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MATERIALS CHARACTERIZATION

AFM characterization of nonwoven material functionalized by ZnO sputter coating

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Abstract

Sputter coatings provide new approaches to the surface functionalization of textile materials. In this study, polyethylene terephthalate (PET) nonwoven material was used as a substrate for creating functional nanostructures on the fiber surfaces. A magnetron sputter coating was used to deposit functional zinc oxide (ZnO) nanostructures onto the nonwoven substrate. The evolution of the surface morphology of the fibers in the nonwoven web was examined using atomic force microscopy (AFM). The AFM observations revealed a significant difference in the morphology of the fibers before and after the sputter coating. The AFM images also indicated the effect of the sputtering conditions on the surface morphology of the fibers. The increase in the sputtering time led to the growth of the ZnO grains on the fiber surfaces. The higher pressure in the sputtering chamber could cause the formation of larger grains on the fiber surfaces. The higher power used also generated larger grains on the fiber surfaces.

Keywords: Nonwoven; AFM; Surface; Magnetron sputtering; Zinc oxide (ZnO)

1. Introduction

Nonwoven industry is one of the fastest growing industries in the world. Nonwoven materials have been widely used in many industries ranging from wipes to filters [1]. The surface properties of nonwoven materials are of importance in these applications as the surface properties of these materials affect their wetting, sorption, wicking, conductivity and optical properties. However, the surfaces of polymer fibers are often not ideal for a particular application. The inert nature of many polymers has prevented expansion of nonwoven material applications. Various techniques have been developed to modify their surface properties [2].

Thin films of zinc oxide (ZnO) have been applied in many devices such as surface acoustic wave devices, solar cells, electromechanical devices and photo-electronic devices because thin ZnO films possess excellent properties; for example high chemical stability, electrical conductivity and optical transparency in the visible range. Recently, ZnO has been recognized as a promising alternative to transparent conducting indium tin oxide and tin oxide because of its advantage of low cost and non-toxicity [3].

A number of processing techniques, such as ZnO composite fibers [4], sol–gel coating of ZnO thin film [5] and chemical vapor deposition (CVD) of ZnO thin film [6] have been tried, to improve the surface properties of

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Table 1
Sputtering (Group 1)

Sample	Pressure (Pa)	Power (W)	Time (s)
A	2.0	180	1800
B	2.0	180	2700
C	2.0	180	3600

Table 3
Sputtering (Group 3)

Sample	Pressure (Pa)	Power (W)	Thickness (nm)
G	0.8	200	100
H	3.0	200	100
I	6.0	200	100

polymer materials. In this study, polyethylene terephthalate (PET) nonwoven material was processed with zinc oxide by magnetron sputter coating. ZnO sputter coating was performed at room temperature. Atomic force microscopy (AFM) was employed to examine the evolution of the topography of the PET nonwoven fibers.

2. Experimental

2.1. Materials preparation

Spun-bonded PET nonwoven samples with an area mass of 100 g/m^2 were used. The samples were first immersed in acetone solution for 30 min to remove the organic solvent and dusts on the material. Then they were washed twice with de-ionized water. The samples were dried at the temperature of 40 °C. The samples were cut into a size of 25 mm × 75 mm for sputtering.

A magnetron sputter coating system was used to deposit the nano-structured ZnO film onto the surface of PET nonwoven substrate at room temperature. A high purity ZnO target was placed below the substrate and the sputtered ZnO particles were deposited on the side of the nonwoven substrate facing the target. To avoid the deformation of substrate caused by high temperature, water-cooling was used to control the temperature of the substrate during the sputtering process. The sputtering chamber was first evacuated to a base pressure of 5×10^{-4} Pa prior to introducing the high purity argon gas as bombardment gas. During the sputtering, the substrate holder was rotating at a speed of 100 rpm to ensure uniform sputtering of ZnO particles on the substrate. The sputtering conditions are listed in Tables 1–3. The coating thickness was measured using a quartz film thickness monitor (FTM-V) fixed in the sputtering chamber.

Table 2
Sputtering (Group 2)

Sample	Pressure (Pa)	Power (W)	Thickness (nm)
D	0.8	150	100
E	0.8	250	100
F	0.8	300	100

2.2. Surface characterisation

2.2.1. Web in SEM

The fibrous structure of the spun-bonded PET nonwoven substrate was examined in the JEOL JSM-5610LV. The JSM-5610LV is a high-performance, scanning electron microscope with a high resolution of 3.0 nm. Images were taken at 5 kV at various magnifications.

