Research Article

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Surface functionalization of nanostructured Cu/Ag-deposited polypropylene fiber by magnetron sputtering

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Abstract: Metallic nanoparticles are widely used due to their superior electrical, antimicrobial, and electromagnetic shielding characteristics. In this work, the surface functionalization of polypropylene (PP) fibers using magnetron sputtering with pure Cu and Ag targets in the presence of Ar gas was systematically investigated, in detail, in terms of surface morphology, tensile, abrasion resistance, moisture regain, antibacterial, and electrostatic properties. The results indicated that the nanocomposite films deposited on the PP surface were even and dense under proper treatment conditions. Compared with pristine fiber, breaking tenacity, abrasion resistance, and antibacterial properties of the Cu/Ag-deposited PP fibers were significantly improved, whereas the extension at break and moisture regain decreased in different degrees. Also, the electrostatic property of treated PP fabrics was studied. This work reveals that surface functionalization of Cu/Ag-deposited PP fiber is versatile, and the surface treatment that uses metallic nanoparticles by magnetron sputtering is a promising approach for achieving multifunctional textiles.

Keywords: magnetron sputtering, surface functionalization, metallic nanoparticle, polypropylene fiber, multifunctional textiles

1 Introduction

The development of functional textile materials has recently gained considerable momentum (1–4), and there is an urgent need to use the fiber itself as a base element since such fibers could easily be processed into yarn and/or fabric structures by conventional textile means. Polypropylene (PP) fiber is a high-performance manmade material that has been widely used in many civil and industrial fields (5,6). However, the innately inert surface of manmade fibers is often not ideal (7). The properties of textile materials closely depend on the molecular structures of their surfaces and interfaces (8). Thus, it is necessary to modify the surface of PP fiber to enrich its surface functionalities without changing in bulk properties for many commercial applications.

Copper (Cu) nanoparticle has good antibacterial and thermal characteristics, superior biodegradability and high stability. Also, its metallic ions can restrict the activities of protein clusters and improve the surface of the deposited films. As a result, copper has gained extensive attention toward its use as a conductive material (9,10). In addition, silver (Ag) nanoparticle has aroused concern due to its unique properties, such as very low resistivity among all metals, heat radiation and chemical resistance, and antibacterial activity (11–16). Therefore, an Ag nanoparticle film is the optimal choice and an ideal candidate for fabricating multifunctional materials. Whether there was a synergism between Cu and Ag nanoparticles or not is an important issue clearly worth thinking.

Currently, a wide variety of surface modification techniques have been employed to build functional nanostructures on materials including dip-coating or spray method (17), chemical treatment (18), plasma treatment (19–24), and surface graft treatment (25). However, some waste solutions and other pollutants may generate in the chemical treatment. The aging behavior of the plasma-treated materials, pollution, and the high cost of surface grafting treatment limit their further use. Besides, the uniformity in

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the dip-coating or spray method is not satisfactory. Recently, magnetron sputtering, a type of physical vapor deposition, provides a promising way for the deposition of thin films due to its advantages, such as simplicity, environmental friendly, high speed, low working temperature, and is suitable for sputtering many kinds of polymer materials (1,26–30). For example, Wei et al. fabricated functional nanocomposite polypropylene nonwovens by surface coating with copper and silver using magnetron sputter coating. Wang et al. constructed polytetrafluoroethylene nanoparticles onto graphene oxide/polyester fabrics for oil adsorption. Long et al. reported a polypropylene nonwoven fabric via surface modification for improving its hydrophilicity. Xu et al. deposited Ag/Fc nanocomposite films on PET nonwoven surface, and the relevant properties were systematically characterized. However, a literature survey showed that little work has been carried out to modify surface of commercial PP fibers. Therefore, one aim of this work is to fill this gap.

The main goal of our research work is to develop easily processable and robust conductive PP fibers that are appropriate for many fields such as antibacterial resistance, Joule heating, and electromagnetic shielding. Herein, the functional surface of the commercial PP fibers was successfully constructed by using the magnetron sputtering technique with pure Cu and Ag targets in the presence of Ar gas. Effects of key process variables with respect to surface morphology, tensile, abrasion resistance, moisture regain, antibacterial and electrostatic properties were systematically investigated. Such fundamental work provides useful information for surface functionalization of polymer textiles in an eco-friendly, convenient, and rapid way.

