

# Influence of oxygen plasma treatment on properties of polyester fabrics coated with copper films

Lingling Meng<sup>1, a</sup>, Qufu Wei<sup>2, b</sup> and Liyue Li<sup>3, c</sup>

<sup>1</sup> College of textile & clothing, Yancheng institute of technology, Yancheng 224051, PR China

<sup>2</sup> Key Laboratory of Science & Technology of Eco-Textile Ministry of Education, Southern Yangtze University, Wuxi 214122, PR China

<sup>3</sup> Department of art and design, Yancheng Textile Vocational Technology College, Yancheng, China

<sup>a</sup> mllwx022@163.com, <sup>b</sup> qufu\_wei@163.com, <sup>c</sup> 274806610@qq.com

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**Abstract:** A lab radio frequency (RF) magnetron sputter coating system was used to deposit the nanoscale copper (Cu) films onto the surface of polyester plain fabric at room temperature. In this study, the polyester fabrics were treated under different duration with low temperature plasma. The effect of oxygen plasma pretreatment on surface morphologies, antistatic properties, mechanical properties of the copper-coated polyester fabrics is investigated. The surface morphology of modified polyester fabrics was characterized by atomic force microscopy (AFM). The AFM observations revealed the obvious corrosion was formed on the fiber surface, the sputtered copper particles were hard to cluster and uniformly distributed on the fiber surface with longer duration of plasma treatment. The antistatic properties of the copper-coated polyester fabrics was greatly improved after the oxygen plasma treatment. The hydrophobic behavior of the coated samples was also significantly improved.

## Introduction

Plasma treatment technology has been widely used to modify the properties of textile, metal and polymer materials [1–3]. Many research studies were found in literature about plasma treatment changing the properties of textiles, such as antistatic properties, mechanical properties, water absorption. However, few researches are focused on properties of Cu-coated fabric after conducting plasma treatment.

In recent years, there are many deposition techniques for preparing copper films, such as chemical vapor deposition (CVD), physical vapor deposition (PVD), pulsed laser deposition and magnetron sputtering deposition [4–7]. Among these techniques, magnetron sputtering is considered a better technique for preparing functional nanofilms in an ideal vacuum conditions and the process is sputtering alone. Moreover, plasma treatment are usually considered environmentally friendly processes which can offer better adhesion between substrate and thin films.

In this study, copper films were deposited onto the surface of polyester plain fabric by the radio frequency (RF) magnetron sputtering at room temperature. The changes in surface morphology of the copper (Cu) coated polyester fabrics before and after treated by oxygen plasma were analyzed using atomic force microscopy (AFM), as well as their effects on the antistatic properties, wetting behavior of the fabrics was also characterized by contact angle measurements.

## Experimental section

### *Preparation of Materials*

100 % white polyester plain fabric of 75 g/m<sup>2</sup> with warp and weft yarn density of 16 tex × 16 tex was used. The fabric were first immersed in acetone solution for 30 min with ultrasonic washer to remove any oil or impurities that might be scattered on the fabric surface randomly during the manufacturing processes. Then they were washed twice with deionised water and dried at 50°C in a drying oven. The dried samples were cut into a size of 20 × 20 cm<sup>2</sup> for sputter coating.

### Plasma Treatment

Plasma treatments of polyester fabrics were carried out by low temperature plasma apparatus HD-1A manufactured by Zhongke Changtai Plasma Technology Co., Ltd (Changzhou,China), which is a radio-frequency (13.56 MHz) etching system. Oxygen was used as carrier gas at the flow rate of 0.3 LPM (litre per minute)with a power of 70W.Each sample was treated at 30Pa for 3min,6 min and 9min, respectively.

### Sputter Coating

A lab radio frequency (RF) magnetron sputter coating system (Shenyang, China) was used to deposit copper thin films in this study. A high purity copper (Cu) target (99.999%) with a diameter of 10 cm was mounted on the cathode, and the fabric sample was placed on an anode with a side facing the target. The distance between the target and the fabric sample was 60 mm. Prior to the Cu deposition process, the target was discharged in argon gas for about 5 min to remove impurities on its surface and sputtering chamber was pumped to a base pressure of  $5.0 \times 10^{-4}$  Pa before the introduction of high purity argon gas (99.99%) as bombardment gas to a certain pressure at the flow rate of 20sccm. To avoid the deformation of the fabric sample caused by high temperature, water-cooling was used to control the temperature of the fabric sample during the sputtering process. During the sputtering, the sample holder was rotating at a speed of 100 rpm to ensure copper particles uniformly deposited on the fabric sample. The sputtering pressure,sputtering power and coating time was set at 0.2 Pa,120 W,15 min respectively in this study.

