Effect of substrate structures on the morphology and interfacial bonding properties of copper films sputtered on polyester fabrics

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Abstract

Purpose – The purpose of this paper is to discuss polyester fabric structures in terms of the surface morphology, crystal structure of copper films and interfacial bonding properties between polyester fabrics and copper films.

Design/methodology/approach – Nanoscale copper (Cu) thin films were deposited onto the surface of polyester fabrics with different structures by the radio frequency magnetron sputtering technique at room temperature.

Findings – Copper films uniformly deposited on the surface of the polyester nonwovens and nanofiber membranes have larger average particle diameters and surface roughness, and higher crystallinity.

Originality/value – Theoretical value: the effects of polyester substrate structures on the morphology and interfacial bonding properties of Cu thin films have rarely been reported.

Keywords Copper film, RF magnetron sputtering, Morphology, Interfacial bonding

Paper type Research paper

1. Introduction

Polyester fabric has high strength, good elasticity, and physical and mechanical properties, and is used in various fields because of its unique performance, low cost and processing advantage. Nanoscale copper film is a new functional material with a small particle size and a unique surface effect, which has good conductivity of electricity and ultraviolet radiation properties, and has broad application prospects in the chemical industry, textile, medicine and other industries.

Nanoscale copper films are prepared by magnetron sputtering, plasma-assisted chemical vapor deposition, physical vapor deposition, sol-gel process and pulsed laser deposition (Kirkpatrick, 1973; Last and Thouless, 1971; Shante and Kirkpatrick, 1971). Magnetron sputtering is considered a better technique for preparing functional nanofilms under vacuum conditions and the process exclusively involves sputtering, which results in better adhesion between substrates and thin films (Bula *et al.*, 2006; Scholz *et al.*, 2005; Banerjee *et al.*, 2006; Meille, 2006). Polyester fabrics sputtered on nanoscale copper films are

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considered for anti-static, UV-protective and electromagnetic shielding features in different applications for the defense, civil textile and electronic industries.

In recent years (FAN Donghua, 2008; Liu *et al.*, 2001), a large number of studies have been reported on the preparation, microstructures and properties of nanoscale copper films sputtered on polyester textile materials (Muppidi *et al.*, 2005; Wang *et al.*, 2007; Lim *et al.*, 2003; Savaloni *et al.*, 2009). Wei *et al.* (2008) reported the use of sputter coating of copper (Cu) to deposit functional nanostructures on the surfaces of polypropylene spun-bonded nonwovens; the surface morphology, pore structures and electrical properties of the functionalized materials were examined. Joanne Yip *et al.* (2009) found that a layer of fine silver particles distributed evenly on the fabric surface with uniform grains and there was no obvious structural damage to the fabric after sputtering and the sputter-coated fabric had a higher value in reflectance and lightness than metallic powder printed fabric, but was lower than that of metallic foil-laminated fabric. Huang *et al.* (2014) studied nanoscale copper films deposited onto the surface of polyester fabrics using a radio frequency (RF) magnetron sputter coating system at room temperature and the conductive properties favoring the electromagnetic shielding effectiveness of Cu deposited on polyester nonwovens and nanofiber substrates were also investigated.

However, only little attention has been paid to the effects of polyester substrate structures on the morphology and properties of nanoscale copper films. Therefore, it is important to further investigate the interfacial bonding properties of nanoscale copper films and substrates for practical applications. In this study, nanoscale copper films were deposited onto the surface of the pure polyester woven fabrics, knitted fabrics, nonwovens and nanofiber membrane by the RF magnetron sputtering method. The purpose of this study was to discuss polyester fabric structures in terms of the surface morphology and crystal structure of copper films. Field emission scanning electron microscopy (FE-SEM), atomic force microscopy (AFM) and video microscope (VM) were used to characterize the structures and surface morphology of the Cu-coated polyester substrates. The crystalline structure of copper films was examined by an X-ray diffractometer (XRD). The interfacial bonding strength between copper layers and substrates is an important factor determining the adhesion level and interfacial durability. In this case, the interfacial bonding properties between polyester fabrics and copper thin films were also analyzed.

