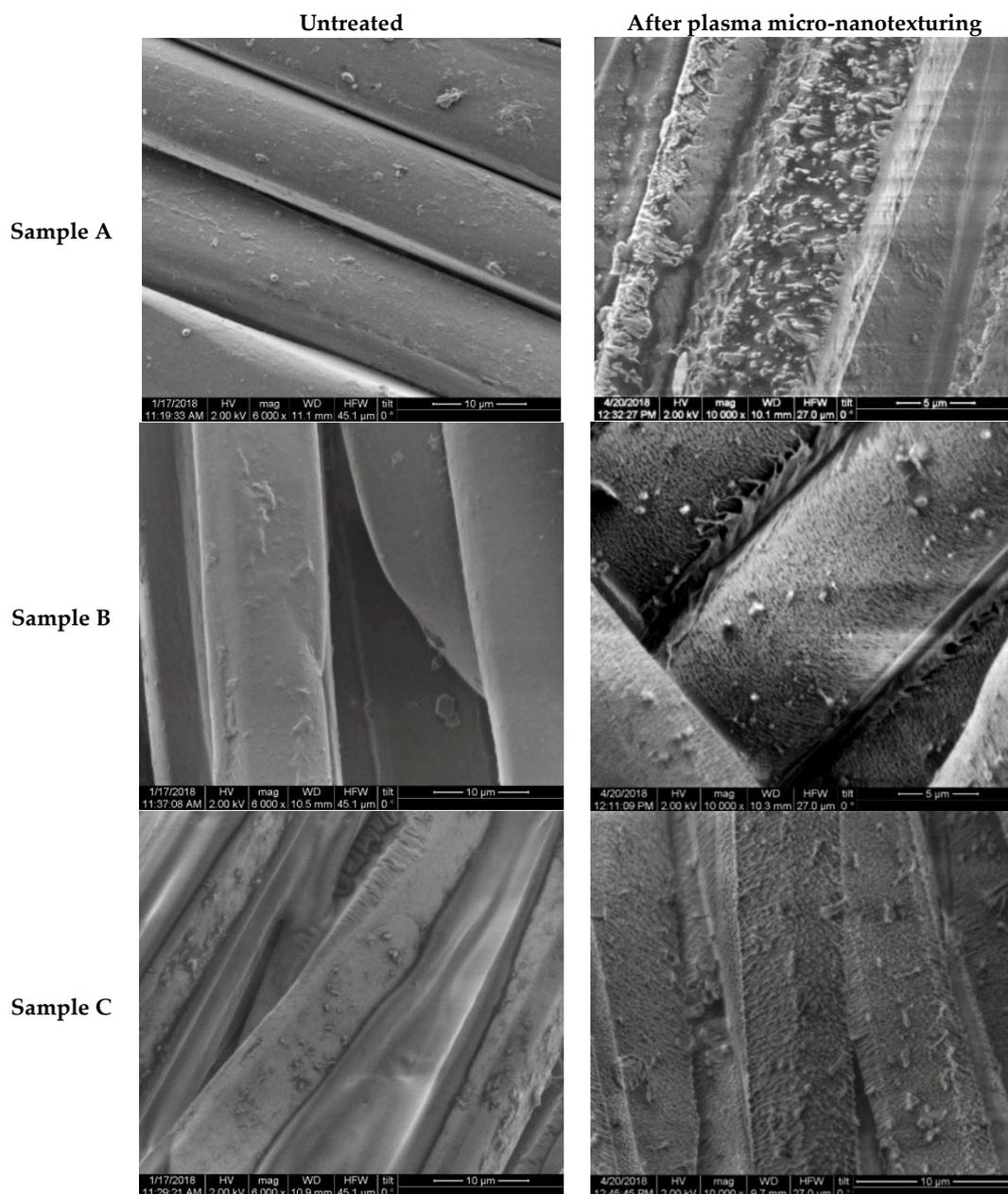


Figure 2. Top-down SEM images of the fabrics before and after the plasma micro-nanotexturing.

However, the topography created is not appropriate for superoleophobicity (as we will see below in Table 1), due to the fabric weaving characteristics and the small size of the topography created after the plasma texturing step. These two factors, as explained above, influence the surface fraction value, keeping it higher than 0.1 which is the upper limit for achieving superoleophobicity (soya oil static contact angle $> 150^\circ$, contact angle hysteresis $\ll 10^\circ$) according to the Cassie equation [25]. Higher etching duration might provide superoleophobic properties by increasing the height and of the topography and providing more undercut structures, but we did not increase the plasma duration in order not to compromise the topography durability, as we have shown in our previous work [24].

3.2. Wetting Properties

The wetting properties of the three fabrics were evaluated using dynamic (hysteresis) and static contact angle (SCA) measurements. All measurements are listed below in Table 1. Contact angle (CA) measurements in three different spots on each sample were done and average water contact angle and standard deviation were calculated.

