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Antibacterial silver coating on poly(ethylene terephthalate) fabric by using high power impulse magnetron sputtering

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ABSTRACT

Antibacterial silver coatings on textiles, formed by various coating processes, have attracted substantial attention. However, the durability of these coatings in practice is poor, limiting their usage. The goal of this study is to prepare antibacterial silver films on poly(ethylene terephthalate) (PET) fabrics by high-power impulse magnetron sputtering (HIPIMS), which is known to provide a high plasma density, so as to form a strongly adhered film at a relatively low substrate temperature. These silver-coated textiles are expected to exhibit antibacterial efficacy and durability.

The experimental results herein reveal that the silver coating can be successfully deposited on PET fabric by HIPIMS with a crystal structure presenting (111) preferred orientation and that the fibers are uniformly covered. Pre-treatment with oxygen plasma for a single minute can effectively enhance film adhesion in dry and wet rubbing tests, such that the color fastness can be ranked Grade 5 and Grade 4, respectively. The coated fabric retains the mechanical properties of its original bare fabric and the coating procedure does not induce damage to PET fabric. Antimicrobial performance testing indicates that a silver film that is deposited for more than 1 min provides strong bacteriostatic (>2.0) and bactericidal (>0) effects, based on the JIS Standard. Additionally, the coated fabrics retain their antimicrobial capability after 20 cycles of washing, demonstrating their long-term durability.

1. Introduction

wear, which has formed a considerable market for antimicrobial textile products [1]. Therefore, many treatments and antimicrobial agents for fabrics and textiles have been used. They date to the early sixties, when a method of using antimicrobial neomycin to adhere on cellulosic fiber with a water-soluble copper-containing fixing agent was demonstrated [2]. Antimicrobial fabrics can basically be categorized in two classes – fabric to which is bound an antimicrobial substance and fabrics [3,4] that are finished with various antimicrobial reagents via different routes. With respect to the latter, antimicrobial finishing is typically based on wet processes, including the electroless plating of antimicrobial metal [5,6], the sonochemical irradiation of nano silver particles [7,8], and impregnation with organic/inorganic substances [9,10], among others.

As is well known, the antimicrobial treatment of textiles must satisfy many requirements [1]. These include effectiveness against a broad range of bacterial and fungal species, harmlessness to consumers, durability when laundered, the absence of any negative effect to the

substances that may harm the manufacturer or the environment. The aforementioned wet treatments may satisfy some of these requirements but, unfortunately, the resultant products are fairly poorly when laundered, and the harmful substances that must be used may have a severe environmental impact [11].

To meet all of the requirements for the antimicrobial treatment of textiles, in recent years, vacuum coating has been developed. Among these vacuum coating processes, magnetron sputtering has been utilized commercially to deposit antimicrobial metal as it is a well-developed coating technique [12,13]. Research and development into such processes are ongoing [14–16]. These efforts move a large step toward making textile treatments cleaner. One emerging method of magnetron sputtering involves the effective control of the power delivery to the target by HIPIMS, where a high voltage pulse is generated by the power source and operated at high peak power for a very short period causing a high degree of ionization of the sputtered species, in a manner similar to that associated with an arc source [17,18]. This process enables HIPIMS to provide high plasma density, and so to produce a strongly adhering film at a reduced relatively low substrate temperature. With this unique power source, an antimicrobial treatment for the textile using silver as the raw material has been

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proposed [19]. This aim of this study is to form antibacterial silver-coated PET fabrics by HIPIMS and to examine their antibacterial efficacy and durability against washing and rubbing.

2. Experimental

2.1. Preparation of silver coating on PET fabric

HIPIMS was carried out in a vacuum chamber in which was placed a single rectangular magnetron source with a target area of 11.4 cm × 34.5 cm (effective eroded area of 282 cm^2, from which power density was calculated) and the HIPIMS system is shown schematically in Fig. 1. The HIPIMS power supply was manufactured by Taiwan Power Tech., and comprised a DC-1020A DC power unit and a SPIK 2000A-20 pulsing unit. The target current and voltage were measured using a Tektronix CT-4 high-current probe and a high-power voltage differential probe, respectively; the signals thus obtained were displayed and recorded using a Tektronix TDS 2022B digital oscilloscope.