2. Experimental

2.1 Preparation of materials

In total, 100 percent white polyester fabrics with different structures were prepared, including polyester plain weave fabric ($260 \times 140/10$ cm), knitted fabric (201 g/m^2), nonwoven fabric (100 g/m^2) and nanofiber membranes (15 g/m^2).

The samples were first immersed in acetone solution for 30 min with an ultrasonic washer to remove any oil or impurities that might be scattered on the fabrics' surface randomly during the manufacturing process. Then, they were washed twice with deionized water and dried at 50 °C in a drying oven.

2.2 Sputter coating

A laboratory RF magnetron sputter coating system JZCK-420B (Shenyang, China) was used to deposit copper thin films. A high-purity copper (Cu) target (99.999 percent) with a diameter of 10 cm was mounted on the cathode and the fabric sample was placed on an anode with one side facing the target. The distance between the target and the fabric sample was 60 mm. Argon (99.99 percent) was used as the bombardment gas. Prior to the deposition, the target was discharged in argon gas for about 5 min to remove impurities on its surface and the sputtering chamber was pumped to achieve a base pressure of 5.0×10^{-4} Pa. To avoid 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. Meanwhile, the sample holder was rotating at a speed of 100 rpm to ensure that copper particles uniformly deposited on the fabric sample. The sputtering pressure was set at 0.2 Pa with a power of 120 W; the coating time was set to 60 min on the basis of a previous experiment in this study.

2.3 Characterization

The structures of substrates were observed by a VM (JVC, Japan) and the surface morphologies of copper films were analyzed using FE-SEM (FE-SEM; Hitachi S-4800 (Japan)) at a magnification of 50,000 × and AFM (AFM; Model: CSPM 4,000 from Benyuan Co., Ltd (Guangzhou, China)) in contact mode. A surface scan of 5 × 5 μm was carried out for the surface analysis and the scanning frequency was adjusted to 1.2 Hz. All samples were scanned at room temperature.

The crystalline structure of copper films was examined by an XRD (Model: Max-2500 from Rigaku Co. Ltd.) with Cu-K radiation.

2.4 Interfacial bonding properties

The observation of interfacial bonding between copper films and the polyester fibers was performed on the cross-section of the fibers by the FE-SEM (Hitachi S-4800, Japan). The influence of the structure of polyester substrates on the adhesion of the copper coatings to the fabric sample was analyzed by FE-SEM images. In order to further compare with the interfacial bonding strength, the performance of peel-off and wear-resisting testing were also analyzed.

The peel-off test was conducted by a universal materials testing machine (Zwick BZ2.5/TNIS, Germany) to examine the interfacial adhesion of the coated layer to the fabric. The test speed was set to 200 mm/min in this study. The initial distance was 10 mm and a 3M600 test adhesive tape was used. The test samples were cut into 7 × 2.5 cm^2 sizes for the peel-off test. The samples were pressed with a load of 400 g for 12 h before the peel-off test. Each test was carried out three times and the average values were used. The above methods of testing were performed at 20 ± 2 °C and 65 ± 2 percent relative humidity.

3. Results and discussion

3.1 Surface morphology

As can be seen from Figure 1(a), the woven fabric had two group of yarns, warp and weft yarns which were interlaced, but its unique interwoven structure made the fiber closely packed. Polyester nonwoven fabric and nanofiber membrane showed similar fibrous structures, in which fibers were randomly oriented, but the nanofiber membrane had much finer fibers compared to the conventional nonwoven fabric, as indicated in Figure 1(c) and (d). Compared to woven fabric, the polyester knitted fabric showed a fluffy structure with many coiled yarns. In order to investigate the effect of substrate structures on the surface morphology of thin films, Cu thin films were deposited on different polyester substrates under the same deposition conditions.

