Ultrashort-Pulsed Laser Modification for Highly Hydrophobic Woven Fabric

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Among superhydrophobic materials, non-wettable textiles arguably come in contact or interact with the human body most frequently. The author presents the proof-of-concept of a novel modification method for a woven fabric surface for realizing excellent hydrophobicity using two kinds of ultrashort-pulsed lasers. The key hypothesis is that to improve the hydrophobicity of the woven fabric, it might be necessary to not only improve the water repellency of the surface but also to prevent liquid seepage between filaments of the woven fabric. A woven fabric of Nylon 6 is addressed by (a) enhancing liquid hydrophobicity by applying micro-periodic structures on the filaments through a femtosecond-pulsed laser processing based on the Cassie-Baxter model, and (b) preventing liquid seepage between the network of filaments by utilizing picosecond-pulsed laser processing via thermal sealing. The optimal conditions for the laser processing were determined experimentally. This laser modification method exhibited an apparent contact angle of 120°, comparable to chemical treatments as a non-wettable application. A processed depth that was onefiftieth of the filament diameter was sufficient for improving the hydrophobicity of the woven fabric. The combination of laser processing effectively enhanced the water repelling and sealing of the woven fabric, indicating that excellent hydrophobicity was achieved. No significant difference was observed in color due to the laser processing, and the decrease in tearing strength was approximately 9%. These results highlight the possibility of strategies that can introduce a novel process for non-wettable woven fabrics as a sustainable technology.

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1. Introduction

Superhydrophobic and anti-wetting characteristics have been among the most intensively studied properties of textiles within the last few decades [1,2]. This attention is mainly due to their potential applications in the design and preparation of self-cleaning, anti-adhesion, and dirt-free textiles [3]. Accordingly, numerous novel approaches to decreasing the surface free energy of filaments have been studied in the last few years, employing wet-chemical finishes based on modern chemical developments such as silane chemistry. Various chemicals have been used, silanes and silicones specifically play very important roles in the preparation of superhydrophobic and superoleophobic materials [4,5]. However, perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS) have attracted global attention due to their chemical durability, wide distribution, biotoxicity, and bioaccumulative properties [6,7]. Currently, regarding sustainability, the textile industry is one of the most threatening production systems to the environment, with negative impacts ranging from crops to finished products [8].

Biomimetic micro- and nano-periodic structures have garnered attention due to their excellent water repellency [9]. In particular, the expression of hydrophobicity and hydrophilicity through periodic structures at micro/nanometer scales is gaining attention as a carbonneutral method that does not require chemical treatment [10]. The static fundamental mechanisms that provide hydrophobicity and hydrophilicity are widely established through the Cassie-Baxter (C-B) [11] and Wenzel [12] models, respectively, which are based on two-dimensional micro-periodic structures [13]. Nanomaterials of various dimensions have been developed, including spherical nanoparticles, graphene nanosheets, and nano-fibers [14]. As a physical approach, attempts have been made to use laser processing on filament surfaces to modify their microstructure using an excimer laser [15,16]. However, there is a possibility that filament breakage may occur because the excimer laser energy cannot be precisely controlled. Laser processing based on ultrashort-pulsed laser (including picosecond-pulsed and femtosecond-pulsed lasers) is a useful technology that can be used to process fine structures at micro/nanometer scales [17,18]. Especially, femtosecondpulsed laser ablation offers advantages over long pulse lasers because there is little or no collateral damage due to heat conduction produced in the processed material [19,20].

As an approach of laser to woven fabrics, the faded or worn effects of denim garments have been achieved as a rapid method that provides precision in processing and will continue to be popular with this market sector. Laser treatment is considered a clean process that offers low cost and low environmental impact because it is a water-free method [21-23].

In this research, physical approaches are proposed to overcome the limitations of chemical approaches. The purpose of this research is to demonstrate the feasibility of an ultrashort-pulsed laser modification method to create a woven fabric surface with excellent hydrophobicity. The key hypothesis is that to improve the hydrophobicity of the woven fabric, it might be necessary to not only improve the water repellency of the surface but also to prevent liquid seepage between filaments of the woven fabric. Laser hydrophobicity processing is performed using а femtosecond-pulsed laser, and laser sealing processing is performed using a picosecond-pulsed laser. Both processing conditions are optimized for fabricating a woven fabric prototype, with both processes carried out on the same surface. Finally, the author also investigates the strength of the woven fabric by conducting a tearing test.

