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Comparative Studies of Silver Nanocomposite Fibers

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ABSTRACT: Functional silver-polypropylene nanocomposite fibers are prepared using melt-compounding and sputter coating techniques. The functional nanostructures formed on the fiber surfaces are characterized by Atomic force microscopy. The Environmental Scanning Electron Microscopy equipped with an energy dispersive X-ray analysis system is employed to examine the chemical compositions of the nanocomposite fibers. It is found that incorporating the silver nanoparticles by melt-compounding caused severe aggregation of the nanoparticles at the polypropylene fiber surface. By contrast, the coverage of the sputter coated fiber surfaces is much more consistent. The anti-bacterial properties of the nanocomposite fibers are also investigated and compared. The antibacterial tests revealed the better performance of silver sputter coated fibers.

KEY WORDS: fiber, silver, nanocomposite, AFM, EDX, surface.

INTRODUCTION

METALLIC NANOSTRUCTURES GENERATED on material surfaces have received considerable attention in recent years, since it has been found they affect wetting, friction, conductivity, adhesion, biocompatibility, and other performance properties [1]. Silver (Ag) has several advantages with respect to other metals. Silver exhibits good optical, electrical, and biocompatibility properties and in recent years it has been used in a variety of applications ranging from optical material to wound dressings [2–3].

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Los Angeles, London, New Delhi and Singapore

Metallic nanostructures can be obtained with a number of different methods generally classified in physical and chemical ones. Nanoparticle-based composites and coatings are commonly used techniques that give solid-state nanostructures under suitable conditions [4–5].

In this study, the silver nanocomposite fibers were prepared using melt-compounding and sputter coating techniques. The nanocomposite fibers were characterized by Atomic force microscopy (AFM) and energy dispersive X-ray analysis system (EDX) in Environmental Scanning Electron Microscopy (ESEM). The anti-bacterial properties of the nanocomposite fibers were also investigated.

EXPERIMENTAL

Materials

Silver nanoparticles (Nanotechnologies) with the size <20 nm were employed as received without further treatment. Nanocomposite fibers were prepared by melt compounding of polypropylene (PP) with silver nanoparticles. PP resins HF445J B2-9037 from Borealis, with a melt flow index (MFI) of 19.0 g/10 min. were used. Nanocomposite fibers were produced on a lab spinning machine (ESL). Before spinning, the polymer chips were first milled into powders and mixed with the silver nanoparticles in a tumbler mixer for half an hour. The weight ratios between the PP polymer and the silver nanoparticles were 100:0.3 and 100:0.5, respectively. The composite fibers were spun at 230°C. The winding speed was 100 m/min and no spin finish was applied during the spinning.

A laboratory D.C. (direct current) sputter coating system was also used to deposit a silver nanolayer on the PP fibers. The PP fibers were prepared on the ESL spinning machine using resins HF445J B2-9037 from Borealis, with a MFI of 19.0 g/10 min. A high-purity silver target was mounted on the cathode, and argon was used as the bombardment gas. The deposition conditions were set at a sputtering current of 20 mA and a vacuum chamber pressure of 10 Pa. The thicknesses of the depositions were 20 and 50 nm.

Surface Characterization

Surface Morphology

The SPM used in this work was a CSPM4000 AFM made by Benyuan Co., ltd. Scanning was carried out in contact mode AFM and all samples were scanned at room temperature in atmosphere. The scanning size was 5000 nm $\times$ 5000 nm, and the scanning frequency was set at 1.0 Hz.

Surface Composition

The Philips XL30 ESEM-FEG equipped with a Phoenix EDX analysis system was used to examine the chemical compositions of the composite fibers. In the EDX analysis, an accelerating voltage of 20 kV with accounting time of 100 seconds was applied.

Antibacterial Property

The antibacterial properties of samples were examined using Shake flask test. The bacteria used were Staphyllococcus aureus. The bacteria were incubated at 37±1°C for 36 hours before counting. The antibacterial properties of the samples were evaluated by calculating the reduction percentage of bacteria [6].

RESULTS AND DISCUSSION

Surface Morphology

AFM observation reveals the surface morphology of the Ag/PP nanocomposite fibers as presented in Figures 1 and 2. The spun Ag/PP composite fiber show rough surface with visible nanoparticle aggregates embedded in the fiber matrix as illustrated in Figure 1(a) when the weight ratio between the PP polymer and the silver nanoparticles is 100:0.3. The rough surface is formed by the Ag nanoparticle aggregations. It is observed that some Ag particles are scattered on the fiber surface as revealed by Figure 1(b), when the weight ratio between the PP polymer and the silver nanoparticles is increased to 100:0.5. The embedded Ag nanoparticles in the fiber matrix are also visible as presented in Figure 1(b).