2 Experimental

2.1 Preparation of Cu/Ag-deposited materials

In this work, the white PP fiber samples of 120 dtex/60 f, which were purchased from CTA High-tech Fiber Co., Ltd., China, were ultrasonically cleaned in an alcohol bath, and then they were repeatedly washed with deionized water. Finally, they were used as the substrates. The breaking tenacity and extension at break of fibers are 33.61 cN/tex and 27.87%, respectively. In addition, two sputtering targets, i.e., Cu and Ag (purity: 99.99%), were used, and the size of the two targets is $\Phi 60 \times 5$ mm.

Figure 1 graphically illustrates the schematic diagram of the JGP-450A magnetron sputtering system that is used to synthesize Cu/Ag nanocomposite thin film onto PP fiber surface. The vacuum system was a cylindrical chamber; the targets were mounted on the bottom, and the PP fibers were placed against the targets. Argon was used as the bombardment gas. During the deposition process, the distance between the targets and the substrate was kept 6 cm, approximately. Prior to the deposition, the Argon gas was entered for about 5 min to remove the impurities of the target surface, and the sputtering chamber was pumped to achieve a base pressure of 5.0×10^{-4} Pa. In addition, the water-cooling system was used to control the temperature to avoid the deformation of PP fibers caused by high temperatures. The sample holder was rotated at a speed of about 100 rpm to ensure that the metallic particles were uniformly deposited onto the fiber surface.

Figure 1: The formation of Cu/Ag nanoparticles over PP surface by magnetron sputtering technique.

Table 1: Sample details

Constant parameter	Varying parameters	Values
The gas flow of Ar gas was kept constant at 15 SCCM (standard cubic centimeter per minute)	Sputtering time (min)	0, 15 , 30, 45
	Sputtering power (W)	0, 10, 30 , 50
	Sputtering pressure (Pa)	0, 0.5, 1 , 2

The processing parameters play an important role in influencing the final properties of the resultant PP; the experimental parameters are given in Table 1. Total nine samples were fabricated covering processing parameters in sputtering time, power, and pressure using a single factorial-analysis technique. The thickness of the layers under each condition was measured. Note that the process parameter values are represented in bolditalic type; the relevant fiber samples were prepared by varying one of the three parameters, keeping the other two parameters constant.

2.2 Characterization

2.2.1 Surface morphology observation

Surface morphologies of Cu/Ag-deposited fibers were observed by using a scanning electron microscope (SEM; Hitachi S-4800), and the size of nanoparticles can be estimated by the bar in SEM images with the aid of ImageJ software.

2.2.2 XRD test

The aggregation structures of PP fibers before and after treatments were analyzed by using an X-ray diffractometer (XRD) analyzer (D8-DISCOVER type) with a CuK α radiation source. The voltage and current used is 30 kV and 200 mA, respectively. XRD spectra were collected in the range of 5–80° at a scanning step length of 0.02°/s with a speed of 5°/min.

2.2.3 Tensile behavior

The tensile behavior of as-prepared Cu/Ag-deposited fibers was measured using a YG020 tensile tester. Forty replicates at a gauge length of 500 mm, test speed of 500 mm/min, and a preload of 0.5 cN/tex were measured per sample according to the China standard GB/T 3916-2013. At least 20 replicates were tested for each sample.

The difference significances between tensile data of fibers with varying technological parameters were calculated using ANOVA using SPSS statistical software 17.0.

Furthermore, box-whisker plots, which can provide some important data features such as central tendency, dispersion, and skewness, were used to characterize the variation of the tensile strength of Cu/Ag-deposited fibers under different technological parameters.

2.2.4 Abrasion resistance behavior

A Y731 cohesive force tester was introduced for evaluating the abrasion resistance of Cu/Ag-deposited fiber samples with different processing variables at a testing speed of 60 times/min. At least, 20 replicates were tested for each sample.

2.2.5 Moisture regain measurement

Moisture regain of the as-fabricated Cu/Ag-deposited PP fibers was adopted to estimate the hygroscopicity by a weight method. The samples with different technological variables were conditioned at $20 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ relative humidity (RH) for 24 h and weighed exactly (G) using a BS200S-WE1 electronic balance. Afterward, the fibers were dried in a YG747 oven at 110°C until a constant weight and weighed accurately again (G_0). The regain for each sample was determined in two replicates and calculated:

$$W = \frac{G - G_0}{G_0} \times 100\%. \quad (1)$$

2.2.6 Antibacterial evaluation

Two test strains, *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*), were used to assess the antibacterial property of Cu/Ag-deposited PP fibers by observing their inhibition zone, in accordance with AATCC 90-2011. First, PP fiber at certain treatment conditions

(30 W, 1 Pa, 45 min) was chosen, and a plain weave fabric made of 100% fiber above was made with a count of 360 ends/10 cm $\times$ 320 picks/10 cm, respectively. Then, a circular specimen was cut by hand with a diameter of 25 mm. In addition, a reference woven fabric made of 100% pristine PP fibers was prepared for comparison.