2.2.2. AFM characterisation

A CSPM4000 AFM made by Benyuan Co, Ltd. was employed to scan the surface morphology of all samples. Scanning was carried out in contact mode AFM [7] with a silicon cantilever. The scanning frequency was set at 2.0 Hz. The effect of sputtering conditions on the surface morphology was analyzed based on the AFM observations.

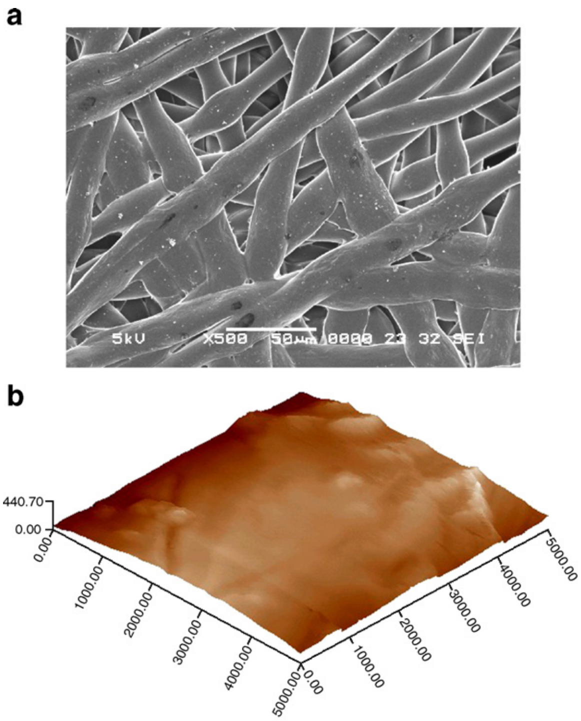

Fig. 1. PET nonwoven substrate: (a) SEM image; (b) AFM image.

3. Results and discussion

3.1. Web of PET nonwoven substrate

Fig. 1a shows the SEM image of the uncoated PET nonwoven substrate. The three-dimensional fibrous web consists of fibers with a diameter of about 25 μm. It can also be seen that the individual fibers have varying diameters formed during the pressing process, as illustrated in Fig. 1a. It can also been seen that the surface of the PET fiber appears to be relatively smooth with some visible particle-like structures on the fiber surface, which are formed during the manufacturing process. More details of the fiber surface can be observed in the AFM, as shown in Fig. 1b. The AFM image clearly shows the particle-like structures on the fiber surface.

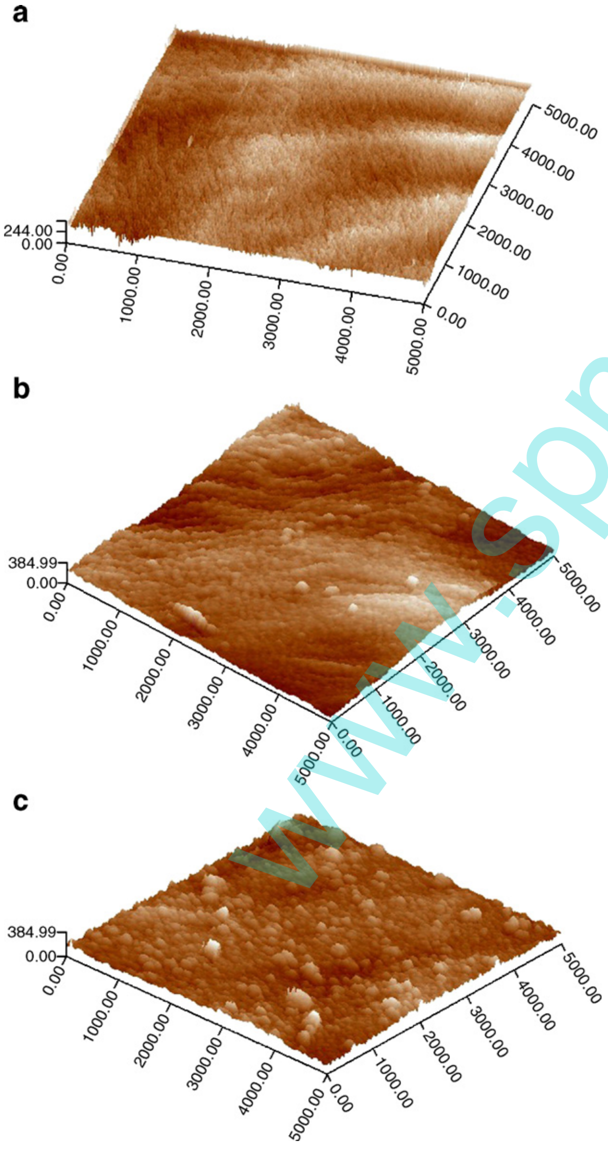

Fig. 2. Surface morphology of PET fiber sputtered for different times: (a) Sample A; (b) Sample B; (c) Sample C.