The images of spun PP fibers and silver sputter coated PP fibers are presented in Figure 2. The spun PP fibers show relatively smooth surface with visible fibril structure on their surfaces, as exhibited in Figure 2(a). It is observed from the AFM image that the fibrils are oriented in the fiber axis direction. Subsequent sputter coating obviously alters the surface structure of the PP fiber, as illustrated in Figures 2(b) and (c). The AFM image in Figure 2(b) reveals the formation of Ag nanoclusters on the PP fiber surface as the coating thickness is 20 nm. It is also observed that the coated fiber losses its original surface structure and the fibril structure is covered up by the silver nanoclusters. The Ag nanoclusters become larger and compacter as the coating thickness reaches 50 nm. The image in Figure 2(c) shows the larger and compacter coating of Ag nanoclusters on the PP fiber surface

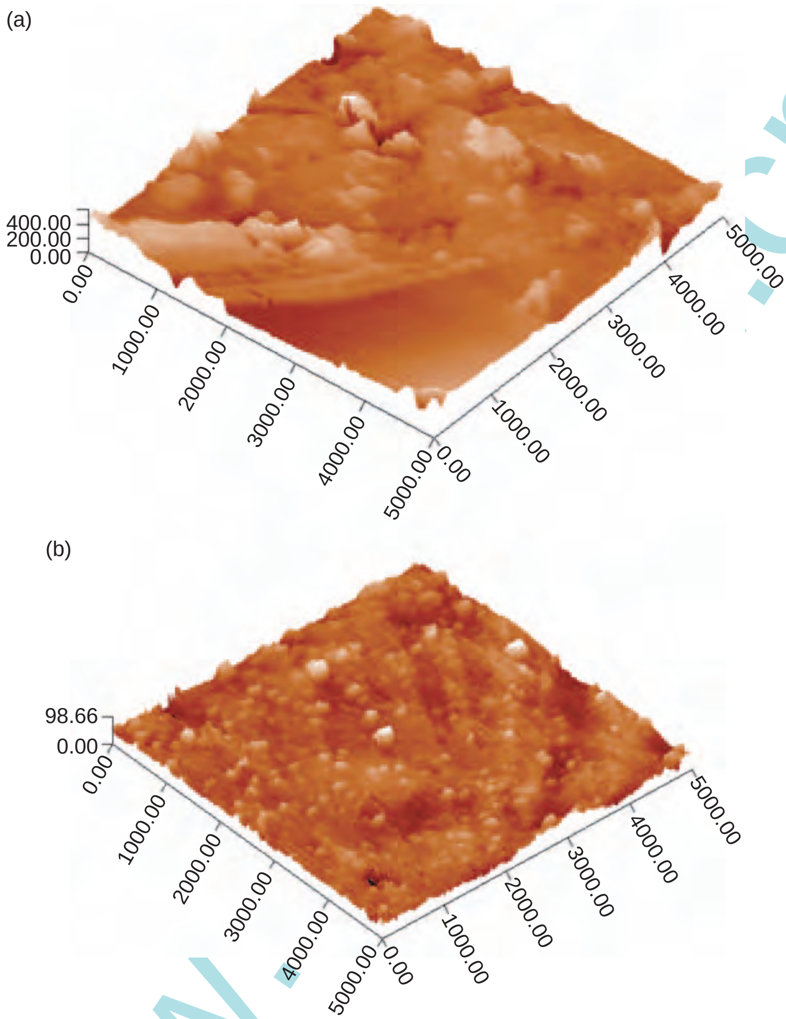

FIGURE 1. Silver/PP spun nanocomposite fiber: (a) weight ratio (PP/Ag)-100:0.3; (b) weight ratio (PP/Ag)-100:0.5.

compared to the image in Figure 2(b). This is attributed to the growth and collision of the sputtered Ag particles as the coating thickness is increased.

