Bingyao Deng[a, b], Qufu Wei[a], Weidong Gao[a], Xiong Yan[b]

[a] Key Laboratory of Eco-textiles, Ministry of Education, Jiangnan University, WuXi 214122, P.R.China

[b] DongHua University, Shanghai 200051, P.R.China
E-mail: qufu_wei@163.com

Surface Functionalization of Nonwovens by Aluminum Sputter Coating

Abstract

Nonwoven materials have been widely used in many industries. The surface properties of nonwovens are of importance in the applications presented here. In this study, magnetron sputter coating was used to deposit functional metal aluminium (Al) nanostructures onto nonwoven material. Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Environmental Scanning Electron Microscopy (ESEM) were employed to study the structure, topography and chemical composition of the material, respectively. The AFM results revealed the formation of functional nanostructures on the fiber surfaces. A full Energy Dispersive X-ray analysis (EDX) mounted on the ESEM was also used to detect the elemental composition of the functional fibers. EDX examination showed the change in the chemical compositions of the fiber surfaces. It was also found that the electrical resistance of aluminium (Al) sputtered nonwoven material was significantly decreased.

Key words: *nonwoven, surface, sputtering, aluminium, AFM, EDX, ESEM.*

Introduction

The nonwoven industry is one of the fastest growing in the world. Nonwovens have been increasingly used in many industries for a wide range of applications ranging from wipes to biomaterials [1].

For a variety of applications it is desirable to produce such nonwoven materials with required surface properties. Nonwoven materials with specific surface properties are also of importance in many technical applications as the surface features affect friction, wettability, electro-optical property, adsorption and adhesion of the fibres. However, the surfaces of polymer fibres are often not ideal for a particular application. Various techniques, such as physical vapour deposition (PVD), electroless deposition, have been developed to modify the surface properties of polymer fibres. In all of these, sputter coating [2 - 5] has proven to be one of the most promising techniques to functionailze textile materials.

The ability to deposit well-defined layers on nonwoven materials would expand the applications of nonwovens, based on changes to both the physical and chemical properties of nonwoven materials. In this study, polyethylene terephthalate (PET) nonwoven was functionalised with metal Al materials using the sputter coating technique. Scanning electron microscopy (SEM), Atomic Force Microscopy (AFM) and Environmental Scanning Electron Microscopy (ESEM) were employed to study the structure, topography and chemical composition of the fibres, respectively.

Figure 1. *Untreated PET nonwoven - SEM image.*

Experimental

Materials preparation

The substrates used in this study were commercial PET spunbonded nonwovens with a mass of 150 g/m^2. Before the sputter coatings, the material was washed with ethanol and distilled water. After washing, the material was dried in an oven at 40 °C for 24 hours.

A magnetron sputter coating system (JZCK-420B) made by Shenyang Juzhi Co, Ltd. was used to deposit a nanolayer on the nonwoven substrate. A high-purity Al target was mounted on the cathode, and argon was used as the bombardment gas. The sputtering pressure was set at