Table 4
Average sizes of the sputtered ZnO nanoclusters

Sample	A	B	C	D	E	F	G	H	I
Average size (nm)	30.4	37.8	43.6	43.9	48.4	52.4	47.5	46.8	48.2

3.2. Effect of sputtering time

The ZnO sputter coating significantly alters the surface characteristics of the PET fibers. The details of the sputtered ZnO nanoclusters on the PET fibers can be seen from the high magnification AFM images obtained by 5.0 × 5.0 μm^2 scan, as illustrated in Fig. 2. The PET fiber sputtered for 30 min shows the rough surface with clearly recognisable ZnO nano-aggregates, as illustrated in Fig. 2a. The size of the ZnO nano-aggregates is in the range of about 10 nm to over 30 nm. The roughness of the fiber surface increases as the sputter coating extends to 45 min. This behaviour can be attributed to the nucleation and island formation on the fiber surface as ZnO grains are growing, as presented in Fig. 2b. The size of the ZnO nano-cluster varies from about 20 nm to over 40 nm as revealed by the AFM analysis. The extended deposition for 60 min causes the formation of large ZnO aggregates on the fiber surface, as illustrated in Fig. 2c. Table 4 indicates the effect of sputtering time on the size of the sputtered ZnO nano-clusters. It clearly shows an increase in the size of ZnO nano-clusters with increasing sputtering time.

3.3. Effect of sputtering power

Sputtering power can change the deposition speed of the target material. The higher power used will increase the deposition speed. The sputtering power can also affect the grain sizes of the sputtered ZnO nanoclusters on the PET fiber surface. The images in Fig. 3 clearly demonstrate the effect of the sputtering power on the surface morphology of the fiber surface. The AFM images are obtained by 3.0 × 3.0 μm^2 scan, as illustrated in Fig. 3. The PET fiber sputtered with a power of 150 W shows the rough surface with ZnO nano-aggregates, as shown in Fig. 3a. The average size of the ZnO nano-aggregates is about 44 nm. The roughness of the fiber surface is increased and the grain size is also increased as the sputter power is increased. This behaviour can be attributed to more particles being knocked off the target as the power is increased. Fig. 3b shows larger ZnO nano-clusters on the

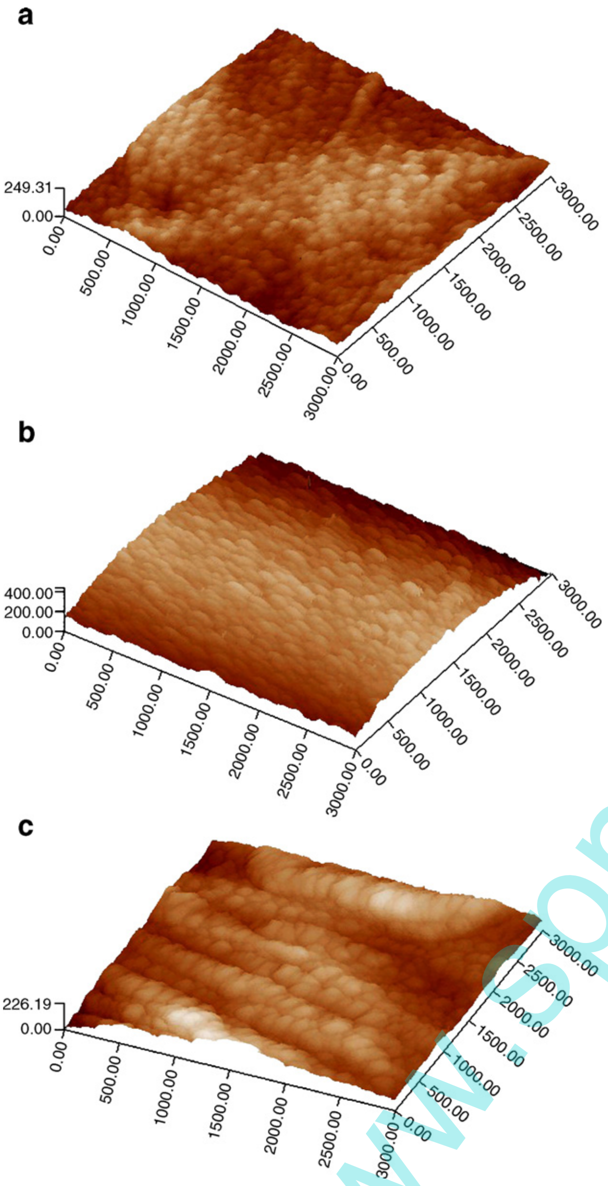

Fig. 3. Surface morphology of PET fiber sputtered with different powers: (a) Sample D; (b) Sample E; (c) Sample F.

fiber surface than those on the fiber in Fig. 3a. The ZnO nano-clusters on the fiber sputtered with a power of 300 W have the largest size, as presented in Fig. 3c. Table 4 lists the results of the image analysis and it clearly shows the increase of the sputtering power from 150 W to 300 W leads to the increase in the size of ZnO nano-clusters from about 44 nm to about 52 nm.