2. Materials and Methods

2.1 Proof-of-concept of Highly Hydrophobic Woven Fabric

Our proof-of-concept of modification method for highly hydrophobic woven fabric use two kinds of ultrashortpulsed lasers for the physical surface processing (Fig. 1). The key hypothesis is that to improve the hydrophobicity of the woven fabric, it might be necessary to not only improve the water repellency of the surface but also to prevent liquid seepage between filaments of the woven fabric. I address this by (a) enhancing liquid hydrophobicity by applying micro-periodic structures on the filaments through femtosecond-pulsed laser (FL) processing based on the C-B model, and (b) preventing liquid seepage between the network of filaments by utilizing picosecond-pulsed laser (PL) processing via thermal sealing. The hydrophobicity is aimed to increase by creating a hierarchical structure in which a short-periodic structure is formed on a long-periodic structure. This combined approach ensures significantly enhanced hydrophobicity of the 3D structure comprising the woven fabric.

2.2 Woven Fabric

A woven fabric made of Nylon 6 (Goldwin Inc., Oyabecity, Japan) was used as shown in Table 1. No chemical surface coatings were applied on the woven fabric.

To increase flatness, the woven fabric was pressed at the manufacturer (calender, Goldwin Inc.) in a machine which

Table 1	I Specifications	of woven	fabric o	f Nylon 6.
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Weaving		Plain weave	
Color		Black	
Linear mass density of fibers*1	Warp	20 Denier	
	Weft	20 Denier	
Diameter of filament, df	Warp	12 - 13 μm	
	Weft	12 - 13 μm	
Number of filaments*2	Warp	20 filaments	
	Weft	20 filaments	
Fabric weight		35 g/m ²	
Pitch of filaments, $\tau_f *^3$	Warp	$11.3\pm0.2~\mu m$	
	Weft	12.3 ± 0.5 µm	

*1 Denier = 1 g/9,000 m

*² A single fiber is formed by twisting multiple filaments.

*3 Distance between two filaments. Physical properties above the dotted line were provided by the manufacturer.

heavy rollers rotate in contact under a mechanical pressure [24,25]. The thickness of the pressed woven fabric measured using a micrometer (MDE-25MX, $\pm 1 \ \mu m$ of maximum allowable error, Mitutoyo Co., Kawasaki, Japan) was 46.2 \pm 1.5 μm (sample number n = 10, mean \pm standard deviation; SD).

2.3 Laser Hydrophobicity Processing

Fig. 2 shows the ultrashort-pulsed laser systems used for direct processing on the surface of the woven fabric. The two laser systems, FL and PL, for laser hydrophobicity processing and laser sealing processing, respectively, have almost the same configuration except for a beam expander in the former. The FL system for laser hydrophobicity processing comprised of (i) a femtosecond-pulsed laser (Pharos-6W, Light Conversion UAB, Vilnius, Republic of Lithuania), (ii) a beam expander (\times 1.2, S6EXZ5311/292, Sill Optics GmbH, Wendelstein, Germany), (iii) an x-ygalvano mirror (CUA32-MST-AC, Newson NV. Dendermonde, Belgium), (iv) a telecentric $f\theta$ lens (f = 56mm, TSL-532-15-67Q, Wavelength Opto-Electronic (S) Pte Ltd, Singapore), (v) an x-y electric-stage (HST-50XY, 2 µm of repeated positioning accuracy, Sigmakoki Co., Ltd., Tokyo, Japan), (vi) a z-stage (OSMS60-10ZF, 5 µm of repeated positioning accuracy, Sigmakoki Co., Ltd.), and



Fig. 1 Proof-of concept of ultrashort-pulsed laser modification for improving hydrophobicity as a physical approach.



Fig. 2 Configuration of ultrashort-pulsed laser systems used for direct processing on the surface of woven fabric of Nylon 6 (femtosecond-pulsed processing: with a beam expander, picosecond-pulsed processing: without a beam expander, PC: personal computer).