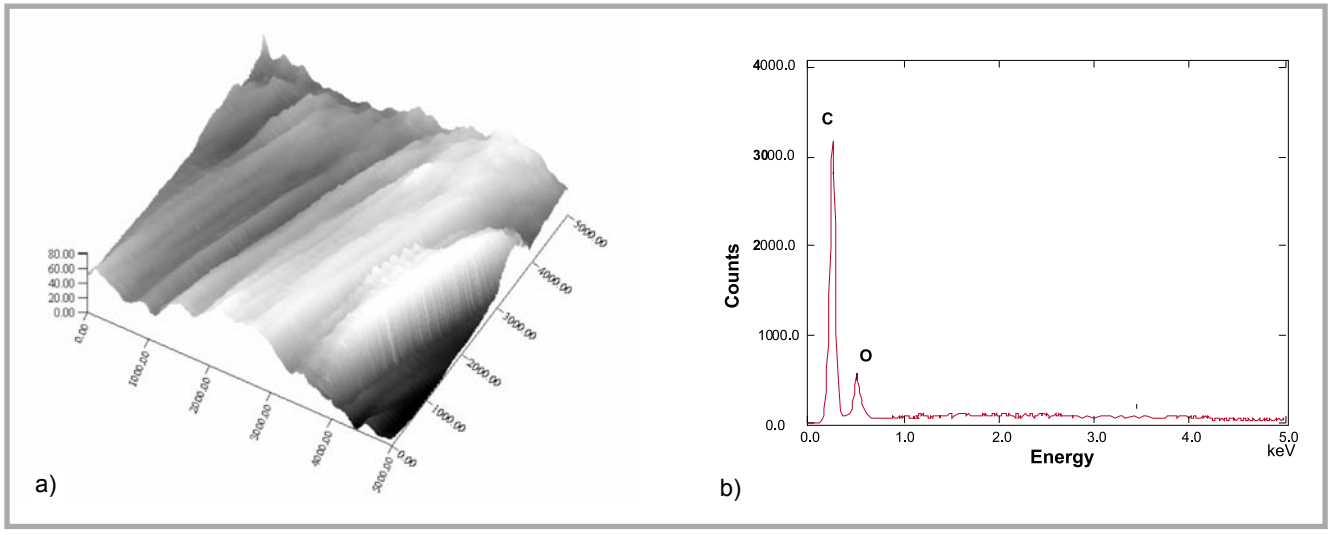

Figure 2. *Untreated PET nonwoven: a) AFM image; b) EDX spectrum.*

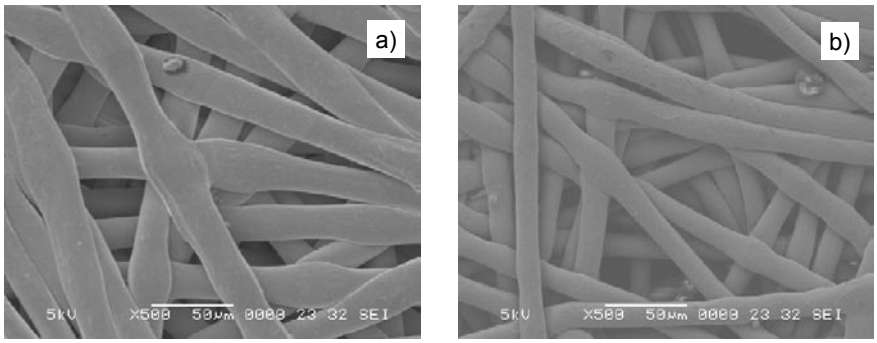

***Figure 3**. Sputter coated nonwoven observed in SEM: (a) 20 min; (b) 80 min.*

0.5 Pa. The power used for Al sputtering was set at 130 W. The sputtering was performed at room temperature for 20 minutes and 80 minutes, respectively.

Surface characterisation

Deposition thickness

The thickness of the deposited layer was examined on-line by a FTM-V quartz detector supplied by Shanghai TAIYAO Vacuum and Technology Co, LTD. during the sputtering process.

Scanning electron microscopy (SEM)

The surface structure of the material was observed in the JEOL JSM-5610LV Scanning electron microscopy (SEM). Images were taken at 5 kV with different magnifications.

Atomic force microscope (AFM)

A Benyuan CSPM3300 Atomic Force Microscope (AFM) was employed to image the morphology of the fiber surfaces. Scanning was carried out in contact mode AFM [6] with a silicon cantilever. All images were obtained at ambient conditions.

Environmental Scanning Electron Microscopy (ESEM)

ESEM is able to image uncoated and hydrated samples by means of a differential pumping system and a gaseous secondary electron detector [7, 8]. The Philips XL30 ESEM-FEG equipped with a Phoenix energy dispersive X-ray analysis system (EDX) was used to examine the chemical compositions of the sputter coated fibres. EDX is available in all modes in the ESEM and all elements down to boron can be detected, including light elements, such as carbon, nitrogen and oxygen [9].