Table 1. Wetting properties of PES fabrics as received (1), after coating with a plasma deposited hydrophobic layer (2) and after plasma processing (3). CAH is given inside brackets. The adhesion behavior of each fabric (sticky, slippery) is given inside parentheses.

Sample	Water Static Contact Angle (WSCA) Samples as Received (1)	Water Static Contact Angle (WSCA) after ONLY Plasma Deposition of Hydrophobic Film (2)	Water Static Contact Angle (WSCA) after Plasma Nanotexturing AND Plasma Deposition of Hydrophobic Film (3)
Fabric A	108° (sticky)	124° (sticky)	157° ± 3° [4°] (slippery)
Fabric B	120° (sticky)	128° (sticky)	153° ± 3° [7°] (slippery)
Fabric C	superhydrophilic	–	154° ± 2° [5°] (slippery)

Note: Standard deviation in SCA hysteresis measurement $\pm 2^\circ$.

All fabrics after the plasma micro-nanotexturing step are superhydrophilic (water contact angle $< 10^\circ$) and after the deposition of the hydrophobic layer become superhydrophobic (column 3). It is evident that the water ($\gamma_{lv} = 72.8 \text{ mN}\cdot\text{m}^{-1}$) contact angle increases by at least 25° in all samples (fabrics A and B are hydrophobic as received). In fabric C we have an initially superhydrophilic fabric that is transformed to superhydrophobic. Fabrics B and C after the process are also oleophobic and the soya oil ($\gamma_{lv} = 34.1 \text{ mN}\cdot\text{m}^{-1}$) SCA exceeds 90° . Interestingly, fabric A is simultaneously superoleophilic and superhydrophobic, indicating that this fabric can be potentially used for oil-water separation applications. As a proof of concept for this application, Figure 3a shows two drops, a water and a soya oil drop on fabric A (drop volume $20 \mu\text{L}$). Soya is completely absorbed, while water is repelled exhibiting CA higher than 150° . In Figure 3b,c water drops on fabrics B and C are shown (WSCA exceeds 150° and CAH is below 10° , water drop volumes are $20 \mu\text{L}$ for visualization reasons). The water and soya drops (volume $5 \mu\text{L}$) that were used to measure the SCA and hysteresis in each fabric, as recorded by the camera of the GBX Digidrop system, are included as insets in Figure 3.

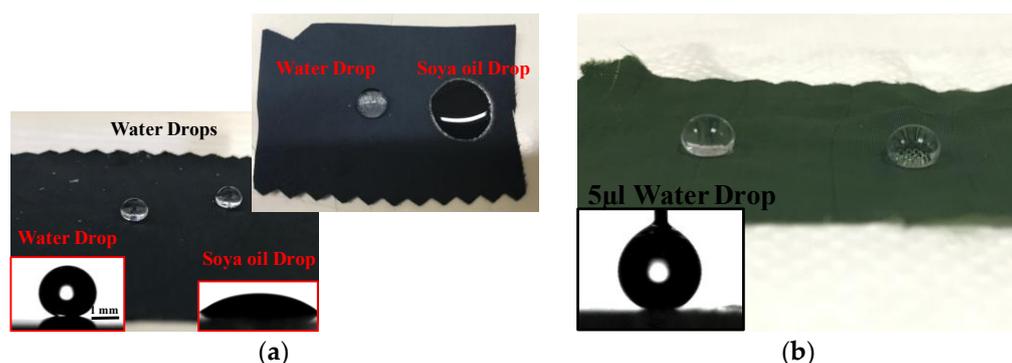


Figure 3. Cont.

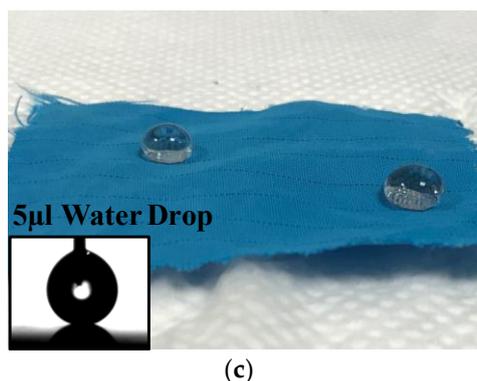


Figure 3. Images of the fabrics after plasma processing: (a) Water and soya oil drops on the plasma micro-nanotextured PES fabric A; (b) Water drops on the plasma micro-nanotextured PES fabric B; (c) Water drops on the plasma micro-nanotextured PES fabric C. Water and soya drops (5 μL) as shown as insets, images captured from GBX Digidrop system.