Poly(ethylene terephthalate), known as PET, in the form of a fabric with dimensions 29 cm × 19 cm was used as a substrate. It had a single filament diameter of 39 inch, a density of 170 D and a weight of 273.4 g/m. Before being coated with silver, oxygen plasma treatment was performed to remove surface contamination and activate the surface of the fabric. In this stage, an RF power supply was used for substrate bias, keeping a constant output power of 50 W to generate glow discharge over the substrate, whilst admitting oxygen gas into the coating chamber to control an working pressure at 1.33 Pa for 0 min, 1 min, 3 min and 5 min, respectively. Treatment parameters are listed in Table 1. During deposition, the PET fabric was maintained at a distance of 25 cm from a pure silver target (99.99%). The working pressure was fixed at a constant (total pressure of 0.133 Pa). Table 1 presents the coating parameters. All of the deposition runs were performed without additional substrate heating. To ensure that the coating was conducted in the HIPIMS mode, the a peak target current was plotted versus target voltage in Fig. 2, which reveals that the peak target current of 13 A (equivalent to a peak target current density of 0.06 A/cm^2 with a corresponding peak power density of 44.8 W/cm^2) flowed when a target voltage of 800 V was applied. This value was at the lower limit for the HIPIMS mode that has been presented elsewhere [17,18]. Deposition time was varied to control the thickness of the films, whose microstructures, mechanical properties and antibacterial efficacies were determined.

Table 1
Experimental parameters for coating PET fabric with silver.

Oxygen plasma pre-treatment parameters	
RF power (W)	50
Working pressure (Pa)	1.33
Processing time (min)	1, 3, 5
Silver coating parameters	
Target voltage (V)	800
Peak target current (A)	13
Peak target current (A/cm^2)	0.06
Average target power (W)	960
Peak target power (W)	10,400
Peak target power density (W/cm^2)	44.8
Duty cycle, T_{on}/T_{total} (μs)	200/1000
Pulse frequency (Hz)	1000
Flow rate of argon sputtering gas (sccm)	50
Working pressure (Pa)	0.13
Sub. rotation speed (rpm)	6
Deposition time (min)	1, 3, 5, 10

2.2. Microstructural characterization of the silver coatings

The crystalline structure of the deposited films was identified using a Bruker D8SSS grazing incident angle X-ray diffractometer (GIXRD) with CuKα radiation at a grazing angle of 1°. A field-emission scanning electron microscope (FE-SEM, HITACHI S4800) was used to observe the surface morphology and cross-section of the deposited silver films. The elemental composition of the obtained silver films was determined using an energy dispersive spectrometer (EDS, HORIBA EMAX400) that was installed on the FE-SEM.

2.3. Durability and mechanical property

Two tests were utilized to evaluate the durability of the deposited silver coatings. The first was the color fastness during rubbing test (ISO 105-X12:2002) [20] and the other was the color fastness to washing test (ISO 105-C02:1989) [21]. For the first, the coated PET

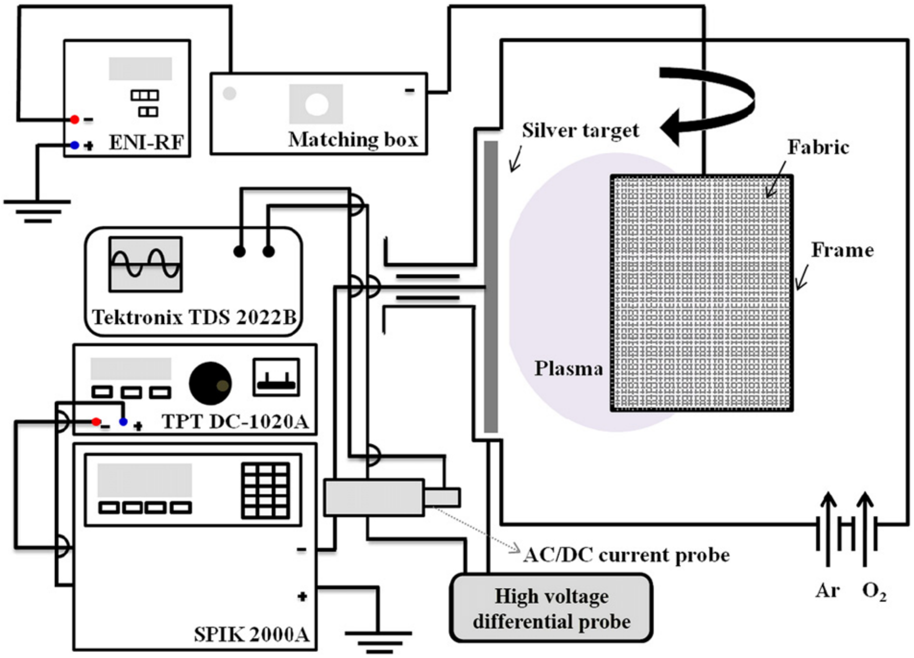

Fig. 1. HIPIMS system used in this work.

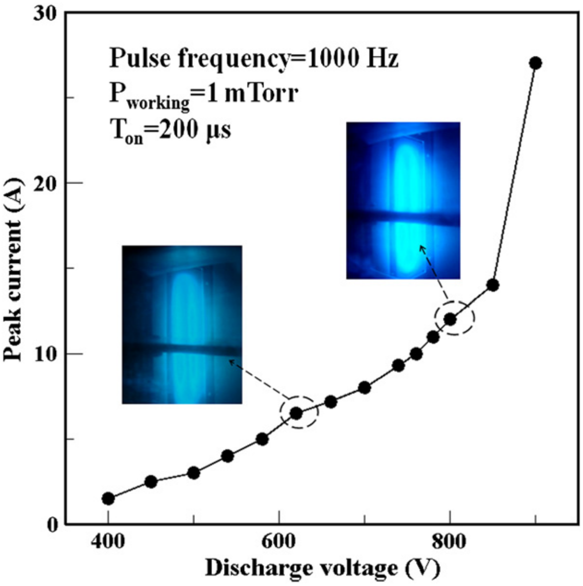

Fig. 2. Peak target current as a function of target voltage; insets show visible plasma discharge.

