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# Characterization of nonwoven material functionalized by sputter coating of copper

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## Abstract

Nonwoven materials consisting of polymer fibers provide an excellent platform for the integration of functional structures to improve the performance of the materials for a variety of applications. In this study, the sputter coating of copper (Cu) was used to deposit functional nanostructures on the surfaces of polypropylene (PP) spunbonded nonwovens. The surface morphology, pore structures and electrical properties of the functionalized materials were examined using Atomic force microscope (AFM), Environmental Scanning Electron Microscopy (ESEM), capillary flow porometer and electrical resistance measurement. The observations by AFM revealed the formation of the functional nanostructures on the PP fibers. The ESEM examination confirmed the introduction of functional Cu nanostructures built on the PP fibers. It was also found that the pore structures and electrical properties of the material were also altered by the sputter coating of copper. The interfacial bonding between the sputtered clusters and the PP fibers was also observed in SEM and AFM.

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## 1. Introduction

Nonwoven materials have been increasingly used for a variety of applications in the world [1]. For these increasing applications it is desirable to produce such nonwoven materials with well-defined surface properties. Nonwovens with specific surface properties are also of interest in many technical applications of the materials as the surface features affect adsorption, abrasion, adhesion, biocompatibility and other properties of the materials. However, the surfaces of polymer fibers are often not ideal for a particular application. The inert nature of many polymer fibers has prevented the expanding applications of nonwoven materials. Various techniques have been developed to modify the surface properties of nonwoven materials [2–4]. In recent years, physical vapor deposition (PVD) [5] has opened up new possibilities in the modification and functionalization of textile materials.

PVD is a process by which a thin film of material is deposited on a substrate. The most promising techniques in PVD technology is sputtering[6], which has been widely used to modify various materials in many industries.

The ability to deposit well-controlled coatings on polymer fibers would expand the applications of polymer fibers, based on changes to both the physical and chemical properties of polymer fibers. In this study, nonwoven material was functionalized using the sputter coating of copper (Cu). The effects of sputter coating on the surface morphology and properties of the material were characterized by Atomic force microscopy (AFM), Environmental scanning electron microscopy (ESEM), Scanning electron microscopy (SEM), Capillary flow porometer and electrical resistance measurement.

## 2. Experimental

### 2.1. Materials

The material used in this study was polypropylene (PP) spunbonded nonwoven with a mass per area unit of 136±4 $g/m^2$.

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The samples of the nonwoven material were washed with ethanol and rinsed with distilled water before the sputter coatings. After washing, the samples were dried in an oven at 40 °C.

A magnetron sputter coating system JZCK-420B was used to deposit a nanolayer on the materials. The metallic coating of Cu was performed using a high-purity Cu target (99.999%). The target of Cu was fixed on the cathode, and the nonwoven sample was placed on the anode with a side facing the target. The power source used for the sputter coating was DC (direct current) power in the range between 5 W and 80 W. The sputtering pressure was adjusted to about 0.9 Pa using the bombardment gas of argon (99.999%). The coating thickness was set at 10 nm, 50 nm and 100 nm respectively, which was monitored by a coating thickness detector (FTM-V) fixed in the sputtering chamber [7].

## 2.2. Characterisation

### 2.2.1. AFM

The surface morphology was examined using atomic force microscopy (AFM). AFM is a very high-resolution imaging tool and has become one of the foremost tools for imaging, measuring and manipulating matter at the nanoscale. The AFM consists of a microscale cantilever with a sharp tip at its end that is used to scan the specimen surface for obtaining high-resolution images [8]. The AFM used in this study was a CSPM 4000 provided by Benyuan Co., LTD. Scanning was carried out in contact mode AFM with a silicon cantilever and all images were obtained at ambient conditions.

### 2.2.2. EDX in ESEM

The surface chemical compositions were analyzed by energy-dispersive X-ray (EDX). The EDX analysis was performed on an environmental scanning electron microscopy (ESEM) Philips XL30 integrated with a Phoenix energy-dispersive X-ray (EDX) detector [9] in this study. The EDX analysis was performed at an accelerating voltage of 20 kV with accounting time of 100 s.