(vii) a personal computer (TG02-0050, Victus, Hewlett-Packard Inc., Palo Alto, CA). Test-pieces of the woven fabric were fixed by an embroidery hoop with dimensions of 105 mm \times 80 mm.

The conditions of the FL processing in the fabrication of the test-pieces were as follows: an oscillatory wavelength, λ , of 515 nm, a pulse width of 277 fs, a repetition rate of 50 kHz, and processing cycle of 0.02 ms. A transistor-transistor-logic signal (TTL signal, 2 kHz of frequency, 5 V of voltage) output from the controller of the galvano mirror was used as the control signal for feedback control of all devices.

FL processing to create micro-periodic structures was performed on one side of a woven fabric in a square area with a length of 3 mm in both the x-y directions (3-mm²) area). Fig. 3A shows the FL processing pattern used for laser hydrophobicity processing. The fluence, F, the number of shots, s, and the pitch, τ , were varied as the FL processing parameter (Table 2). The optimal FL processing conditions were identified based on the time-course changes of the apparent contact angle of 2 μ L of distilled water, θ ', using a commercial contact angle analyzer (DM-701, Kyowa Interface Science Co., Ltd., Saitama, Japan). The measurements were repeated five times and the mean \pm SD was showed (n = 5). The geometries of the micro-periodic structures were measured using a non-contact laser confocal microscope (10 nm resolution for depth, OLS4100, Olympus Co., Tokyo, Japan). The geometric parameters of the micro-periodic structure included pitch, τ , tooth width, f_1 , groove width, f_2 , and depth, h, (where pitch, $\tau = f_1 + f_2$), which can be controlled by the spot diameter, the spot pitch, and the number of shots.

Adjusting the focal position is important for processing the micro-periodic structure on flexible materials such as woven fabric using FL processing. In this experiment, testpieces of the woven fabric were placed at the focal position in the depth direction, *z*, and were set appropriately using the *z*-stage. The theoretical spot diameter at the focal position, d_{f_s} of a Gaussian beam on the test-piece surface was calculated to be 13.1 µm by $1/e^2$ using the focal length of the f θ lens f = 67 mm, a laser beam quality of $M^2 = 1.117$, and an expanded (× 1.2) laser beam diameter $d_b = 4.2$ mm as follows [26]:

$$d_f = \frac{4 f \lambda M^2}{\pi \, d_b} \tag{1}$$

Consequently, the focal position, i.e. rayleigh length, z_R , is calculated to be $\pm 261 \ \mu m$ as follows [27]:

$$z_R = \pm \frac{\pi w_0^2}{\lambda} \tag{2}$$

where, ω_0 is theoretical spot radius at the focal position, 2 $\omega_0 = d_f$.

As the fabric thickness is 46.2 μ m, the focal position was set to \pm 261 μ m (= 522 μ m in total) by using the *z*-stage which is more than ten times the woven fabric thickness.

2.4 Laser Sealing Processing

The PL system for laser sealing processing comprised of (i) a picosecond-pulsed laser (TruMicro5250, Trumpf SE + Co. KG (Holding), Ditzingen, Germany), (ii) an *x*-*y* galvano mirror equipped with a telecentric f θ lens (f = 300 mm, AXIALSCAN-20, Raylase GmbH, Wessling, Germany), (iii) an *x*-*y* electric-stage (XRL130-110, \pm 0.3 µm of bidirectional repeatability, Akribis Systems Pte Ltd, Singapore), (iv) a *z*-stage (ZA10A-W2C02, \pm 0.5 µm of repeatability, Kohzu Precision Co.,Ltd., Kawasaki, Japan), and (v) a personal computer (TG02-0050, Victus, Hewlett-Packard Inc., Palo Alto, CA).

PL processing was performed over a square area with a length of 4 mm in both the *x*-*y* directions (4-mm² area) on one side of a woven fabric (Table 2). Fig. 3B shows the PL processing pattern. Both the spot pitch, *p*, and the beam diameter, d_p , were standardized at 43 µm to avoid overlapping the laser spots. The PL processing was performed by repeating rectangular orbits (track orbits), with the process time required for processing each orbit defined as the processing pattern period. The time need for PL processing area, the processing pattern period, and the number of shots. Therefore, a shorter process pattern period can realize high-speed processing.