3.4. Effect of sputtering pressure

The pressure in the sputtering chamber also has an effect on the surface morphology of the PET fibers. The AFM images are shown in Fig. 4 and the effect of the pressure can be clearly seen. The sputtered ZnO nano-clusters on the PET fibers show round shapes as the pressure is at about 0.8 Pa (Fig. 4a), but the shapes of the ZnO nano-clusters are altered as the pressure is increased to 3 Pa (Fig. 4b). They look like triangles on the fiber

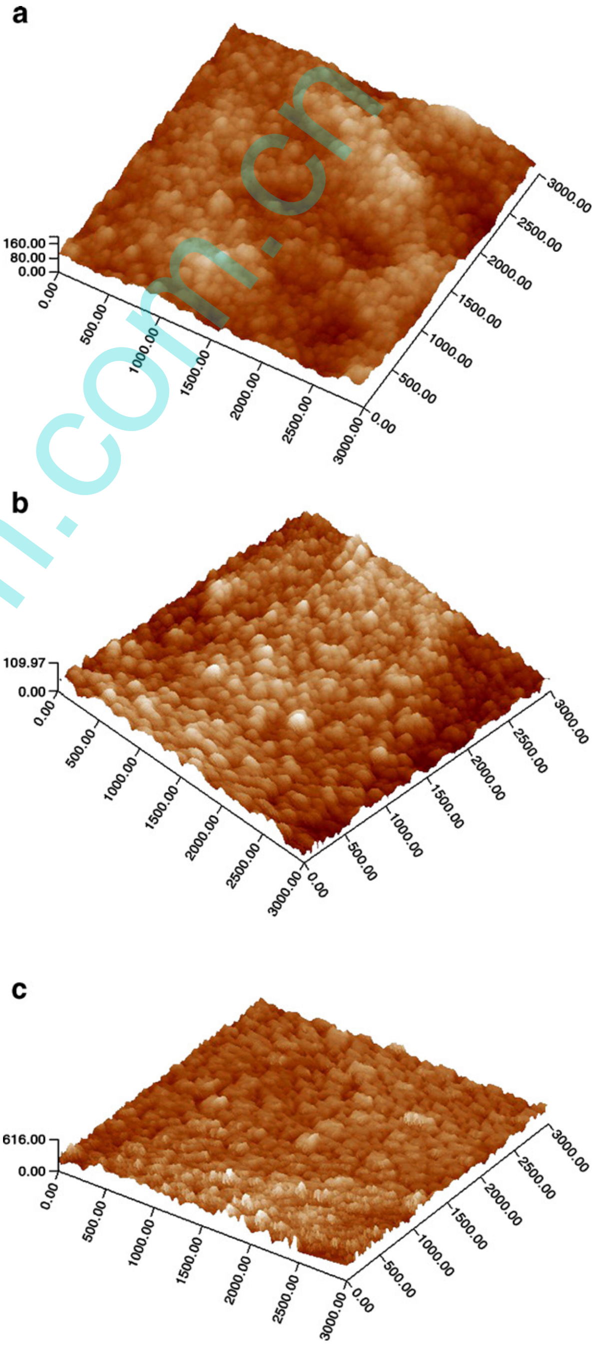

Fig. 4. Surface morphology of PET fiber sputtered at different pressure: (a) Sample G; (b) Sample H; (c) Sample I.

surface. The ZnO nano-clusters change their shape further as the pressure is increased to 6 Pa. The AFM image in Fig. 4c shows the polygon shapes of the ZnO nano-clusters on the PET fiber surface. The change in pressure does not show any obvious effects on the size of the ZnO clusters, as confirmed in Table 4.

4. Conclusion

This work has explored the surface morphology of the treated polymer fibers by sputter coating. The layer of zinc oxide significantly altered the surface of the fibers. The surface morphology was also affected by the sputtering conditions, such as sputtering time, sputtering power and pressure in the sputtering chamber. AFM provides a new approach to the characterization of functional nano-structures. The work has shown that surface properties of polymer fibrous materials can be enhanced or altered using sputtering coatings for a wide range of applications.

Acknowledgements

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