### 2.2.3. Pore size

The effect of the sputter coating on the porous structures of the material was characterized using a capillary flow porometer. The porometer used was CFP-1100A made by PMI Inc.

### 2.2.4. Electrical properties

The electrical property of the material was examined by resistivity analysis. The resistivity was measured using a collinear four-probe array. The apparatus used was SX1934 made by Baishen Technologies. In order to minimize the deviations brought by the unevenness of textile surface, the resistivity of each sample was measured three times, and the average values were used.

### 2.2.5. Interfacial observation

The interfacial bonding between the sputtered clusters and the PP fibers was observed in SEM and AFM. The SEM used was QUANTA-200. Scanning was performed at 10 kV with a magnification of 5000. The observation of interfacial bonding between the sputtered clusters and the PP fibers was performed on the cross-section of the fibers. The samples were cut in cross-section direction using a sharp blade to avoid the compression

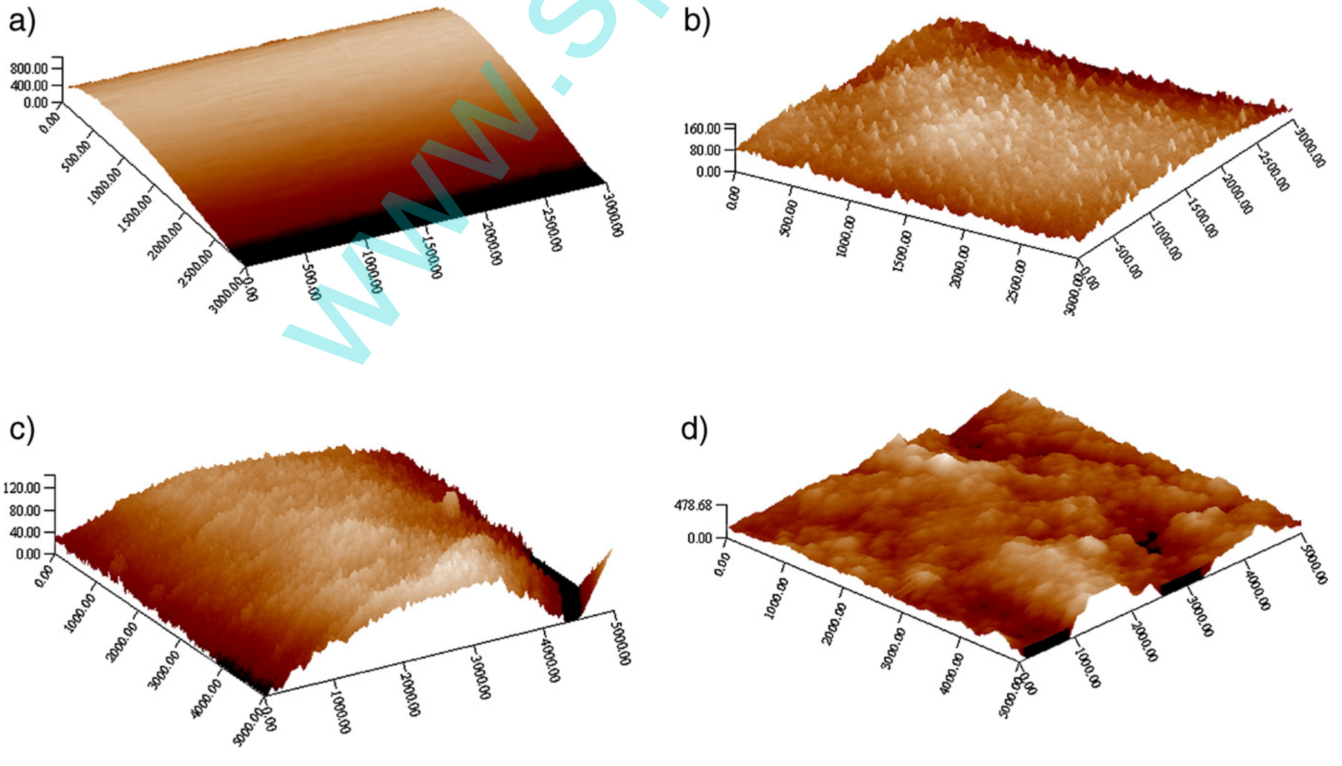

Fig. 1. Surface morphology of the PP fiber in AFM: (a) original fiber; (b) 10 nm coating; (c) 50 nm coating; (d) 100 nm coating.

of the fibers. The AFM observations were performed according to the settings detailed in Section 2.2.1.