Figure 2 shows the FE-SEM images of the surface morphologies of copper thin films deposited on the polyester substrates with different structures. The particles sizes of Figure 2(b) are the largest and agglomerated obviously which is mainly related to the formation process of nanoscale copper films and the nature of substrates. The morphologies of different structures polyester fabrics are shown in Figure 1; the surfaces of knitted fabrics with apparent ups and downs have some impacts on the copper particle size. According to the kinetics principles of magnetron sputtering, sputtered copper particles were first deposited the substrate defects. The copper particles were first deposited in the lower circle arcs position, and then gradually gathered into a nucleus until the formation of a three-dimensional island structure, resulting in

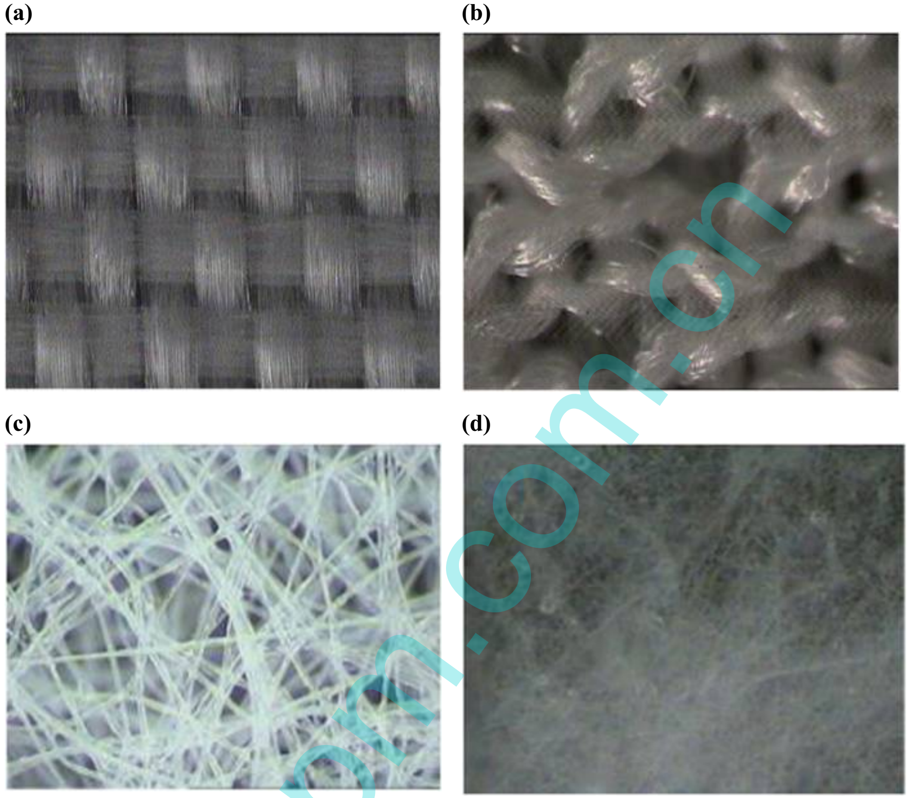
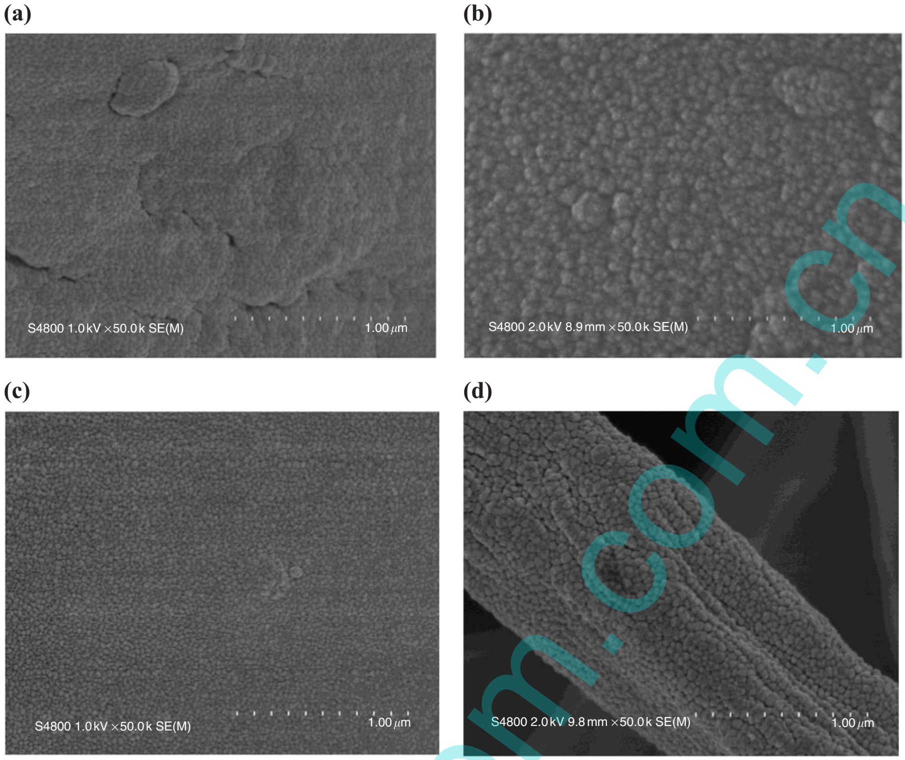


