Preparation and Characterization of Copper Nanocomposite Textiles

QUFU WEI,* LIANGYAN YU, NING WU AND SHANHU HONG
Key Laboratory of Eco-textiles of Ministry of Education
Jiangnan University, Wuxi 214122, China

ABSTRACT: Fibrous textiles have been increasingly used in a variety of industries. In these applications, the surface properties of textile materials play a very important role. The surface properties of textile materials can be modified by various techniques. Copper (Cu) nanocomposite textiles are prepared by magnetron sputter coating. The nanocomposite textiles with different thickness of coatings are investigated by atomic force microscope (AFM), energy-dispersive X-ray analysis, and opto-electrical tests. The AFM observations reveal the growth of the Cu nanostructures formed on the fiber surface as the sputtering time increases. The surface conductivity of the textiles coated with Cu nanostructures shows a significant increase compared to the uncoated ones. The increased coating thickness leads to better electrical conductivity. The coated textiles also show considerable improvement in UV and visible light shielding, examined by UV/Vis spectrometer.

KEY WORDS: sputter coating, nanocomposite, copper, AFM, EDX.

INTRODUCTION

TEXTILE MATERIALS IN various forms have been increasingly developed and used in many industries for a wide range of applications [1]. The properties of textile materials closely depend on the molecular structures of their surfaces and interfaces. Surface coatings by metallic materials have attracted a lot of attention in recent years due to their special surface properties, such as heat radiation resistance, antimicrobial, electrical conductive, and chemical resistant properties [2]. Various techniques, such

*Author to whom correspondence should be addressed. E-mail: qfwei@sytu.edu.cn
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as electroless deposition, sol–gel coating, and chemical vapor deposition (CVD) have been used to build functional nanostructures on textile materials [3–5]. Electroless deposition and sol–gel coating are usually performed in liquid solution; therefore the use of these techniques may generate waste solutions and other pollutants. CVD technology is very expensive and often used in semiconductor industries. Compared to these techniques, magnetron sputter coating offers an alternative technique with such advantages as uniform and compact deposition, stronger bonding between the coating and its substrate, and being environmentally friendly [6]. Sputter coating is preformed by applying a high voltage across a low-pressure gas to create plasma, which consists of electrons and gas ions in a high-energy state. During sputtering, energized gas ions strike a target, composed of the desired coating material, and cause atoms from the target to be ejected with high energy to travel to and bond with the substrate, forming a functional coating [7].

In this paper, copper (Cu) nanostructures with different thickness were deposited on the surfaces of polypropylene (PP) nonwovens by magnetron sputter coating. Atomic force microscope (AFM) was employed to analyze the surface morphology of Cu nanostructures formed on the fiber surface. The surface chemical compositions and the optical and electrical characteristics of the coated materials were also examined.

EXPERIMENTAL DETAILS

Materials

Spun-bonded PP nonwovens with an area mass of $50\,g/m^2$ and a thickness of 0.35 mm were used. The fibers in the material had an average diameter of about 17 μm. The samples were first immersed into acetone solution for 30 min to remove the organic solvent and dusts attached on the materials, and then were washed with de-ionized water twice. The samples were dried at 40°C in an oven after washing. The dried samples were cut into a size of $5.0 \times 5.0\,cm^2$ for sputtering.

Sputter Coating

A magnetron sputter coating system supplied by Shenyang Juzhi Co, Ltd. was used to deposit the Cu nanostructures onto the surface of PP nonwovens at room temperature. A high purity Cu target (diameter: 50 mm; purity: 99.999%) was placed below the substrate at a distance of 80 mm and the sputtered Cu particles were deposited onto the PP nonwovens.

To avoid the deformation of substrate and the diffusion movement of Cu particle caused by high temperature, water-cooling was applied to control the temperature of the substrate during the sputtering. The sputter chamber was first pumped to a base pressure of 5×10^{-4} Pa prior to the introduction of high purity argon gas as a bombardment gas. During the sputtering, the substrate holder was kept rotating at a speed of 100 rpm to ensure the uniform coating on the substrate. The sputter coating was carried out with a power of 60 W at a pressure of 2 Pa. The coating thickness was 20, 50, and 100 nm for the three samples, respectively. Thickness of the coating was monitored using a coating thickness detector (FTM-V) fixed in the chamber.

Surface Morphology

Scanning probe microscope (SPM), particularly in the form of AFM has been increasingly applied in textiles research [8]. The SPM used in this work was a CSPM4000 AFM made by Benyuan Co., Ltd. Scanning was carried out in lateral force mode AFM and all samples were scanned at room temperature in atmosphere. The scanning size was $3000 \times 3000\,nm^2$, and the scanning frequency was set at 1.0 Hz.

EDX Analysis

The energy-dispersive X-ray analysis (EDX) integrated with a Phoenix energy-dispersive X-ray detector adds extraordinary capabilities to the entire system. It allows analyzing of elemental compositions down to boron including the light elements, such as carbon, nitrogen, and oxygen. The charging artifacts can be eliminated due to the existence of gas in the ESEM chamber [9]. In this study, the fiber surface was examined by the environmental scanning electron microscope (ESEM) at an accelerating voltage of 20 kV with accounting time of 100 s.

Optical Properties

Many molecules absorb UV or visible light. In molecular absorbance spectroscopy a beam of UV or visible light is directed through a sample. Some of the light may be transmitted through the sample. Light that was not transmitted through the sample was absorbed. An absorbance spectrum depicts what wavelengths of light are absorbed by a sample [10]. The UV/Vis absorbance spectrum, in this study, was obtained by passing different wavelengths of light ranging from 200 to 600 nm through a sample. The UV/Vis spectroscopy used was a PerkinElmer Lambda 900.