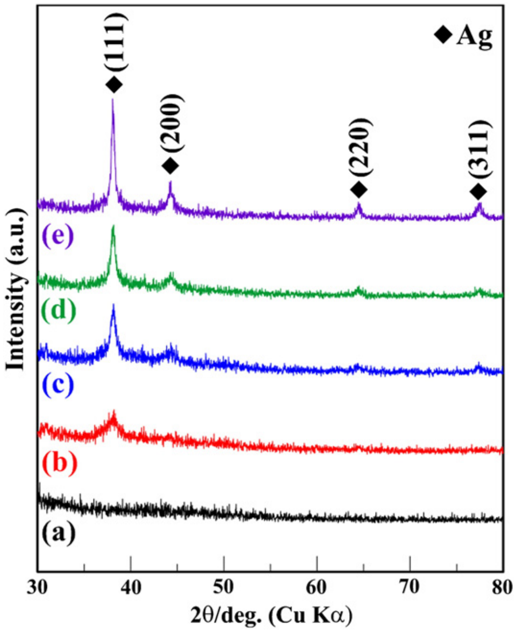

Fig. 3. XRD pattern of (a) PET raw fabric and the silver-coated fabric formed by deposition for (b) 1 min, (c) 3 min, (d) 5 min and (e) 10 min.

fabric as the specimen was rubbed against a standard cotton fabric for 100 cycles, and the extent of the transfer of the silver stain onto standard cotton fabric was evaluated using grey scales from Grade 1 (least color fastness, or the highest degree of transfer of the silver coating to the white cotton fabric) to Grade 5 (the highest color fastness). For wet rubbing, the standard cotton fabric was wetted with water and rubbed against the specimen as mentioned above. In the color fastness during washing test, each washing cycle involved mechanical agitation at 50 °C ± 2 °C for 30 min using a soap solution, followed by rinsing in water and drying at 60 °C ± 2 °C for 20 min. After 20 cycles of washing, the difference between the color of the silver-coated fabric with that of the as-coated fabric was evaluated using a Datacolor SF600X color meter. The color difference value is an index of the color fastness during washing.

The mechanical property test of the silver-coated fabrics concerns the change in mechanical properties that is caused by coating with silver. A vertical automatic test stand (ALGOL, JSV-H1000) with a digital push-pull gauge (ALGOL HF-100) was used to perform the tensile test to determine the ultimate load of the fabric. The test piece, clamped by the fixture, was a piece of the coated fabric with dimensions 10.0 cm × 1.5 cm. A strain gauge (KFP-2-120-C1-65L1M2R, Kyowa Electronic Instruments Co. Ltd., Tokyo, Japan) was attached to the surface of the test piece and connected to a data acquisition unit (DBU-120 A, Kyowa Electronic Instruments Co. Ltd.), which recorded elongation throughout the tensile test.

2.4. Antibacterial efficacy test

The antibacterial efficacy was quantitatively evaluated according to the JIS L 1902: 2008 [22]. *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) bacteria were the test inoculums. The test was performed aseptically to ensure the absence of any contamination. Each of the tests was carried out using an initial concentration of 1 ± 10^5 bacteria/ml. The inoculum was inoculated onto the raw PET fabric and the coated fabric of dimensions 28 mm × 28 mm. (The pieces of fabricated were hosted in sterilized Petri dishes at a temperature of 37 °C ± 1 °C for 18 h. Then, the bacteria in the Petri dishes were completely washed out, serially diluted and finally applied to standard agar plates. After 24 h of culturing, colonies of bacteria appeared on these agar plates in amounts that depended on the antimicrobial efficacy of the pre-inoculated specimens. Quantitative results were obtained by counting the colonies on the agar plates to determine the number of viable bacteria. The bacteriostatic value (*S*) and bactericidal value (*L*) of the silver-coated fabrics were calculated. The significance of *S* and *L* is described in detail in JIS L 1902: 2008 [22].