Figure 1.
The JVC images of different structures of polyester substrates

Notes: (a) Woven fabric; (b) knitted fabric; (c) nonwoven fabric; and (d) nanofiber membranes

the aggregation of Cu particles and the formation of larger copper particle sizes. As can be seen from Figure 1(c), the nonwoven fabric has good continuity. Therefore, nanoscale copper films emerged in a short time and then copper particles would bombard the grains on the surface of substrates. Figure 1(d) shows that the surface of the nanofiber is uneven because of the volatile solvent in the solution during electrostatic spinning, which may increase its specific surface area, and improve binding strength between nanofibers and copper films. However, particle size and root mean square (RMS) roughness of the nanocopper films sputtered on the polyester fibers need to be further analyzed.

The surface RMS roughness and particle sizes of Cu films deposited on the surface of polyester fibers are shown in Table I. The copper particle sizes sputtered on different polyester substrates measured about 121.9, 114.5, 132.3 and 146.4 nm, respectively, by means of AFM as shown in Figure 3. The sputtered Cu films on the surface of polyester woven and knitted fabric had relatively small particle diameters and RMS roughness, forming a small island on the more flat fiber surface with the same deposition as shown in Figure 3(a)-(b); nevertheless, Figure 3(c)-(d) indicate that nanoscale Cu films deposited on the surface of nowoven and nanofiber membrane have an uneven distribution of particles, presenting island distribution on the ups and downs of the surface of the fibers and larger RMS roughness. Besides, the nanoscale Cu films on the surface of the nanofiber membranes have the largest particle size and RMS roughness as shown in Table I, revealing the most obvious irregularity.



Notes: (a) Woven fabric; (b) knitted fabric; (c) nonwoven fabric; and (d) nanofiber membrane

Figure 2. FE-SEM images of nanoscale copper films deposited on polyester substrates

3.2 Crystalline structure characterization

Figure 4 shows the X-ray diffraction patterns of Cu films deposited on polyester fabrics with various structures under the same sputtering conditions. These samples have a strong preferred orientation toward the Cu(111) plane crystal diffraction peak. It was found that the crystallization of copper films deposited on the polyester woven and knitted fabrics was not ideal because of a very weak Cu(111) plane diffraction peak. On the contrary, the Cu(111) crystal plane diffraction peak was obviously stronger when copper films deposited on the surface of nonwoven fabric and nanofiber membrane, and Cu(200) and Cu(220) crystal planes appeared on the obvious diffraction peak. In general, the larger the size of particles, the better the crystallinity. Figure 1 presents nanofiber membrane for a relatively thin fiber diameter and a smaller specific surface area. Figure 3 shows that nanoscale Cu films deposited on the surface of nonwoven fabric and nanofiber membranes have a larger