## Surface Characterization

### AFM observation

The untreated and O<sub>2</sub>-plasma treated samples of copper-coated samples were analyzed using the atomic force microscopy [AFM; model: CSPM 4000 from Benyuan Co., Ltd (Guangzhou,China)] in contact mode. The surface scan of 5 $\mu\text{m} \times 5\mu\text{m}$  was carried out for the surface analysis and the scanning frequency was adjusted to 1.2 Hz. All samples were scanned at room temperature in the atmosphere.

### Antistatic property

The antistatic property of the fabric samples was measured with YG (B)342D type fabric induced static tester (Da-rong Textile Instrument, Wenzhou, China).The developed static charge and half decay time in the fabric samples were measured. Relative humidity of  $42 \pm 2\%$  and temperature of  $20 \pm 2^\circ\text{C}$ were maintained during the experiments. Sample size was kept at 60 $\times$ 60 mm<sup>2</sup>, they were fixed in a stainless steel device holder and charged for 30 s by applying corona discharge voltage of +10 kV at a discharge gap of 20 mm.The samples were rotated at 1500 rpm during the test. An average of five readings was taken for each sample.

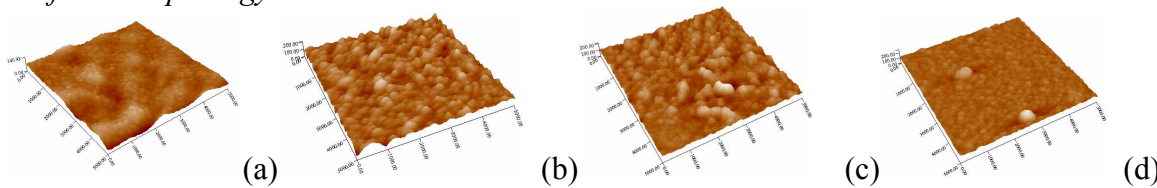
### Wettability measurements

The wettability of samples was determined by measuring the contact angle of Cu-coated fabrics.

The contact angle were conducted using a DCAT21 tester (Beijing,China). Distilled droplets were dispersed on the sample from a needle of microsyringe during the test.The picture of each droplet was taken 10 s after it set onto the sample.

## Results and analysis

### Surface Morphology



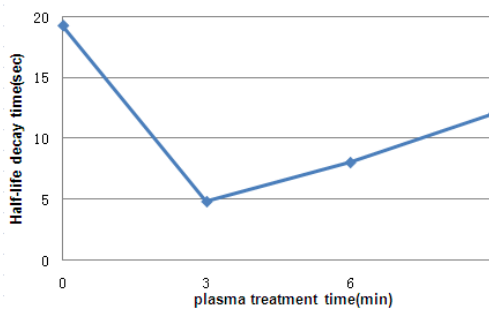
**Fig. 1: AFM images of copper-coated polyester fibers (a)untreated;(b) plasma treated for 3min;(c) plasma treated for 6min;(d)plasma treated for 9min**

The AFM images of  $5.0 \times 5.0 \mu\text{m}^2$  in Fig.1 reveal the surface structures of the copper-coated polyester fibers before and after  $\text{O}_2$ -plasma treatment, as presented in Fig.1. It is clearly observed that the untreated sample looks even with small copper nanoparticles formed on the fiber surface as shown in Fig.1(a). Contrarily, the surface of the treated fiber is obviously roughened and copper nanoparticles aggregate structure with large sizes can be seen on the fiber surface after the oxygen plasma treatment for 3min, as shown in Fig. 1(b) and Table 1. The phenomena may have some connection with the adhesion of the deposited copper film to the fibers. Fig. 1(c) and Table 1 show some larger and compacter clusters are formed on the fiber surface after plasma treatment for 6min. The larger cluster size is formed because of the frequent collision of the sputtered Cu particles. The various sizes of aggregate particles is caused by the uneven effect of the fiber surface with the use of plasma treatment, which is attributed to the preferential etching of the softer amorphous parts of the fiber during plasma treatment [8]. It also can be observed that the coated fibers formed the sputtered Cu nanoparticles become smoother and more uniform as presented in Fig. 1(d), indicating the improvement in the interfacial adhesion between copper film and the fiber.