3. Results and discussion

3.1. Structural identification and morphological observation of silver coatings

Fig. 3 presents the XRD patterns of PET fabric and the silver coated PET fabric that was obtained using different deposition times. The PET raw fabric yielded no significant diffraction peak, indicating that the PET polymeric material was amorphous. The silver-coated PET fabric yields diffraction peaks that are attributed to the silver; their intensities increase with deposition time, clearly because of the increase in

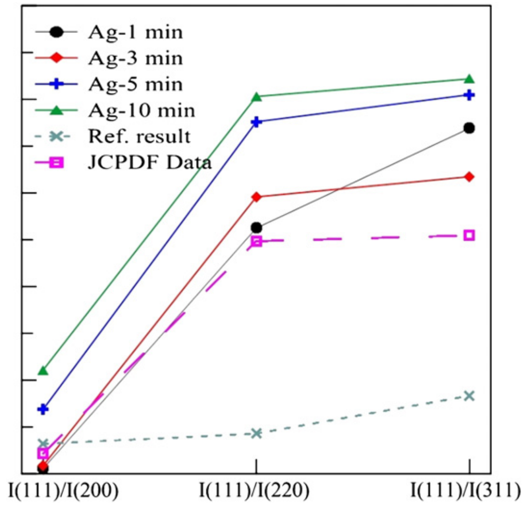

Fig. 4. Ratios of intensity of diffraction peak (111) to those of (200), (220) and (311) peaks, calculated from Fig. 3. For comparison, the result obtained by Wang using RF sputter deposition and JCPDF data are presented.

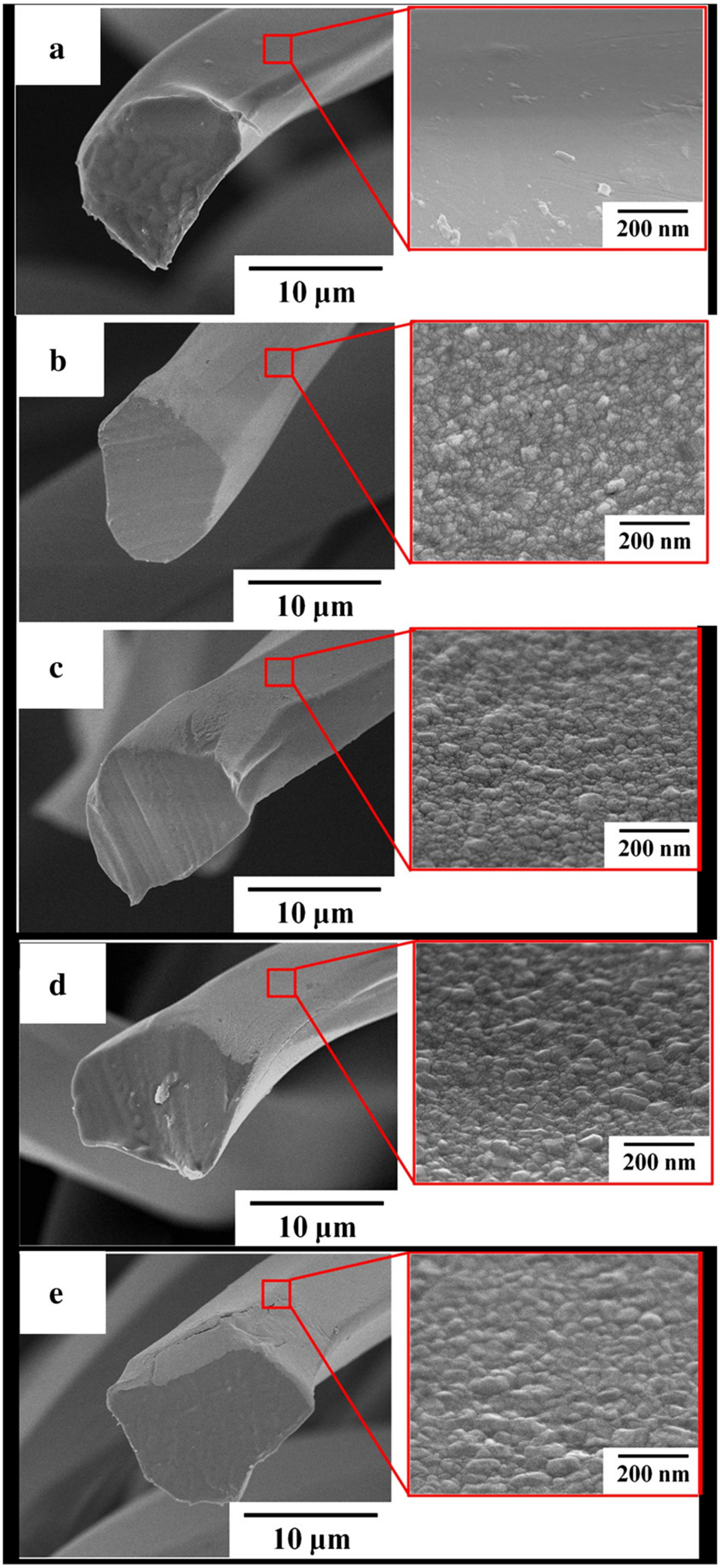

Fig. 5. Surface morphology of the (a) single bare PET fiber and silver film deposited for (b) 1 min, (c) 3 min, (d) 5 min and (e) 10 min, respectively.