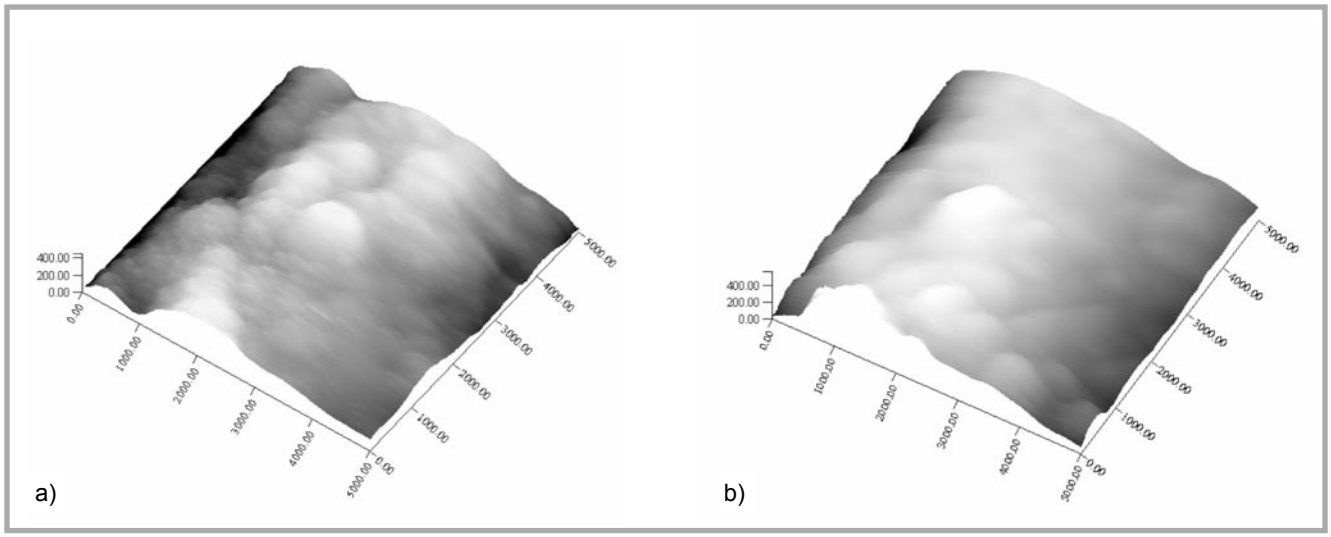

***Figure 4.** Surface morphology of sputter coated fiber: (a) 20 min; (b) 80 min.*

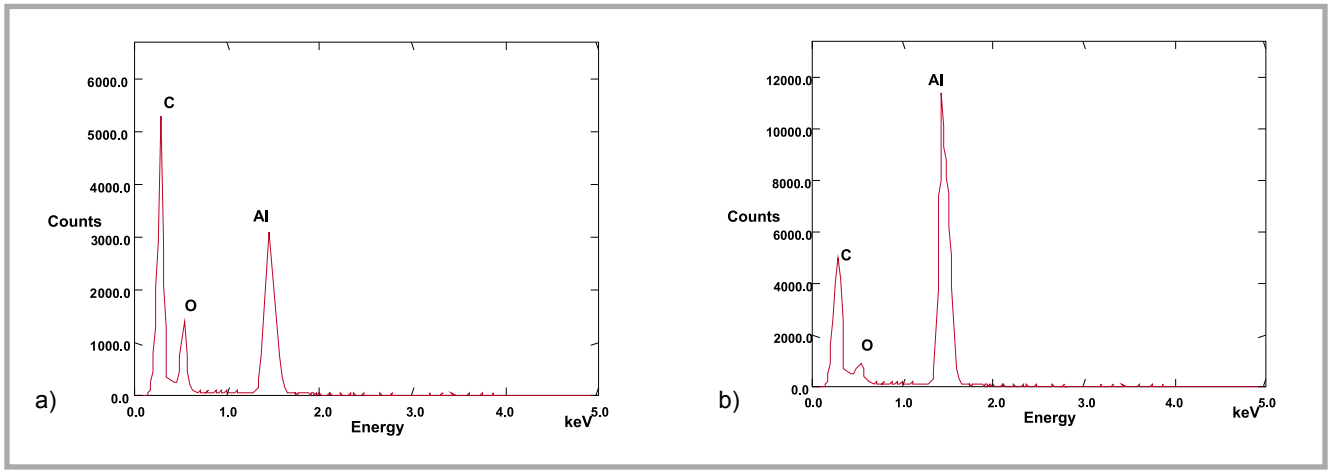

***Figure 5.** EDX spectra of sputter coated nonwoven: (a) 20 min; (b) 80 min.*

Table 1. Resistance values.