## 3. Results and discussion

### 3.1. Surface morphology

The surface morphology of the uncoated fiber is presented in Fig. 1a. The AFM image clearly shows the relatively smooth surface of the PP fiber. The sputter coating of Cu significantly alters the surface characteristics of the PP fiber, as indicated in Fig. 1b. The image clearly shows the formation of island-like nanostructures on the fiber surface, as the thickness of the Cu sputtered at 60 W reaches 10 nm. The islands are the Cu clusters deposited from the Cu target onto the fiber surface. The Cu clusters have variable sizes ranging from few nanometers to over 10 nm. The growth of the Cu clusters formed on the PP fiber is observed as the coating thickness is increased to 50 nm. The AFM image in Fig. 1c reveals the structures of the 50 nm Cu coating on the PP fiber. The Cu clusters cover up the surface of the PP fiber and the sizes of the Cu clusters increase to about 25 nm, as analyzed using the AFM software. The increase in the cluster size is attributed to the collision of the sputtered Cu particles, leading to the formation of larger clusters. The sizes of the clusters are further increased to about 32 nm as the coating thickness is increased to 100 nm. The AFM image in Fig. 1d clearly shows the formation of larger and compacter clusters on the fiber surface.

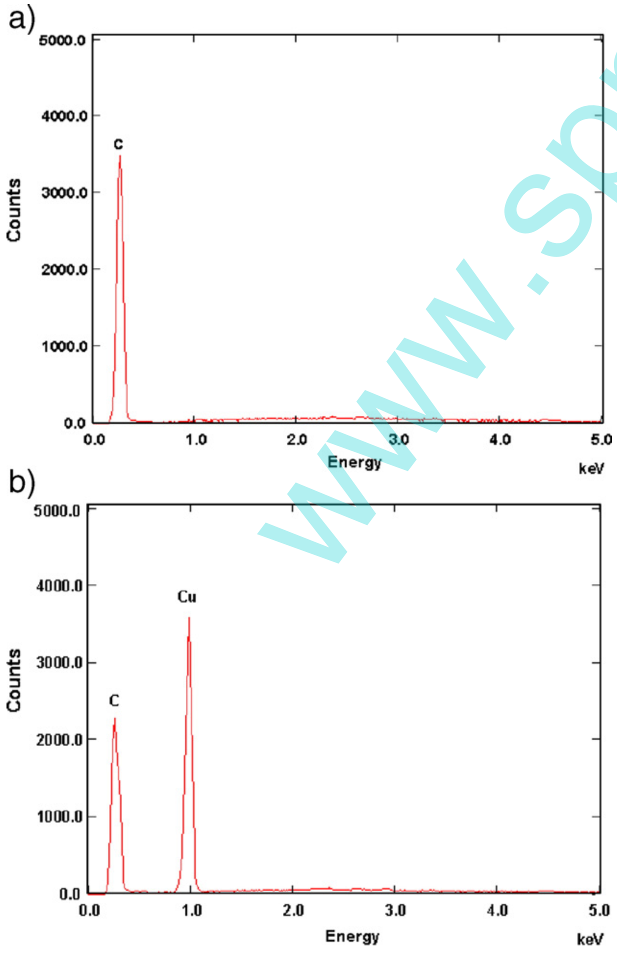

Fig. 2. EDX spectra of the materials: (a) original; (b) 10 nm coated.