EDX Analysis

The EDX analysis confirms the silver nanoparticles dispersed in the PP nanocomposite fibers and the growth of silver nanoclusters on the sputter

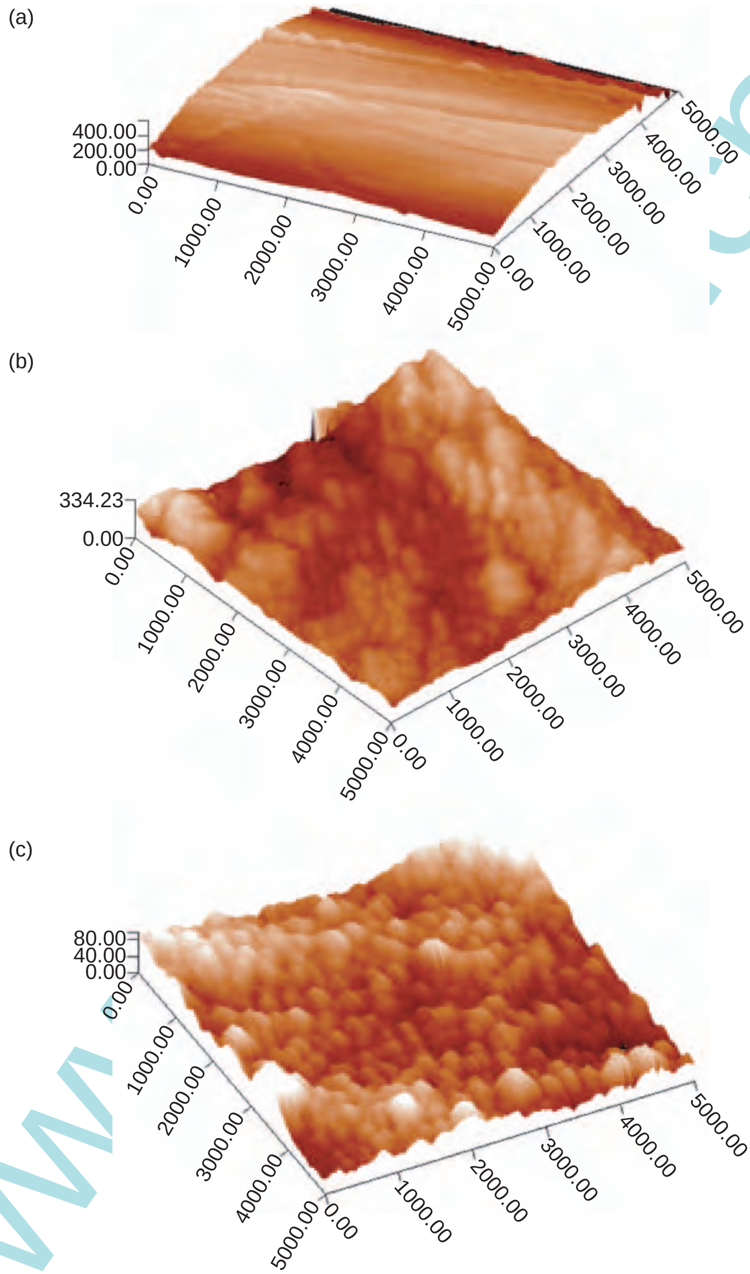

FIGURE 2. Silver sputter coated PP fiber: (a) PP fiber; (b) 20nm coating (c) 50nm coating.

coated PP fiber surface, as shown in Figure 3. The EDX spectrum reveals the main composition of C in the PP fiber and the component of H is too light to be detected in the EDX analysis. The introduction of Ag in the PP fiber matrix is detected from the Ag/PP nanocomposite fibers, as displayed in Figure 3.

The EDX spectrum also shows the chemical compositions of the Ag sputter coated PP fibers. It can be seen that Ag is introduced on fiber surface after Ag sputter coating. The qualitative analysis reveals that the amount of Ag on the fiber surface is much higher than that of Ag/PP nanocomposite fiber. This is attributed to the surface coverage of the Ag sputter coating.

Antibacterial Properties

The antibacterial performance of the PP and Ag/PP fibers is given in Table 1. It can be seen that the samples show different antibacterial properties.

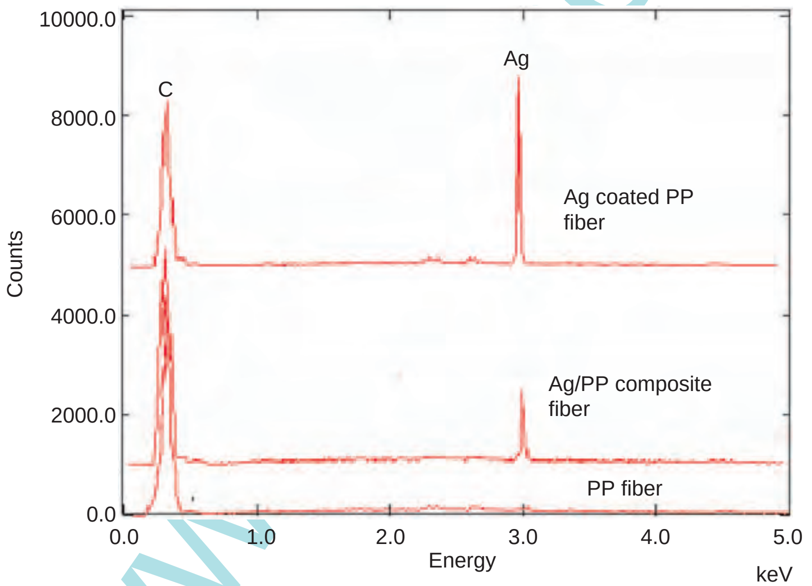

FIGURE 3. EDX spectra of the fibers.