film thickness. Notably, the silver film that was formed in this study may grow with a preferred orientation for during various deposition times, yielding a strong (111) diffraction peak. Wang et al. used conventional RF sputter deposition to form silver coatings on nylon fabric and PET fabric. The I(111)/I(200), I(111)/I(220) and I(111)/I(311) ratios in this investigation were much higher than were those obtained by Wang et al. [23]., as presented in Fig. 4, which reveals that the ratios of intensity of the (111) peak to those of other diffraction peaks of the silver coatings, obtained using HIPIMS in our study, considerably exceed that of those obtained by RF sputter deposition or from the JCPDF data. Also, increasing the deposition time increases the overall ratios of the intensity of (111) to those of other peaks. Accordingly, the strong (111)-preferred orientation of the grown silver film is evident herein in this study. It is attributed to the intense high-energy ion bombardment that results from the HIPIMS plasma [24]. This effect may improve both the adhesion and durability of the film, as demonstrated in the following section.

Fig. 5 displays the surface morphologies of a randomly selected (a) single bare PET fiber and silver films that were deposited for (a) 1 min, (b) 3 min, (c) 5 min and (d) 10 min. As observed, the bare PET fiber had an average diameter of 10 μm and a smooth surface. After being coated with silver, the surface of the PET fibers was granular and rough, independently of the deposition time, revealing the successful deposition of silver on the PET fabric by HIPIMS. The silver-coated PET fiber does not exhibit observable damage by the HIPIMS plasma, nor any sign of degradation of the PET fiber, itself.

Fig. 6 presents the cross-sectional view of the silver films that were grown on the fabric for various deposition times. The thicknesses of the silver films that were deposited for 1 min, 3 min, 5 min and 10 min are estimated to be approximately 35 nm, 56 nm, 65 nm and 176 nm, respectively. The literature has demonstrated that the silver coating that is formed by using conventional sputter deposition exhibits Volmer-Weber growth [25,26] (or so called island-by-island growth) because of the large cohesive force among the silver atoms. Fig. 6(a) clearly presents the island-like grains of the film that was formed in 1 min of deposition, which somewhat coalesce. The films that were deposited for 3 min, 5 min and 10 min exhibit much stronger coalescence of silver grains, and so are continuous, as shown in Fig. 6(b), (c) and (d). These results indicate that the silver film deposited by HIPIMS exhibits Volmer-Weber growth as in conventional sputter deposition. Similarly, the HIPIMS-derived silver films reportedly exhibit Volmer-Weber growth [27] in which the use of HIPIMS plasma reduced time that is required for island coalescence. Unfortunately, owing to its limited resolution, SEM cannot distinguish individual islands that may form when the silver film is formed in less than 1 min of deposition. Therefore, the time for island coalescence cannot be resolved and detailed observation using a high-resolution microscope is required to do so. Clearly, in this study, 1 min of deposition time suffices for islands to begin to coalesce. The growth mechanism found in this study is Volmer-Weber growth rather than layer-by-layer (or Frank–van der Merwe) growth [28], partly because of the result of using lower peak power (several kW/cm^2) for coating herein than normally used (44.8 W/cm^2) [18,27].

Table 2
Grade of color fastness during rubbing (under both dry and wet conditions) of HIPIMS silver-coated PET fabric prepared either without or with O_2 plasma pre-treatment.

Samples	Grade for color fastness to rubbing	
	Dry rubbing	Wet rubbing
Silver coated, without O_2 plasma pre-treatment	4	3
Silver coated, with 1 min O_2 plasma pre-treatment	5	4
Silver coated, with 3 min O_2 plasma pre-treatment	5	4
Silver coated, with 5 min O_2 plasma pre-treatment	5	4

3.2. Durability and mechanical properties of silver-coated fabric

Table 2 presents the results of color fastness during rubbing (both dry and wet). To examine the influence of pre-treatment with oxygen plasma on the coating adhesion, the fabric was pre-treated with oxygen plasma for various times and a fixed deposition time of 5 min for silver coating was utilized. The results clearly demonstrate that all of the fabrics that had undergone oxygen plasma pre-treatment had a