Materials	Resistance, Ω/cm
Untreat samples	Out of range (over 10^6)
Treated for 20 minutes	5.3×10^2
Treated for 80 minutes	2.1×10

In the EDX analysis, an accelerating voltage of 20 kV for a counting time of 100 s was applied.

Resistance

The electrical resistance was measured with a digital ohmmeter. The tests were performed three times for each sample and the mean values were reported.

Results and discussion

Untreated nonwoven

Figures 1 and 2 (see page 90) displays the fiber characteristics of the uncoated PET nonwoven fibres observed in SEM, AFM and ESEM. SEM image shows the 3-D fibrous structure web composed of fibres with a diameter of approximately 25 μm in Figure 1. The surfaces of the fibres in the web are smooth except for some visible particle-like structures on the fibre surface, which are formed during the manufacturing process, but more details of the fibre surface can not be observed through SEM. The high magnification image (5000×5000 nm^2) obtained in the AFM clearly reveals the surface topography as displayed in Figure 2.a. The fibril structure can be seen on the fiber surface. The fibril's diameter is in the range between 100 nm and 500 nm. The EDX spectrum in Figure 2.b indicates the main compositions of C and O on the fiber surface.

Sputter coated nonwoven

Thicknesses of the coated layer are 50 nm for 20 minutes coating and 200 nm for 80 min coating according to the readings of the FTM-V monitor.

Al sputter coating formed a functional nanolayer on the fiber surfaces as shown in Figure 3 (see page 91). The SEM images are similar to the 3-D web of fibrous structure in Figure 3, compared to that in Figure 1, but the surface topography of the Al sputter coated fibres is not very clear in the SEM images. Therefore, it is difficult to tell the difference between 20 minutes coating and 80 minutes coating by SEM.

The functional structure, however, can be clearly seen in the AFM image (5000×5000 nm^2) as shown in Figure 4 (see page 91). The surface of the fiber is covered with the sputtered Al aggregates and the fibril structure is no longer visible. The increase in coating time leads to the formation of larger aggregates, as shown in Figure 4 (see page 91). From the AFM image in Figure 4.a, it appears that the grain sizes of the nanocluster are variable in the range from about 20 nanometers to over 50 nanometers after coating for 20 minutes. The grain sizes are increased to 200 nm after coating for 80 min. This can be attributed to the nucleation and growth of Al nanoparticles.

EDX analysis

The funtionalisation of the PET nonwoven by sputter coating is also confirmed by EDX analyses. Figure 5 (see page 91) shows the EDX spectra of the nonwoven after sputter coating. It can be seen that a significant amount of Al on the nonwoven after sputter coating can be seen in Figure 5. The increase in coating time also causes the increase of Al in the spectrum. This is attributed to the covering of nanoclusters on the fiber surface.

Resistance measurement

Table 1 shows the results of resistance measurements of the samples. The table clearly shows a significant decrease in surface resistance after sputter coating. The table also reveals that coating time has an obvious effect on the surface resistance of the materials. The substrate coated for 80 min shows much lower resistance (2.1×10 Ω/cm) than the resistance (5.3×10^2 Ω/cm) for 20 minutes. This is attributed to the increase of the thicker layer on the material and density of the nanostructures on the fiber surface, as well as a better connection in the web of the nonwovens.

Conclusions

Functionalisation of nonwoven materials can be obtained using sputter coating technique at low temperature. The study reveals that the fibrous structure of the nonwoven substrate keeps intact after the sputter coating, but the functional nanolayer of metal Al materials significantly alters the surface of the fibres. It is also found that the increase in coating time resulted in the lower resistance of the substrate. This is attributed to the increase of the coating layer and density of the nanostructures. These functionalised nonwoven materials have great potential for applications ranging from conductive shields, packing and protective materials to technical products. Sputter coating provides an approach to the functionalisation of nonwoven materials.

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