**BIOGRAPHY**

Professor Qufu Wei obtained his PhD from Heriot-Watt University in 2004. He now is director of the Technical Textiles Research Centre at Southern Yangtze University in China. One of his principal research activities is in the functionalisation of polymer fibres using plasma related technology for a wide range of applications. In recent years, this research has involved the application of advanced microscopies to investigate the structure and behaviour of nanostructured polymer fibres.

**ABSTRACT**

In this study, magnetron sputter coating was used to generate functional nanostructures on polyethylene terephthalate (PET) fibre surfaces. Conductive aluminium film, UV absorption zinc oxide film, and transparent and conductive aluminium-doped zinc oxide films were deposited onto the PET fibres by magnetron sputtering at room temperature. AFM and ESEM were used to study the morphology and chemical composition of the functionalised fibres. The observations by AFM also show the change in the morphology of the fibres with different coatings. An energy dispersive X-ray analysis system mounted on the ESEM was used to examine the change in the chemical composition of the functional surfaces.

**KEYWORDS**

atomic force microscopy, environmental scanning electron microscopy, energy-dispersive x-ray microanalysis, sputter coating, polymer fibres, surface

**ACKNOWLEDGEMENTS**

This research was supported by Southern Yangtze University Research Funds (2004LLY005).

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Microscopy and Analysis 19(6):21-23 (UK), 2005

**INTRODUCTION**

Textile materials have been increasingly developed and applied in many industries, ranging from carpets, geotextiles, filtration, medical devices and electronics [1].

The properties of textile materials closely depend on the molecular structures of their surfaces and interfaces. Surfaces are important because in many cases their properties determine how the materials will perform. Many performance properties of textile materials, such as wetting, friction, adhesion, biocompatibility all begin at the surface [2].

Recently, the formation of functional nanostructures on fibre surfaces has been achieved using a variety of techniques [3-5]. In all of these, sputter coating [6] is one of the most promising techniques to generate functional nanostructures on the fibre surface. Functionalisation of polymer fibres is essential for realising their potential in electronic textiles, textile composites, tissue engineering and medical implants.

In this study, magnetron sputter coating was used to generate functional nanostructures on polymer fibre surfaces. Conductive aluminium (Al) film, UV absorption ZnO (zinc oxide) and transparent and conductive aluminium-doped zinc oxide (ZnO:Al) film were deposited onto polyethylene terephthalate (PET) fibres by magnetron sputtering at room temperature. Atomic force microscopy (AFM) and environmental scanning electron microscopy (ESEM) were used to study the morphology and chemical composition of the functionalised fibres.

**MATERIALS AND METHODS**

**Materials**

The materials used in this study were polyethylene terephthalate (PET) spun bonded non-wovens (supplied by BBA Nonwovens). Before sputter coating, the fibres were washed with ethanol and distilled water and dried in an oven at 40°C for 24 hours.

**Sputter Coating**

The sputter coatings were performed in a laboratory magnetron sputtering system and all the sputter coatings were carried out at room temperature. Aluminium deposition was performed at 0.5 Pa in a pure argon atmosphere. The power was set at 100 W and the sputtering was performed for 10 minutes.

For zinc oxide deposition, a pure ZnO target was used and the sputtering pressure was set at 0.3 Pa. Argon and oxygen (95:5) were used as the sputter gases. The power was set at 100 W and the sputtering was performed for 10 minutes.

For the sputtering of aluminium-doped zinc oxide (ZnO:Al), a target of pure ZnO with 3% wt Al\(_2\)O\(_3\) was used. The sputtering pressure was set at 0.3 Pa. Argon was used as the sputter gas. The power was set at 100 W and the sputtering was performed for 10 minutes.

**Figure 1:** Environmental scanning electron microscope image of nonwoven polyethylene terephthalate fibres.
SURFACE CHARACTERISATION

Atomic force microscopy
Scanning probe microscopy (SPM), particularly in the form of atomic force microscopy (AFM), has been increasingly applied in textiles research [7]. AFM uses a sharp probe to examine surface features. AFM is also being used to probe processing and materials including thin and thick film coatings, ceramics, composites, glasses, synthetic and biological membranes, metals, polymers and semiconductors [7]. The AFM used in this work was a Digital Instruments Nanoscope III. Scanning was carried out in tapping mode and all samples were scanned at room temperature in a normal atmosphere.

Environmental scanning electron microscopy and energy-dispersive X-ray microanalysis
The specimens were examined in a Philips XL30 ESEM integrated with a Phoenix energy-dispersive X-ray detector which added extraordinary capabilities to the entire system. It allows the analysis of elemental compositions down to boron including the light elements such as carbon, nitrogen and oxygen. When EDX analysis is used in the ESEM, problems due to charging and interference of sample coatings are no longer an issue [8]. The lack of charging artifacts in the ESEM has direct benefits for X-ray microanalysis. In this study, the fibre surface was examined by the EDX system at an accelerating voltage of 20 kV with a counting time of 100 seconds.

RESULTS AND DISCUSSION

Nonwoven polyethylene terephthalate
Figure 1 shows the fibrous structure of the uncoated nonwoven PET as observed in the ESEM. The web of the material was formed by individual fibres in random orientations. At this magnification the surface topography of the fibre in the web is not clear.

The high magnification image obtained in the AFM clearly reveals the surface topography as displayed in Figure 2a. The fibril structure can be seen on the fibre surface. The fibril dimensions ranged between 200 and 500 nm. The EDX spectrum in Figure 2b indicates the main compositions of C and O on the fibre surface.

Al Sputtering
Al sputter coating formed a functional cluster layer on the fibre surfaces as shown in Figure 3a. The AFM image obtained at a higher magnification clearly displays the nanosized Al clusters on the fibre surface. The nanosized clusters were not uniform on the fibre surface. The sizes of the Al clusters were in the range from a few nanometers to over 20 nm. The presence of seed-like clusters was also observed on the surface. The fibril structure was not visible due to the formation of the Al clusters on the fibre surface. The EDX spectrum in Figure 3b shows the high amount of Al on the fibre surface.

ZnO Sputtering
The ZnO sputter coating generated varying nanostructures on the PET fibre surfaces as revealed in Figure 4a. The nanosized aggregates were formed on the fibre surface after ZnO sputter coating. The high magnification AFM image clearly reveals the rough surface of the fibre. The nanoscale structure of the ZnO aggregates on the fibre surface has sizes ranging from about 10 nm to over 100 nm. The fibril structure was covered by the ZnO nanosized
layer. The EDX spectrum in Figure 4b confirms the high amount of Zn and O on the fibre surface.

**ZnO:Al Sputtering**

Figure 5 shows the AFM image and EDX spectrum of a sputtered aluminum-doped zinc oxide film on a PET fibre.

It can be seen from Figure 5a that the ZnO:Al coating formed a rough coating on the fibre surface. Aggregates of ZnO:Al with varying sizes roughened the fibre surface and covered up the fibrils. The sizes of the ZnO:Al aggregates were variable. The EDX spectrum in Figure 5b reveals the high amount of Zn, O and Al on the fibre surface.

**CONCLUSION**

Sputter coatings have been shown to be a promising technique in obtaining functional nanostructures formed on the surfaces of fibres.

The functional nanostructures formed on fibre surfaces are likely to affect a variety of technological properties, which are surface dependent. These nanostructured fibres have great potential for applications ranging from conductive shields, packing and protective materials to electronic sensors.

**REFERENCES**


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