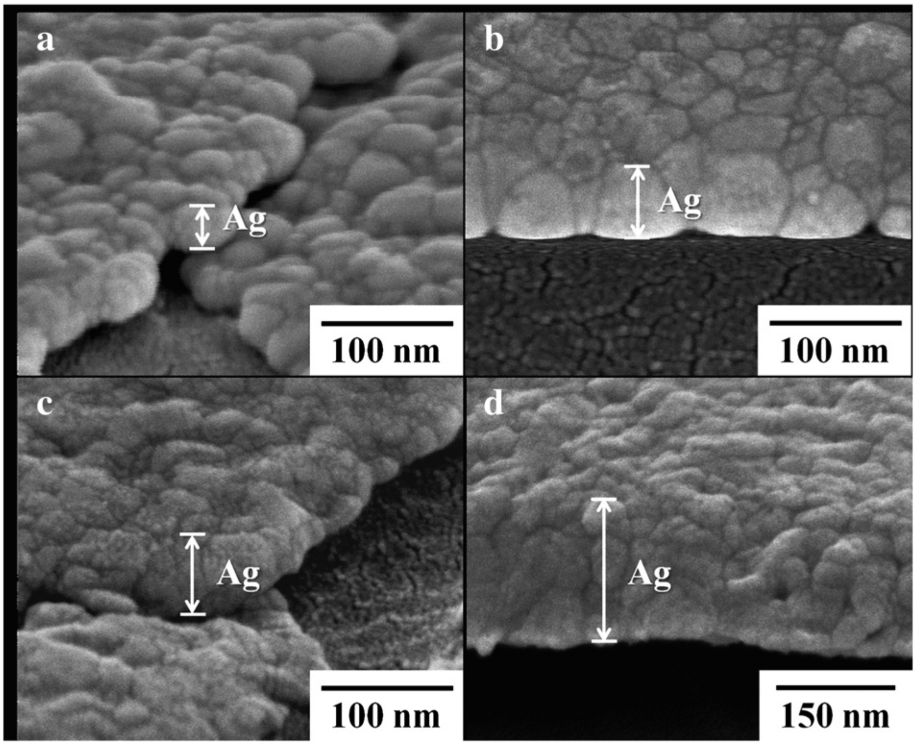

Fig. 6. Cross-sectional image of silver coating obtained at various deposition times of (a) 1 min, (b) 3 min, (c) 5 min and (d) 10 min.

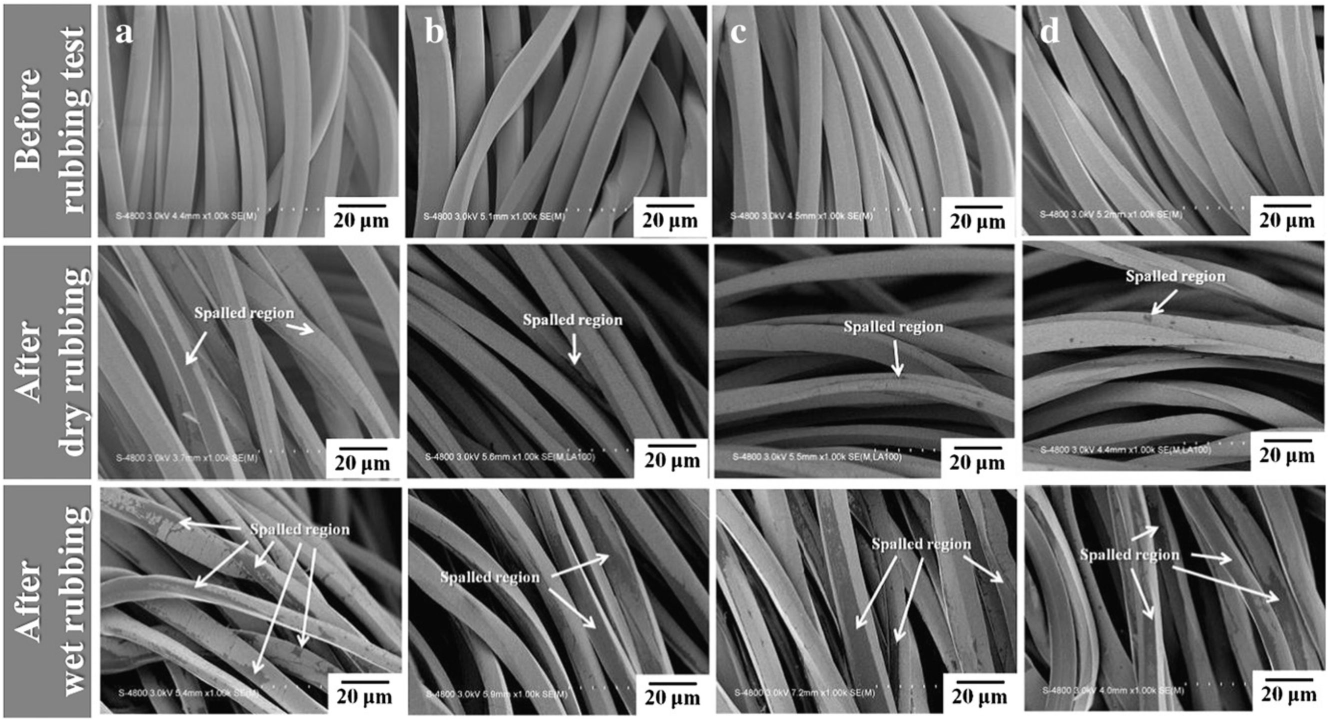

Fig. 7. Surface morphology of silver-coated PET fabric sample following test of color fastness during rubbing; (a) without pre-treatment with oxygen plasma and (b), (c) and (d) with pre-treatment with oxygen plasma for 1 min, 3 min and 5 min, respectively.