Polyester substrates	Particle size (nm)	RMS roughness (nm)
Woven fabric	121.9	14.4
Knitted fabric	114.5	9.41
Nonwoven fabric	132.3	13.1
Nanofiber membrane	146.4	15.6

Table I. Particle size and RMS roughness of the nanocopper films sputtered on the polyester fibers

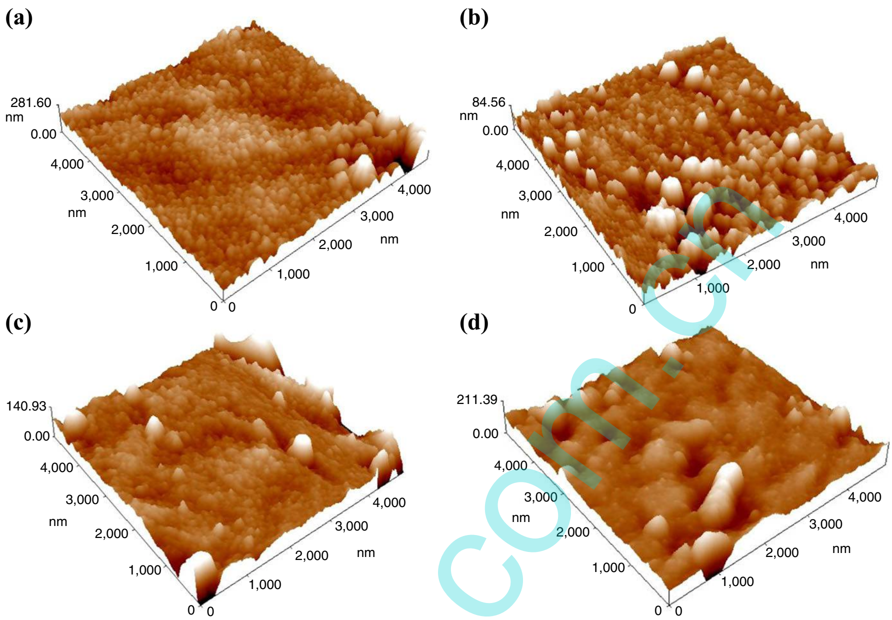


Figure 3. AFM images of nanoscale copper films deposited on polyester substrates

Notes: (a) Woven fabric; (b) knitted fabric; (c) nonwoven fabric; and (d) nanofiber membrane

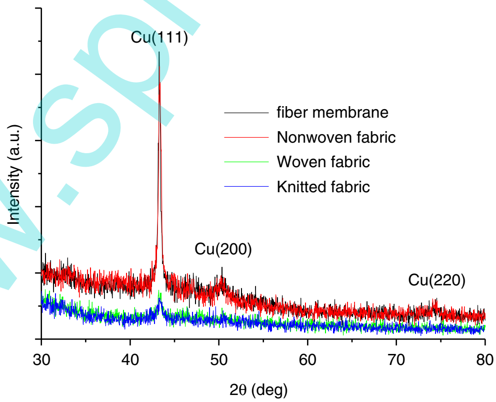


Figure 4. XRD patterns of copper-coated polyester fabric with different structures