2.2.7 Electrostatic measurement

Further, the electrostatic property of the above Cu/Ag-deposited PP fabric samples (with a size of 45 $\times$ 45 mm) was measured using a YG(B)342E induction-type electrostatic tester, in accordance with GB/T 12703.1-2008. A discharge test with 10 kV was performed in this work. Two indices, i.e., electrostatic voltage and half decay time, were obtained. At least three replicates were tested for each sample.

3 Results and discussion

3.1 Morphological structure and XRD analysis

3.1.1 Surface morphology at different sputtering stages

The morphologies and microstructures of Cu/Ag-deposited PP fibers at different magnetron sputtering stages were examined by using SEM, and the results are shown in Figure 2. The pristine PP fibers are slightly rough and a certain stripping phenomenon is evident, as shown in Figure 2a. During the sputtering process,

the nanocomposite film deposited onto the fiber surface is not very even. In some regions, the bare fiber surface exists. After a certain sputtering treatment time, the as-deposited Cu/Ag coating covers all the surface of PP fibers gradually and completely, as shown in Figure 2b. Finally, the coated fiber looks visually metallic, as shown in Figure 2c.

Based on the above SEM analysis, a reasonable growth process of Cu/Ag nanocomposite film deposited onto PP fiber surface can be explained as follows: The growth of thin film is a non-equilibrium state. During the sputtering process, three possible behaviors of Ag and/or Cu atom, that is, deposition, desorption, and migration exist, illustrated in Figure 2d. A Cu/Ag-deposited thin, homogeneous film gradually formed onto the fiber surface after three stages (I, II, and III), converting from discontinuous to continuous state. Finally, a Volmer–Weber growth mode can be concluded.

3.1.2 Effect of technological parameters on surface morphology

Several factors affect the final performance of Cu/Ag-deposited PP fibers by magnetron sputtering, in this work, three important influential factors namely, sputtering time, sputtering power, and sputtering pressure were used to clarify their effects on the morphological structure of samples. On a whole, compared with the pristine PP, the surface of fibers after magnetron sputtering became rough and sputtered nanoparticles, and a Volmer–Weber growth mode of a thin film can be seen in Figure 3. The structure morphology of fiber for various sputtering times is shown in Figure 3a. As expected, the nanocomposite film thickness increased with increased

Figure 2: (a–c) SEM images of Cu/Ag-deposited PP fibers at different magnetron sputtering stages and (d) the growth process of Cu/Ag thin film deposited on PP fiber.

Figure 3: Effects of technological parameters by magnetron sputtering, viz., (a) sputtering time, (b) sputtering power, and (c) sputtering pressure on surface morphologies of PP fibers.

sputtering time. Observation by SEM of PP at various times of deposition indicated that the fiber surface roughness increased and became denser after being sputtered. Figure 3b displays the surface morphologies of nano-metal films that were deposited for varying sputtering power. As observed, the original PP fiber had a smooth surface, whereas after being treated, the surface of PP fiber became granular and rough, independently of the sputtering power, revealing the successful deposition of Cu/Ag on PP fiber. In addition, as shown in Figure 3c,

with increased sputtering pressure, metal atoms had increased opportunities to collide into each other and form clusters, thereby creating larger particle sizes.

3.1.3 XRD analysis

Figure 4 illustrates three typical XRD patterns of Cu/Ag-deposited PP fiber samples with varying treatment conditions, that is, the conditions were selected based on the

Figure 4: XRD patterns of PP samples with varying treatment conditions: (a) 30 W, 1 Pa, 45 min; (b) 50 W, 1 Pa, 15 min; and (c) 30 W, 2 Pa, 15 min.

highest value of predetermined processing parameter in a single-factor experiment. It can be clearly seen that four significant characteristic peaks around 38.48° , 44.68° , 66.71° , and 78.82° , corresponding to crystallographic planes (111), (200), (220), and (311) of Ag, respectively (30). In addition, a typical characteristic sharp peak around 47° can be observed, which is assigned to the crystal plane (200) of Cu. In short, based on the above XRD analysis, we conclude that the Cu and Ag nanoparticles were both successfully deposited on the surface of treated PP fibers.

3.2 Tensile behavior and statistical analysis

3.2.1 Experimental analysis of tensile data

The morphological structure of PP fibers has substantial impacts on the final behavior. Herein, the effects of sputtering technological parameters on the tensile response of Cu/Ag-deposited PP fibers were experimentally investigated.