higher a higher resistance against rubbing than those that had not been pre-treated, apparently because of the pre-treatment improved film adhesion. Oxygen plasma pre-treatment for 1 min effectively improved film adhesion. In all cases, the color fastness of the silver-coated fabric during rubbing under wet conditions was lower than under dry conditions. Ultimately, oxygen plasma pre-treatment yielded the PET fabric, coated with silver by HIPIMS, with Grade 5 with dry rubbing. Based on these results, plasma pre-treatment is important and effective when HIPIMS is employed for film preparation just as it is when conventional sputter deposition is used [29]. Pre-treatment with oxygen plasma improves film adhesion because it provides chemical activation and the required PET surface functionalization. The chemical effect of oxygen plasma pre-treatment, in this regard, is crucial, beyond its effectiveness in the physical cleaning of the surface [30,31].

Fig. 7 presents SEM photographs of the samples after the color fastness during rubbing test. In Fig. 7(a), whereas the as-deposited silver coating that uniformly covers all of the fabrics, the coating without oxygen plasma pre-treatment exhibits significant spalling off from the fibers after the rubbing test under either dry or wet conditions, but particularly under wet conditions. In Fig. 7(b), (c) and (d), the as-deposited samples that underwent oxygen plasma treatment exhibited similar surface morphologies to that of the sample that did not undergo oxygen plasma treatment. However, the silver-coated fabric samples were damaged in a way by dotted flake-off under dry rubbing conditions, whereas those coated under wet rubbing conditions exhibited delamination over a large area. Evidently, the silver-coated fabric sample without oxygen plasma pre-treatment exhibited more severe spallation than the samples that had not undergone oxygen plasma pre-treatment. Interestingly, 1 min of pre-treatment is as effective as longer pre-treatments and this pre-treatment was applied to the samples in the washing test. The above SEM observations are consistent with the quantitative results in Table 2.

Fig. 8 shows the results of the test of color fastness during washing. Fig. 8(a) reveals the change in the appearance of the silver-coated PET fabric upon washing. The coated fabric is covered with local stains. Further examination by SEM indicates that the visible stains are caused by the spallation of the silver coating from the PET fiber, which is presented in Fig. 8(b). Notably, however, despite the spallation, most of the silver coating adheres strongly to the fabric and the remaining parts of the films exhibit no sign of attrition loss, but adhere strongly to the fibers, as shown in Fig. 8(c). The results of the test of color fastness during washing reveal that the silver

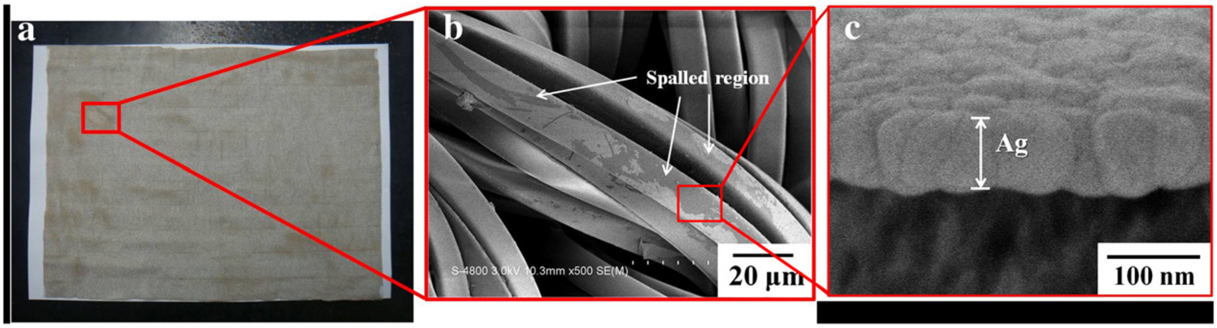

Fig. 8. (a) Appearance to the naked eye), (b) SEM surface image and (c) SEM cross-sectional view (at high magnification) of silver coating that remains on washed fabric.

coating on the PET fabric is resistant to critical washing environmental and these findings are consistent with those of studies in which HIPIMS technology is utilized [19]. The strong (111) preferred orientation, such that the closest packing of the grown silver film is in the direction of growth, synergistically contributes to this effect, and the oxygen plasma pre-treatment undoubtedly also plays significant role for chemical modification.

The effect of the silver coating on the mechanical properties of the fabric is determined by performing a tensile test. Fig. 9 plots the resulting load-displacement curves of bare PET fabric and silver-coated fabric. The curve for bare PET fabric is monotonic until fracture at a tensile force of 7.59 kgf, corresponding to a displacement of 35 mm, revealing that the PET fabric is rather brittle. In contrast, the curve of silver-coated fabric reaches an ultimate tensile force of 9.83 kgf, followed by elongating with a zigzag manner to a lower fracture tensile force close to 7.59 kgf. This corresponds to a displacement of the silver-coated fabric of 45 mm, which exceeds that, 35 mm, of the bare PET fabric. The silver coating increases the tensile force and the displacement over those of the bare PET fabric by enlarging the diameter of the fiber due to the ductile nature of silver metal. The silver coating influences the mechanical properties of the PET fiber in a way that increases both tensile force and elongation, revealing that the HIPIMS technique does not degrade the PET fabric.