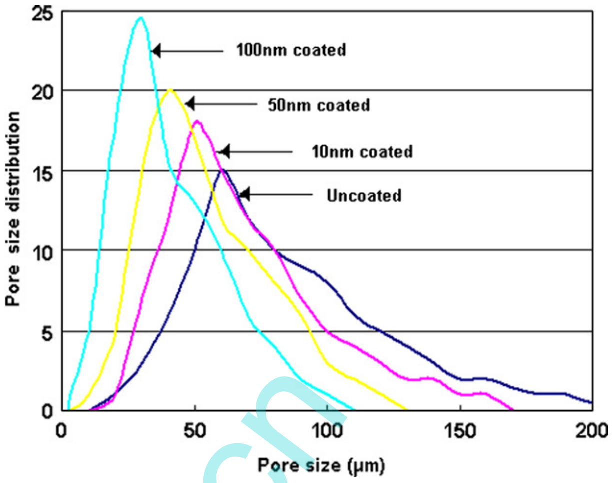

Fig. 3. Pore structures of the nonwoven material.

### 3.2. EDX analysis

The functionalization of the fiber surfaces by sputter coating is confirmed by EDX analyses. The EDX spectrum in Fig. 2a clearly shows the component of the nonwoven before the sputter coating of Cu. It can be seen that the fiber dominantly consists of C before the sputter coating. The composition of hydrogen (H) in the material is too light to be detected in this EDX analysis. A significant amount of Cu on the fiber surface after the sputter coating with a thickness of 10 nm is revealed Fig. 2b. The EDX spectrum confirms the formation of the functional coating on the fiber surface.

### 3.3. Pore size

The randomly distributed fibers in the nonwoven web form numerous pores with various shapes [10]. The effect of the sputter coating on the pore structure of the material is shown in Fig. 3. The sizes of the pores in the original material cover a wide range from about 10 microns to 200 μm. The average size of the pores is about 65.4 μm. The curves clearly indicate the shift of the pore sizes towards the left side as the coating thickness is increased. The average size of the pores drops to about 57.8 μm as the coating thickness is 10 nm. The average size of the pores is further reduced to about 49.8 microns as the coating thickness is 50 nm. The increase in coating thickness to 100 nm leads to the decrease of the average pore size to about

Table 1
Resistivity values

| Sample | Original | 10 nm coating | 50 nm coating | 100 nm coating |
|---|---|---|---|---|
| Resistivity (Ωcm) | Out of range (over $10^6$) | 128.4±10.5 | 0.7±0.2 | 0.025±0.01 |

43.6 μm. It is also found that the pore distribution becomes narrower as the coating thickness is increased.

### 3.4. Electrical property

The results of resistance measurements of the samples are listed in Table 1. It clearly shows the significant decrease of all samples in surface resistivity after sputter coating. The surface resistivity of original material before sputter coating is over $10^6$ Ωcm, indicating electrical isolation property. After the coating with a thickness of 10 nm, the surface resistivity drops from over $10^6$ Ωcm to about 128 Ωcm, indicating a considerable decrease in surface resistivity. The increase in sputtering thickness leads to the further decrease in the surface resistivity of the material, as presented in Table 1. It can also be seen from Table 1 that the deviation of the surface resistivity is obviously reduced as the coating thickness is increased. This is attributed to evener distribution of Cu clusters on the fibers as the coating becomes thicker, revealed in Fig. 1.