Figure 5a reports the breaking tenacity of Cu/Ag-deposited PP fibers based on sputtering times. Compared

Figure 5: Effects of technological parameters: (a and b) sputtering time, (c and d) sputtering power, and (e and f) sputtering pressure on the tensile properties of PP fibers.

with the pristine PP fiber, the breaking tenacities of Cu/Ag-deposited fibers were higher. The tensile strength progressively increases with increasing sputtering time up to 45 min. This is because the deposited Cu/Ag nanoparticles gradually cover the fiber surface, strengthening some weak parts of the pristine fiber. Therefore, the breaking strength of fibers increases consequently. As can be seen in Figure 5c, compared with the untreated fiber, the breaking tenacities of Cu/Ag-deposited fibers were higher. The tensile strength progressively increases with an increase of the sputtering power up to 50 W. The number of nanostructured Cu/Ag particles sputtered out the target increases with an increase in sputtering power, and the effective particles deposited onto the fiber surface gradually increases, which, in turn, results in an increased breaking strength. As seen in Figure 5e, compared with untreated fiber, the breaking tenacities of Cu/Ag-deposited fibers were higher. Tensile strength progressively increases with increasing sputtering pressure. The moving speed of Cu/Ag nanoparticles sputtered out of the target increases, and the nanoparticles deposited onto the fiber surface increases correspondingly. Finally, a relatively compacter nanocomposite film was formed at the fiber surface, and the strength increases correspondingly.

With respect to the extension at the break, on the whole, when sputtered with a metal film, the lower values of tensile strain were obtained, irrespective of

the sputtering processing parameter considered (seen in Figure 5b, d, and f). This is because the rigidity of Cu/Ag is greater than the PP; so, the rigidity of the material increased when sputtered with metal atoms.

In addition, the difference in mean values between groups was analyzed for statistical significance using one-way ANOVA. As given, the difference between each processing variable was significant with P value (<0.05), indicating that the preparatory parameters were fundamentally essential.

3.2.2 Boxplots for quantifying tensile data

The correspondence between the box-whisker plot shape and tensile data distribution is illustrated in Figure 6a. Three typical distributions, namely symmetric, negative/left skewed, and positive/right skewed may appear in box-whisker plot form. The results in Figure 6b–d show boxplots for tensile strength of Cu/Ag-deposited fiber samples with respect to varying processing parameters. The experimental results demonstrate that the strength of coated fibers was remarkably higher than that of the untreated ones. The conclusion is drawn from the lower position of the box and whiskers associated with the untreated fiber and the smaller median, which indicates the midpoint of distribution. On a whole, a higher value

Figure 6: (a) Schematic of typical distributions in a boxplot form; boxplots of (b) sputtering time, (c) sputtering power, and (d) sputtering pressure-dependent tensile strength of PP fibers.

of the sputtering time, power, and pressure results in higher tensile strength, irrespective of the processing variables considered, and vice versa. With consideration of the shape of the tensile distribution, the boxplot for the untreated group is positively skewed, whereas for all the treated groups, the shapes look symmetric. Interestingly, the tensile variation trend of fibers between 30 and 50 W is a bit different from the result shown in Figure 5c, which further indicates that the box-whisker plot is a simple but powerful analytical tool that can reveal more information about the underlying characteristics of the tensile distribution.

3.3 Abrasion resistance

The working mechanism of the Y731 cohesive force tester used herein is graphically illustrated in Figure 7a. An as-prepared Cu/Ag-deposited PP fiber sample with a “W” letter pattern was fed into the machine and fixed by the hooks, then the metal card panel was moved back and forth until it was broken. Finally, the corresponding abrasion-resisting number was recorded.

It can be clearly observed in Figure 7b–d that the abrasion-resisting number of Cu/Ag-deposited PP fibers quickly increased, irrespective of the processing parameters considered. For example, the abrasion-resisting number of pristine PP fiber was about 360, whereas the maximum number reached about 440 after the magnetron sputtered (see Figure 7c). The reason may be ascribed that after magnetron sputtering, the surface of PP fibers became rough and the metal nanoparticles were dispersed uniformly on the surface of PP fibers. The increased weight of metal-coated fibers is also responsible for enhanced abrasion resistance property. In addition, it should be noted that the enhanced abrasion resistance performance of as-prepared Cu/Ag-deposited PP fibers illustrated that metal nanoparticles were successfully deposited on the fiber surface and combined strongly.

3.4 Moisture regain

Moisture regain is often used to evaluate the hydrophobic behavior of a textile material. As seen in Figure 8, the

Figure 7: (a) Schematic of abrasion resistance test; (b–d) effects of technological variables, i.e., sputtering time, power, and pressure, on the abrasion resistance behavior of PP fibers.