3.3. Antimicrobial activity of the silver-coated fabric

Table 3 presents the bacteriostatic values (*S*) and bactericidal values (*L*) of the bare PET fabric and silver-coated fabric. The bare PET fabric has values of almost zero, as expected. The coated fabrics with the silver film that was deposited for 1 min had *S* values for *S. aureus* and *E. coli* of 5.7 and 6.2, respectively. These values represent satisfactory antimicrobial performance, according to the JIS standard, which states that an *S* value of less than 2.0 indicates absence of antibacterial efficacy. Interestingly, deposition of the silver coating for 1 min provides the optimal antimicrobial performance. Additionally, all of the silver-coated fabrics have an *L* value of 3.2, which indicates antibacterial efficacy according to the JIS standard, whereas the bare PET fabric has an *L* value of 0.0. Twenty cycles of washing do not change the *S* or *L* values of the silver-coated values. The results clearly indicate that the silver-coated PET fabric that is formed using the HIPIMS technique exhibits strong antimicrobial properties for both *S. aureus* and *E. coli* even after only 1 min of deposition and 20 washing cycles. Doubtlessly, the silver-coated fabric exhibits excellent antibacterial efficacy even when it had undergone 20 cycles of washing, with local spallation of the coating. These facts demonstrate that the remaining silver film on the fabric persists to provide excellent antibacterial efficacy [32], supporting the claim that the remaining silver film releases some antimicrobial Ag^+ ions even when it underwent 20 cycles of washing [33].

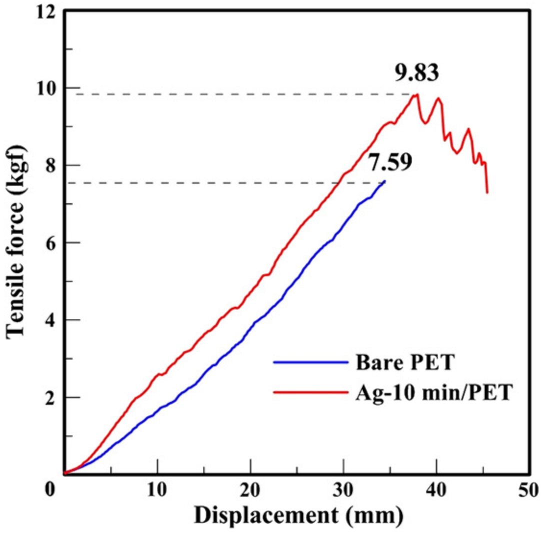

Fig. 9. Load-displacement curve of bare PET fabric and silver-coated fabric.

Table 3
Antimicrobial efficacy of bare PET fabric, silver-coated PET fabric with silver film formed using different deposition time, and a specimen that had undergone 20 cycles of washing.

Test method: JIS L 1902: 2008 (Quantitative test and absorption method)

Bacterial	Sample	*S*	*L*
S. aureus	Bare PET	0.0	<0
	Ag coated PET for 1 min	5.7	3.2
	Ag coated PET for 3 min	5.7	3.2
	Ag coated PET for 5 min	5.7	3.2
	Ag coated PET for 10 min	5.7	3.2
	Ag coated PET for 5 min and wash tested	5.7	3.2
E. coli	Bare PET	0.2	<0
	Ag coated PET for 1 min	6.2	3.2
	Ag coated PET for 3 min	6.2	3.2
	Ag coated PET for 5 min	6.2	3.2
	Ag coated PET for 10 min	6.2	3.2
	Ag coated PET for 5 min and wash tested	6.2	3.2

**S*: Bacteriostatic value; *L*: Bactericidal value.

4. Conclusion

A silver coating can be deposited on PET fabric by HIPIMS, with a crystal structure with a (111) preferring orientation, such that it uniformly covers the fibers of the fabric. Pre-treatment with oxygen plasma for just a single minute effectively improves film adhesion and its resistance in both dry and wet rubbing tests, in which its color fastness is grade 5 and grade 4, respectively. The coated fabric retains the mechanical properties of the original bare fabric. Antimicrobial performance tests reveal that a silver film that is deposited for more than 1 min has strong bacteriostatic (>2.0) and bactericidal (>0) effects, according to the JIS Standard. Furthermore, coated fabric retains its antimicrobial efficacy after 20 cycles of washing, indicating its long-term durability. In summary, the HIPIMS silver coating improves film adhesion, color fastness during washing and antimicrobial efficacy and can potentially be applied to antimicrobial textiles.

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