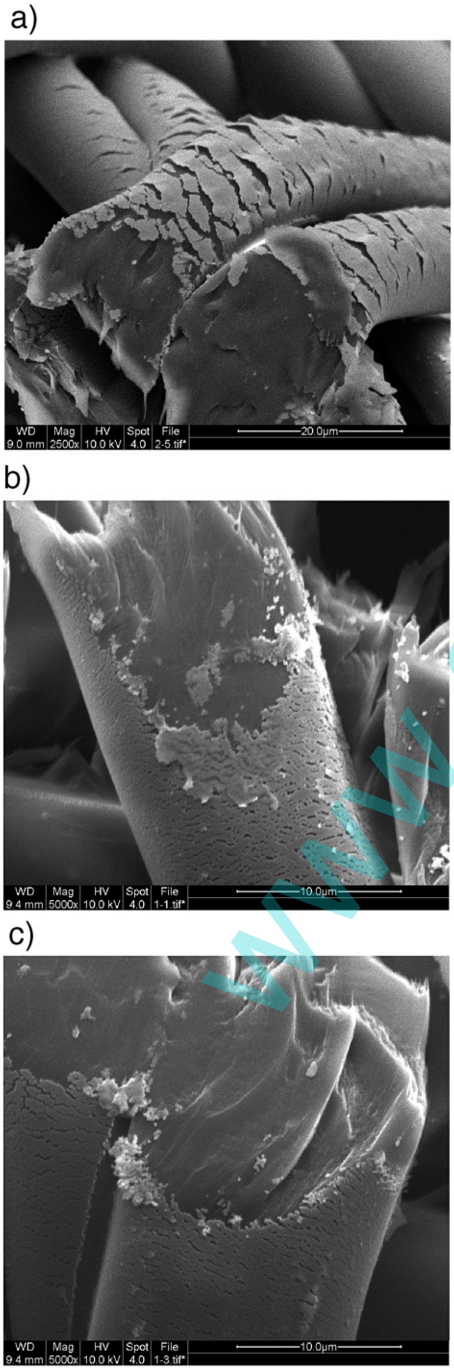

Fig. 4. Interfacial bonding observed in SEM: (a) 5 W sputtering; (b) 30 W sputtering; (c) 60 W sputtering.

### 3.5. Interfacial observation

The bonding between the coated clusters and fibers is one of the most important factors influencing the duration of the coatings. The SEM observations reveal the effects of sputtering power on the interfacial bonding between the coated clusters and fibers, as presented in Fig. 4. The coating clusters deposited on the fiber surface forms floating layer with cracks when the sputtering power is only 5 W, as shown in Fig. 4a, indicating the poor bonding between the Cu clusters and fibers. The image clearly reveals that the some part of the coating layer is separated from the fiber surface. It is believed that lower sputtering power does not provide sufficient energy for the sputtered particles to bond with the fiber. The bonding between the clusters and the fibers is improved as the sputtering power is increased to 30 W, as revealed in Fig. 4b. The image shows no floating layer on the fiber surface. It can be seen that the coating layer becomes compact with some smaller cracks, which may form during cutting of the samples. The bonding is further improved as the sputtering power is increased to 60 W, as shown in Fig. 4c. The image shows the compact coating with less cracks. The improvement in interfacial bonding is attributed to the increased energy provided for the sputtered Cu particles to hit the fiber surfaces.

The effect of the coating thickness on the interfacial bonding can also be seen in Fig. 1b–d. The sputtered particles form a thin layer bonded with the fiber as the coating thickness is less than 50 nm. The coating layer show some obvious crack structures on the fiber surface, indicating poor adhesion with the fiber, as presented in Fig. 1d. This is attributed to the increased internal tension of the coating layer as the coating thickness is increased.

## 4. Conclusion

This study investigated the functionalization of nonwoven materials by the sputter coating of copper. The surface conductivity of the materials was significantly improved. The sputter coating reduced the pore sizes of the nonwoven materials to some extent, but the porous characteristics of the materials remained unchanged. Metallic functionalization of nonwoven materials by sputtering coating provides new approach to improving the surface properties of the polymer fiber materials. The conductive surface is often required for such applications as anti-static, conductive shields, packing and protective materials and the metallic coating by sputtering provides an environmental friendly technique to the surface modification of textile materials.

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