Figure 8: Effects of technological parameters: (a) sputtering time, (b) sputtering power, and (c) sputtering pressure on the moisture regain property of PP fibers.

pristine PP fibers exhibited excellent hydrophobic properties, and the moisture regain was below 2%. When sputtered with a metal film, Cu/Ag-deposited PP fibers became more hydrophobic, irrespective of the process variables considered. The experimental results revealed that the moisture regains decreased sharply. The reason may be ascribed that the Cu/Ag nanoparticles changed the surface structure of PP fibers and made the fiber surface rough after magnetron sputtering, the nanoscale roughness and lower surface energy by the metal nanoparticles generated the robust hydrophobic performance successfully.

3.5 Antibacterial durability

In this study, a woven fabric made of 100% Cu/Ag-deposited PP fibers with a condition of 30 W, 1 Pa, 45 min was chosen as a representative for assessing antibacterial activity. As seen from Table 2, the widths of the inhibition zone of Cu/Ag-deposited PP fabric were 5.90 and 6.40 mm for *E. coli* and *S. aureus*, respectively. The experi-

mental result reveals that the thickness of the Cu/Ag nanocomposite layer was 46 nm for the deposited PP fiber (30 W, 1 Pa, 45 min), and the thin layer was responsible for the excellent antibacterial performance. By contrast, the pristine fabric was not antibacterial for both strains.

To comprehensively evaluate the antibacterial durability of treated PP fabric, the antibacterial variation of fabric undergoing various external damages was also studied, containing bending deformation and tape peeling. As seen in Table 2, the antibacterial activity remained essentially unchanged, maintaining the widths of 5.80 and 6.20 mm for *E. coli* and *S. aureus*, respectively, after bending deformation for 50 cycles. Also, a 3M tape was used to conduct the peeling test. The result revealed that the antibacterial activity remained at a high level after 20 tape-peeling cycles, with only a 5.1% and 7.8% decrease. The all-encompassing antibacterial reliability demonstrates the mechanical robustness and fastness of as-prepared Cu/Ag coating.

3.6 Electrostatic evaluation

The four fabric samples mentioned earlier were further used to evaluate the electrostatic behavior. It is seen from Table 3 that, with respect to the Cu/Ag-deposited PP fabrics, the introduction of nanocomposite film results in shorter half decay times compared with that of the pristine PP one. Furthermore, the half-decay time of treated fabrics increased marginally following 50 bending and/or 20 tape-peeling cycles. The minor damage of Cu/Ag nanolayers to varying degrees was responsible for the increased half decay time. In short, the electrostatic reliability reveals the mechanical robustness and fastness of Cu/Ag deposition.

Table 2: Antibacterial results of PP fabric samples under different conditions

PP fabric samples	Width of inhibition zone (mm)	
	<i>E. coli</i>	<i>S. aureus</i>
Pristine fabric	0.00	0.00
Cu/Ag-deposited fabric	5.90	6.40
Cu/Ag-deposited fabric (50 bending cycles)	5.80	6.20
Cu/Ag-deposited fabric (20 peeling cycles)	5.60	5.90

Table 3: Electrostatic results of PP fabric samples under different conditions

PP fabric samples	Instantaneous electrostatic voltage (V)	Half-decay time (s)
Pristine fabric	930	16.87
Cu/Ag-deposited fabric	516	4.25
Cu/Ag-deposited fabric (50 bending cycles)	547	5.42
Cu/Ag-deposited fabric (20 peeling cycles)	631	6.21

4 Conclusions

Herein, we successfully prepared Cu–Ag thin films onto polypropylene fiber using the magnetron sputtering technique. Properties such as surface morphology, tensile, abrasion resistance, moisture regain, antibacterial, and electrostatic properties were systematically characterized. The following conclusions can be drawn.

SEM revealed that the Cu/Ag nanocomposite films were deposited onto the PP fiber surface evenly and densely. With respect to the tensile behavior, higher value of sputtering time, power, and pressure results in a higher tensile strength, irrespective of process variables considered and vice versa. Furthermore, the boxplots can be properly used to quantify the variation in yarn strength. In addition, compared with the pristine PP fiber, the abrasion resistance of Cu/Ag-deposited fibers was enhanced remarkably, whereas moisture regain decreased in different degrees depending upon treatment conditions. Importantly, the Cu/Ag-deposited PP fabric achieved excellent antibacterial and electrostatic properties, and an extraordinary durability was demonstrated in such fabric after undergoing vigorous external damages. It is promising that metallic nanocomposite polymer materials treated by the magnetron sputtering technique are a prerequisite for the next wave of multifunctional textiles.

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