Table 1. Antibacterial properties.

		Ag/PP spun fibers		Ag coated PP fibers	
Materials	PP fibers	100:0.3	100:0.5	20 nm	50 nm
Bacteria reduction Percentage (%)	0	62.5	75.4	100	100

The spun PP fibers do not show any antibacterial properties, but the Ag/PP spun fibers and Ag coated PP fibers show much better antibacterial properties compared to the PP fibers.

The Ag/PP spun fibers show the bacteria reduction of 62.5%, when the weight ratio between PP and Ag is 100:0.3. The bacteria reduction is increased to 75.4%, when the weight ratio between PP and Ag is 100:0.5. The increase in silver content contributes to the improvement in the antibacterial property of the spun PP fibers. The results in Table 1 also reveal that the Ag coated samples have better antibacterial property than the Ag/PP spun fibers. This can be attributed to silver nanostructures formed on fiber surface. The nanolayer of silver clusters formed on PP fibers surface by sputter coating provides better covering as indicated in Figure 2 and therefore the better antibacterial performance. It has been reported that the antibacterial effect of silver mainly depended on the total amount of silver released from the coating [7]. It is believed that increasing the surface coverage obviously leads to the release of a larger amount of silver ions, which contributes to the antibacterial performance.

CONCLUSION

Functional nanostructures can be built on polymer fibers using various techniques. This study has proven that silver sputter coating provide better antibacterial performance compared to silver spun nanocomposite fibers. Sputter coating offers new possibilities in the surface functionalization of polymer fibers and textiles. The coating can be made using a wide range of materials, such as metal, semiconductor, and polymer to improve the surface properties for the fast growing applications of fibrous materials.

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REFERENCES

1. Camacho, J.M. and Oliva, A.I. (2005). Morphology and Electrical Resistivity of Metallic Nanostructures, *Microelectronics Journal*, **36**(3–6): 555–558.

2. Bosetti, M., Massè, A., Tobin, E. and Cannas, M. (2002). Silver Coated Materials for External Fixation Devices: *In vitro* Biocompatibility and Genotoxicity, *Biomaterials*, **23**(3): 887–892.
3. Mohebbi, M., Fedosejevs, R., Gopal, V. and Harrington, J.A. (2002). Silver-coated Hollow-glass Waveguide for Applications at 800 nm, *Applied Optics*, **41**(33): 7031–7035.
4. Park, S.H., Yang, P.X., Corredor, P. and Weaver, M.J. (2002). Transition Metal-coated Nanoparticle Films: Vibrational Characterization with Surface-enhanced Raman Scattering, *Journal of The American Chemical Society*, **124**(11): 2428–2429.
5. Friedlander, S.K., Ogawa, K. and Ullmann, M. (2001). Elasticity of Nanoparticle Chain Aggregates: Implications for Polymer Fillers and Surface Coatings, *Powder Technology*, **118**(1–2): 90–96.
6. Wang, H.B., Wang, J.Y., Hong, J.H., Wei, Q.F., Gao, W.D. and Zhu, Z.F. (2007). Preparation and Characterization of Silver Nanocomposite Textile, *Journal of Coatings Technology Research*, **4**(1): 101–106.
7. Dowling, D.P., Betts, A.J., Pope, C., McConnell, M.L., Eloy, R. and Arnaud, M.N. (2003). Anti-bacterial Silver Coatings Exhibiting Enhanced Activity Through the Addition of Platinum, *Surface and Coatings Technology*, **163–164**: 637–640.

BIOGRAPHY

Dr Qufu Wei is a professor of textiles science and engineering and director of the Key Laboratory of Eco-textiles (Jiangnan University), Ministry of Education of PR China. His research interests lie in the surface funtionalization of textile materials and the development of nanostructured textiles, particularly the synthesis of composite nanofibers and the use of plasma related techniques to create well-defined nanostructures on textile materials. One of his principal research activities is in the complex relationships among the processing, structure, and properties of functional textiles. His research has also involved the application of advanced microscopies to investigate in intimate detail the structure and behavior of various textile materials.