The fluence, F, processing pattern period, η , and number of shots, s, were varied as the PL processing parameter. To identify the geometries of the surfaces, both the non-contact laser confocal microscope and a scanning electron microscope (SEM; 8 nm resolution for depth, VE-9800, Keyence Co., Osaka, Japan) were used. Prior to observation, samples for SEM were fixed with a photocurable acrylic resin (LCR D-800, Toagosei Co., Ltd., Tokyo, Japan) and then cut with an ultramicrotome (Leica EM UC6, Leica Microsystems, Co., Tokyo, Japan).

2.5 Tearing Test of Woven Fabric

A test-piece of woven fabric with both FL and PL processing was fabricated under the optimal conditions for both processes. Due to processing time constraints, FL

A. Femtosecond-pulsed laser (FL) processing



B. Picosecond-pulsed laser (PL) processing



Fig. 3 Laser processing method on one side of woven fabric.

Fluence	Number of shots	Spot diameter	Pitch	Spot pitch	Processing pattern	Processing cycle
F (mI/cm ²)	s (shots)	a (um)	τ (um)	p (um)	period η	t (ms)
	EL massagin	(μ)	(µ111)	(µIII)	(IIIS)	(IIIS)
FL processing						
15.1	2	13.1	13	4	—	0.02
22.6	1					
	2		12			
			13			
			14			
	3		13			
30.1	2		13			
PL processing						
32.0	900	43	43	—	2	0.01
37.0	600					
	900				1	
					2	
					3	
					4	
	1200					
42.0	900				2	

Table 2 FL processing conditions of test-pieces for Fl and PL processing.

processing was performed on square area with a length, l = 5 - 20 mm (5 - 20 --mm square), and PL processing was performed on a 25-mm square (Fig. 4). Therefore, the relationship between the tearing strength, F, and the processed area, S, was measured.

A tearing test was conducted on the test-piece based on the Japanese Industrial Standard of testing methods for woven and knitted fabrics [28]. A test-piece (50 mm of short side and 250 mm of long side) was incised in the center for 100 mm length. Both ends of the cut were held by the clamps of a t universal material testing machine (TENSILON RTC-1250A, A&D Co., Ltd., Tokyo, Japan) and pulled in opposite directions at a speed of 100 mm/min. The maximum tearing strength was measured five times, and the average value was calculated.

Color differences in terms of lighter shades, L^* , axis a^* (green – magenta opponent colors), and axis b^* (blue – yellow opponent colors, CIE 76) of the woven fabric both with and without processing were measured using a color difference meter (CM-26d, Konica Minolta Japan, Inc., Tokyo, Japan).

2.6 Statistical Analysis

Statistical analyses were performed with the Statistical Package for the Social Sciences (SPSS) version 20.0 (SPSS Inc, Chicago, IL). Unless otherwise stated, all data are expressed as mean \pm standard deviation (SD, n = 5).



Fig. 4 Test-piece with both FL and PL processing for tearing test.

3. Results and Discussion

3.1 Laser Hydrophobicity Processing

Table 3 shows the measured results of the surface geometries and the apparent contact angles when fluence was used as the FL processing parameter. Where, the number of shots, s, and the pitch, τ , were set to be constant at 2 and 13 µm, respectively. While concerns arose regarding the potential impact of woven fabric undulations on the focal point, exemplary processing reproducibility was nonetheless attained, even when employing flexible woven fabric. This was achieved by fixing the woven fabrics with an embroidery hoop. Both the f_2/f_1 ratio and the depth, h, increased in proportion to the fluence. However, the apparent contact angle did not change with these parameters and reached its maximum value when the fluence was 22.6 mJ/cm². Additionally, in the time-course changes, the apparent contact angle also remained in maximum until 1 min when the fluence was 22.6 mJ/cm² (Fig. 5A). In addition, the micro-periodic structure is an important factor to achieve water repellency and pressure resistance, however, gradual water seepage was inevitable.