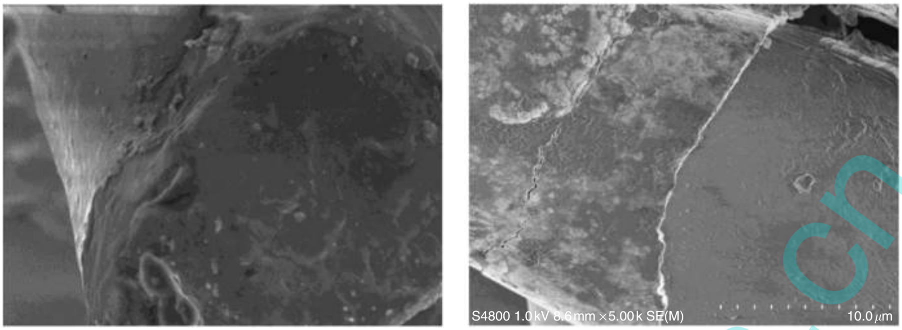
average particle size, which is further illustrated by the fact that the Cu(111) plane has higher crystallinity when copper films are deposited on the surface nonwoven fabric and nanofiber membrane.

3.3 *Interfacial bonding properties*

The FE-SEM observations reveal the interfacial bonding between the copper coating and the polyester substrates, as indicated in Figure 5.

The adhesion of the sputtered Cu nanoclusters to the polyester substrates was also examined by a peel-off test. The results of the test are listed in Table II.

(a) (b)



Notes: (a) Knitted fabric; (b) nonwoven fabric

Figure 5. FE-SEM images of the cross-sections of samples with a nanocopper film after 60 min of coating

Table II. Breaking strength between copper film and substrates

Sample	Peel strength (*N*)
PET woven + 60 min sputtering	4.87
PET knitted fabrics + 60 min sputtering	4.65
PET nonwoven + 60 min sputtering	4.95
PET fiber membranes + 60 min sputtering	6.25

Figure 5 shows that a cross-section of nanoscale copper films sputtered on the surface of polyester knitted fabric and nonwoven fabric under the same deposition condition. It can be seen from Figure 5(a) that nanoscale copper films were densely completely covered with the fiber surface by 60 min sputtering on the surface of the knitted fabric. The possible reason for the phenomenon could be the good adhesion of the copper coatings to the fibers. Besides, the image reveals the bonding of the coating layer to the fibers, which shows that the sputtered copper particles have sufficient energy to penetrate into the fiber (Wei *et al.*, 2008). Figures 3 and 4 also illustrate that copper films have an uneven distribution with a smaller average particle size and surface roughness, and a weaker diffraction peak on the surface of polyester woven and knitted fabric. It can be seen from Table II that the average peel-off strength of the sputtered Cu nanoclusters on the polyester knitted fabric is about 4.65 N when the sputtering time is 60 min. Meanwhile, Figure 5(b) shows that dense and uniform nanoscale copper particles covered the surface of polyester nonwoven fabric after 60 min of sputtering, and formed a continuous film. Based on the experimental results, the copper layer on the surface roughness and interface bonding strength have an obvious corresponding relation. Figure 3 shows that nanoscale copper films on the surface of polyester nonwovens and nanofiber membranes have larger surface roughness; thus, the sputtered Cu particles have better coverage of copper layers on the fabric and the peel-off strength further improved to 4.95 and 6.25 N as shown in Table II. According to the above analysis, the copper layer on the rough surface can effectively enhance the physical interlocking between the copper films and substrates, which improves the bonding strength between copper films and substrates.

4. Conclusions

This paper found that fabric structures have an apparent influence on the morphology and interfacial bonding properties of copper films sputtered on different structures of polyester substrates using the RF magnetron sputter coating method. The results showed the nonwoven and nanofiber membrane polyester substrates with a sputtered Cu thin film exhibited larger average particle diameters and surface roughness. The larger the size of copper particles deposited on the surface of nonwoven fabric and nanofiber membrane, the higher the crystallinity of the Cu(111) plane. The copper layer on the rough surface can effectively enhance the physical interlocking between thin films and substrates, which significantly improved the bonding strength between copper films and nanofibers under the same sputtering process.

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