3.3. Mechanical Evaluation

Durability is the main obstacle for the successful application of any superhydrophobic surface [8]. Here, in particularly as wind turbine is exposed to various types of whether conditions of long periods of time, long-lasting superhydrophobicity is required. We evaluated the durability of the fabrics using a qualitative evaluation test (hand-rumpling). The fabrics showed remarkable stability of their wetting properties even after hand-rumpling them eight times. The evaluation test against mechanical rumpling is shown in Figure 4. It shows that WSCA remains above 150° and CAH below 10° after rumpling for five (5) times. After the 5th rumpling WSCA starts to decrease and consequently CAH increases. More specifically after the 5th rumpling, CAH in fabrics B and C exceeds 10° which is considered the upper limit for superhydrophobicity. In fabric B it continues to increase and reaches 15° after rumpling eight times, while in fabric C it remains stable around 11° – 12° . A possible explanation for the good mechanical durability that all fabrics exhibited is the enhanced adhesion of the hydrophobic coating. Previous studies with the same coating imply that the coating adhesion is enhanced by the oxygen plasma micro-nanotexturing step done prior the deposition [27]. We believe that the same effect is observed here. Additionally, to this enhanced durability against rumpling, all fabrics retained their superhydrophobic properties after one year of storage in the laboratory environment. However, the best performance is observed on fabric A where hysteresis does not exceed 10° even after rumpling eight times. The video showing the rumpling tests of the fabrics can be found in supporting information.

The enhanced durability of fabric A is due to the combination of two additional factors: (a) fabric A properties (fabric A is the only stretchable one) and (b) the thicker microscale features created after the plasma micro-nanotexturing step (compared to other two fabrics, see Figure 2), which makes the topography more robust against mechanical stresses. This finding is confirmed by the SEM image after rumpling eight times (insert in Figure 4), which shows that no significant changes of the topography of fabric A are observed after rumpling.

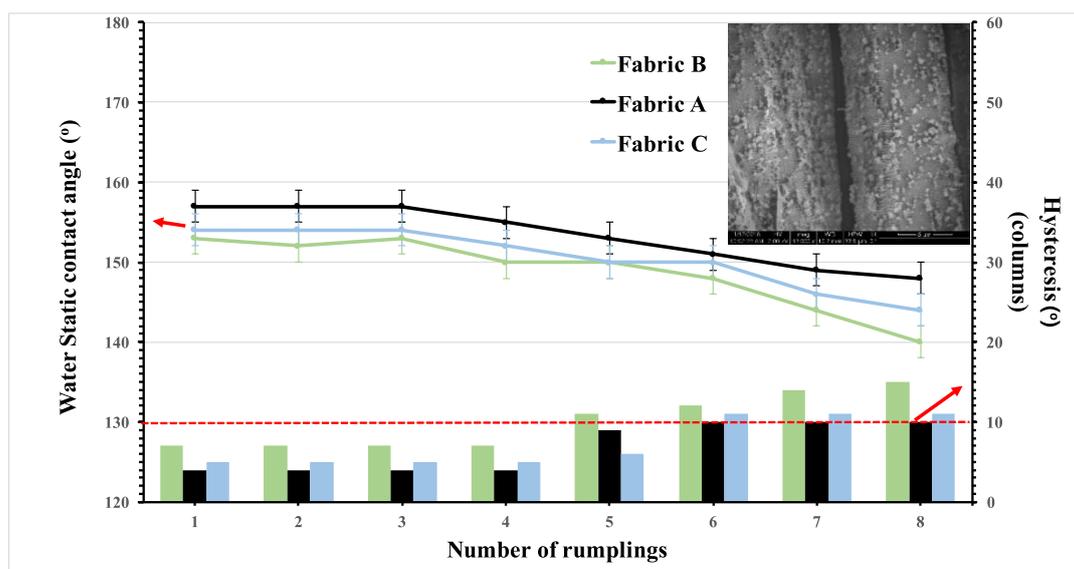


Figure 4. Stability against rumpling for the three different fabrics. Fabric A exhibits the highest durability against rumpling. On the right axis hysteresis is shown. The red dashed line is the Hysteresis upper limit for superhydrophobicity. An SEM image of fabric A after rumpling the fabric eight times is given as insert. If one compares this image with the SEM image of the fabric A in Figure 2, it is clear that the topography created after plasma micro-nanotexturing is not destroyed after rumpling.

4. Conclusions

We have developed a simple and generic method to produce superhydrophobic fabrics based on direct plasma-induced surface modification. All materials tested exhibit good mechanical stability after rumpling the samples at least five times. Our approach involves two steps: (1) plasma micro-nanotexturing and (2) plasma deposition to render the fabrics superhydrophobic. Here, PES fabrics were tested as a proof of concept, but the method can be easily adapted to other polymeric fabrics. The fabrics herein are intended for use as self-cleaning fabrics covering wind turbine blades, which is expected to improve the wind turbines performance and reduce their maintenance cost. Similar fabrics can also be used in other applications (i.e., antibacterial fabrics, oil-water separation). We intend to test them for such applications in the future.

Supplementary Materials: The following are available online at <http://www.mdpi.com/2079-6412/8/10/351/s1>. Table S1: Fabrics properties; Video S1: Fabrics new.

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Conflicts of Interest: Authors hold equity in Nanoplasmas Private Company, which is commercializing plasma micro-nanotexturing technology. Authors declare no conflict of interest or other financial conflict. The funding agency (Stavros Niarchos Foundation) had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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