The structure of a woven fabric, in which filaments with a circular cross section are lined up, can be a typical C– B model [29,30]. In this research, the diameter and pitch of the filaments of the tested woven fabric were $d_f = 12 - 13$ µm and $\tau_f = 11.3 - 12.3$ µm, respectively (Table 1). As both values were almost within the same range, they were not the optimal geometries of the micro-periodic structure [31,32]. Therefore, a hierarchical structure was formed in which the short-periodic structures were modified on the aligned filaments by FL processing. The hierarchical structure had the geometry of a superhydrophobic structure [33,34]. However, in the C–B model, the apparent contact angles should increase in proportion to the f_2/f_1 ratio [35,36], and the pressure resistance should increase in proportion to the depth [37,38]. These facts suggest that the present results for the short-periodic structure cannot be explained only by the C–B model. It has been reported that a micro-structure created on filaments by laser processing improved their hydrophilicity [39]. The short-periodic structure was considered to have the function of trapping water droplets. Typically, both long-periodic and short-periodic structures in the hierarchical structure satisfy the C-B model [40]. The hierarchical structure in this study potentially consisted of a hydrophilic short-periodic structure on a hydrophobic long-periodic structure.

Fig. 5B shows the measured results of the apparent contact angles when the pitch was used as the FL processing parameter. Where, the fluence and the number of shots were set to be constant at 22.6 mJ/cm² and 2, respectively. the apparent contact angle reached its maximum value when the pitch was 13 μ m. FL ablation offers advantages over long-pulse lasers due to its minimal collateral heat damage within the processed material [41]. However, Stoian *et al.* [42] reported the existence of a violent explosive ablation. This latter phase is attributed to thermal decomposition, potentially influenced by the material's thermal conductivity. Therefore, irradiation in closed proximity might lead to thermal effects in materials with low thermal conductivity such as organic materials as woven fabric.

Fig. 5C shows the measured results of the apparent contact angles when the number of shots was used as the FL processing parameter. Where, the fluence and the pitch were set to be constant at 22.6 mJ/cm² and 13 μ m, respectively. Depths was determined to directly proportional to the number of shots. No regular changes were observed in f_2/f_1 ratio under different number of shots. The optimal value for the number of shots was 2. It was estimated that the depth was optimal for hydrophilicity at this condition.

Based on these results, under a f_2/f_1 ratio of 2.6 and a depth of 0.25 µm, the optimal conditions for the laser hydrophobicity processing were identified to be 22.6 mJ/cm² of fluence, 13 µm of pitch, and 2 of number of shots. Therefore, the optimal pitch was almost the same value as the diameter of the filaments. A depth of one-fiftieth of the diameter was sufficient for this physical modification.

3.2 Laser Sealing Processing

Table 4 shows the measured results of the surface geometries and the apparent contact angles when the fluence was used as the PL processing parameter. Where, the processing pattern period and the number of shots were set to be constant at 2 ms and 900, respectively. Fusion-like traces of the filaments were observed in SEM images of the sample, especially in the area enclosed by the dotted circle when the fluence exceeded 37 mJ/cm². The apparent contact angle remained at their maximum until 5 min when the fluence was 37.0 mJ/cm² and was increased by 60% compared to the unprocessed woven fabric (Fig. 6A). Therefore, the sealing effect of PL processing was observed in wettability. Although the suitability of PL processing for precision processing is similar to that of FL processing, the former has a greater thermal effect than the latter. Heat conduction in laser-irradiated solids is normally determined by the bulk thermal conductivity. Pronko et al. [43] reported



Table 3 Measured results of the surface geometries and the apparent contact angles when the fluence was used as the FL processing parameter.

Fig. 5 Measured results of the time-course changes of apparent contact angles by using FL processing.

that heat pulse in a metal will spread over approximately 20 Å in a time interval of 100 fs. In this study, I attempted to precisely control thermal processing using PL to avoid damaging the filaments, and the optimal value of fluence was identified experimentally.