$\text{O}_2$ -plasma treatment significantly alters the surface roughness of the copper-coated polyester fibers, as displayed in Table 1. The change of the surface morphology is also demonstrated by surface roughness analysis using the AFM software. The untreated fiber has an average roughness of 7.23 nm then the surface roughness of the coated fibers increased to 14.5nm after treated for 3min, as showed in Table 1, indicating the fiber surface surface is remarkably etched by plasma treatment which can be confirmed in Fig.1(b). The surface roughness is decreased with the increasing of plasma treatment duration. This demonstrated the effect of etching weakened as showed in Fig.1(c) and (d).

**Table 1 Surface roughness and particle size**

Treatment parameters	Gas: oxygen; power: 70W; working pressure: 30 Pa			
Duration of treatment (min)	0	3	6	9
Surface roughness (nm)	7.23	14.5	13.6	8.01
Particle size (nm)	85.1	106	110	104



**Fig. 2 Effect of treatment duration of  $\text{O}_2$ -plasma treatment on the antistatic property of coated polyester fabric.**

#### *Antistatic properties*

The elapsing time, termed as half-life decay time, is the time required for discharging half of the charge present in the fabric samples as accumulated during the charging-up process. Measurement of the half-life decay time is the common method to evaluate the antistatic property of fabrics. The shorter the half-decay time is, the better the antistatic property of the fabrics will be.

There are three parameters during the low temperature plasma treatment, i.e. discharge power, system pressure and treatment duration, which resulted in different effects on the half-life decay time of plasma treated polyester fabric. The effect of plasma treatment duration on the antistatic property of copper-coated polyester fabrics is displayed in Figure 2.

The half-life decay time of copper-coated fabric is 19.2 s. This higher value might be considered to be relatively poor antistatic property. The half-life decay time for the coated fabric after  $\text{O}_2$ -plasma treatment is obviously lower than untreated sample. The time for the surface charge to reach half of the initial surface charge is 4.8 s, indicating excellent antistatic properties as plasma treatment for 3min. It may be due to the influence of more cracks on the surface of thicker coatings, which block the discharge of surface assembled static charges, as confirmed by Fig. 1(c). The half-life decay time increases slowly to 8 s when treatment duration increased to 6min. After which, it reaches 12s with plasma treatment time for 9min. With the increase of treatment duration, the half-life decay time is decreased compare with untreated accordingly which means that the

antistatic property of samples is improved with a longer treatment duration. It can be explained that more interaction would occur between the fiber surface and the plasma species. Therefore, the alteration on the fiber surface would become more significant[9].

#### *Wetting behavior*

The surface contact angles of polyester fabrics before and after plasma treatment are illustrated in Figs. 3(a)-(d). The contact angles are caught as the first contact between water droplet and the fiber. Fig. 3(a) displays the contact angle of original polyester fiber that is  $93^\circ$  because of its hydrophobic nature. The contact angle of polyester fiber is dropped to  $60^\circ$  when copper particles cover with the surface of fiber, as presented in Fig. 3(b), indicating the wetting behavior of copper-coated fabrics is improved. The copper particles deposited on the surface of fabrics is likely to form hydrogen bond with water when they contact water. Therefore, the water absorption of polyester fabrics was significantly improved by copper coating on the surface.

However, there is a significant change in contact angle when the copper-coated polyester fiber was subjected to 3min  $O_2$ -plasma treatment as shown in Fig. 3(c). It is attributed to roughening of the fiber surface by plasma treatment [10], as shown in Table 1. The increase of surface roughness from 7.23 to 14.5 nm leads to an obvious increase up to  $89^\circ$  in the contact angle, as indicated in Table 1. It can be seen that there is a clear increase in the surface contact angle from  $113^\circ$  to  $123^\circ$  when the exposure duration is 6min and 9min as shown in Fig. 3(d) and (e) respectively. There is about 33–51% increase when compared to the untreated polyester fiber coated with copper films, which might be caused by the formation of hydrophobic groups on the fiber surface. The coated samples show a clear hydrophobicity with a longer treatment duration.



**Fig. 3 Contact angles of copper-coated polyester fabrics: (a)original;(b) untreated; (c) plasma treated for 3min; (d) plasma treated for 6min; (e) plasma treated for 9min.**

## Conclusion

Oxygen plasma treatment has some effects on the properties of polyester fabric deposited with copper films. The surface morphologies of the copper(Cu) coated polyester fabrics before and after treated by oxygen plasma have an obvious change. Analysis in morphology and tensile testing indicated that the uniform deposition of copper films on the fabrics could be achieved by plasma treatment with higher mechanical properties. The contact angle of coated fabrics with plasma treated is found to be improved with longer treatment duration.

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