Apparent contact angle, $\theta'(^{\circ})$

Fig. 6B shows the measured results of the apparent contact angles when the processing pattern period was used as the PL processing parameter. Where, the fluence and the

number of shots were set to be constant at 37.0 mJ/cm² and 900, respectively. When the processing pattern period was 1 ms, the filament completely melted and contracted, deforming the woven fabric. The apparent contact angle at 5 min decreased when the processing pattern period shortened to 2 ms. The cause of this phenomenon was thought to be heat damage resulting from the processing pattern period being too short. Fig. 6C shows the measured apparent



Table 4 Measured results of the surface geometries and the apparent contact angles when the fluence was used as the PL processing parameter.

* Fusion-like traces were observed in the area surrounded by dotted circle.



Fig. 6 Measured results of the time-course changes of apparent contact angles by using PL processing.

contact angles when the number of shots was used as the PL processing parameter. Where, the fluence and the processing pattern period were set to be constant at 37.0 mJ/cm² and 2 ms, respectively. The balance point between the thermal sealing and the heat damage was 900 of number of shots.

Based on these results, the optimal conditions for the laser hydrophobicity processing were identified to be 37.0 mJ/cm^2 of fluence, 2 ms of processing pattern period, and 900 of number of shots.

3.3 Tearing Test for Textiles

Both the FL processing and the PL processing were applied to one side of the same woven fabric under the experimentally determined processing conditions. The times needed to process a 20 mm area of FL processing and a 25 mm area of PL processing were 2 h 53 min and 56 min, respectively. Processing time mainly depends on the laser power and the speed of the scanner.

Table 5 shows the measured results of the apparent contact angles and the surface geometry of a woven fabric with processing. Finally, the apparent contact angle immediately after dropping was 120°, which is equivalent to that obtained by chemical treatments such as silane chemistry. The apparent contact angle after 5 min also improved from 47° on the untreated woven fabric to 82° on the treated woven fabric.

Table 5 Measured results of the apparent contact angles and the surface geometry of a woven fabric with both FL processing and PL processing.



Fig. 7 shows the measured results of tearing test of the woven fabric with processing. The tearing strength of the unprocessed woven fabric was 16.0 N. The tearing strength of the woven fabric with processing was 14.9 N for a testpiece with an area of 5-mm² area, and remained constant at 14.5 N after the area of the test piece exceeded 10-mm² area. Therefore, it was concluded that the tearing strength of the woven fabric with processing decreased by 9% due to the simultaneous application of both the FL processing and the PL processing. Although a decrease in strength due to laser processing is unavoidable, this problem could possibly be overcome by increasing the filaments diameter and/or the number of filaments for the woven fabric.



Fig. 7 Measured results of tearing test of test-piece with both FL and PL processing.

The L^* , a^* , and b^* of the woven fabric without processing were 21.76 ± 0.15 , 0.85 ± 0.01 , and -0.34 ± 0.01 , respectively. The L^* , a^* , and b^* of the woven fabric with processing were 21.26 ± 0.11 , 0.69 ± 0.04 , and -0.48 ± 0.04 , respectively. In this study, a black colored-woven fabric was selected, whose color possibly affected the laser processing. However, no significant difference was observed between the color differences.

4. Conclusions

This study highlights a remarkable laser modification method to improve the hydrophobicity of woven fabric physically. A combination of femtosecond-pulsed laser processing and picosecond-pulsed laser processing effectively enhanced the water repelling and sealing of the woven fabric, indicating that the excellent hydrophobicity was achieved. This laser modification method showed a 120° of apparent contact angle, comparable to chemical treatments as a non-wettable application. No significant difference was observed in color due to the laser processing, and the decrease in tearing strength was approximately 9%.

Therefore, it was demonstrated that the ability to exhibit hydrophobicity equivalent to chemical treatment could be achieved. There have been reported that the global textile industry is responsible for having a serious environmental impact with remarkable greenhouse gas emissions over 3.3 billion metric tons per year [44,45]. If hydrophobicity can be improved through physical approaches such as ultrashortpulsed laser processing, greenhouse gas savings can be achieved in the production, application, and drying processes of chemical substances.

However, the limitations inherent to this study require further exploration. It is necessary to demonstrate durability and processing speed at a level that can withstand industrial applications, presenting a significant engineering challenge.

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