Electrical Resistance

Electrical conductivity of samples was measured by point contact using an ohmmeter. Resistances at 1 cm longitudinal intervals of every sample were tested. In order to minimize the deviations brought by the unevenness of textile surface, resistances of each sample were tested 10 times, and the average values were used.

RESULTS AND DISCUSSION

Surface Morphology

The AFM image of the uncoated PP surface is presented in Figure 1(a). The AFM images of PP surfaces coated with nano-structured Cu films are given in Figure 1(b)–(d). The AFM image in Figure 1(a) clearly shows that the uncoated PP fiber had a smooth surface without any particles but with clear periodic stripes on its surface. The periodic stripes were probably induced by drawing during the post processing of PP fibers, leading to the orientation of the molecular chains along the fiber axis [11].

The sputter coating significantly altered the surface characteristics of the PP fibers, as revealed in Figure 1(b)–(d). It shows the surfaces of the PP fiber Cu sputter coated with thickness of 20, 50, and 100 nm, respectively. The Cu clusters scattered on the PP fiber surface after the 20 nm coating, but the clusters had variable sizes from <10 nm to over 20 nm. The surface structures of strips were still visible at this stage, as illustrated in Figure 1(b). As the coating thickness increased to 50 nm, the sputtered functional layer covered up the original surface structures, therefore the strip structures on the fiber surface were not visible any more. The Cu clusters coated on the PP fiber surface looked more even and the sputtered Cu clusters had an average size of 24.6 nm, as revealed in Figure 1(c). The AFM image in Figure 1(d) indicates that the increased coating thickness from 50 to 100 nm led to compact distribution of the Cu clusters on the fiber surface. The growth of the Cu clusters from 24.6 to 28.5 nm was also observed based on the AFM image analysis. This was attributed to the growth of the sputtered Cu grains. The increase in sputter coating time led to the growth of the Cu clusters and more compact deposition.

EDX Analysis

The metallic funtionalization of the PP fiber surfaces by sputter coating was also confirmed by EDX analyses. Figure 2 shows the EDX spectra of

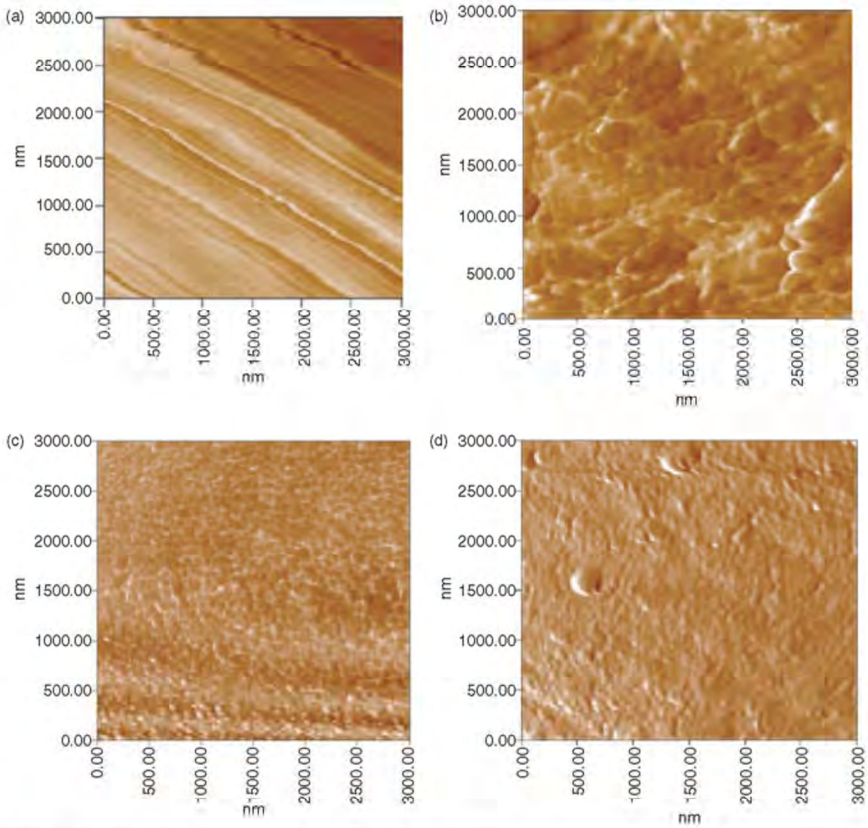

FIGURE 1. Surface morphology of textile fiber: (a) original polypropylene fiber; (b) 20 nm copper coated polypropylene fiber; (c) 50 nm copper coated polypropylene fiber; and (d) 100 nm copper coated polypropylene fiber.

the PP nonwoven material before and after the Cu sputter coating. It can be seen that the surface of the nonwoven material dominantly consisted of C before the sputter coating. The composition of hydrogen in the material was too light to be detected in the EDX analysis. A significant amount of Cu on the nonwoven surface after Cu sputter coating of 20 nm can be seen in Figure 2, but the amount of C reduced in the EDX spectrum, indicating the coverage of the surface by Cu coating. The amount of Cu increased further as the coating thickness reached 50 nm and the composition of C was not detected any more, indicating the full coverage of the Cu clusters on the fiber surface as revealed in Figure 1(c). Therefore it was expected that the 100 nm coating would further increase the amount of Cu in the EDX analysis.

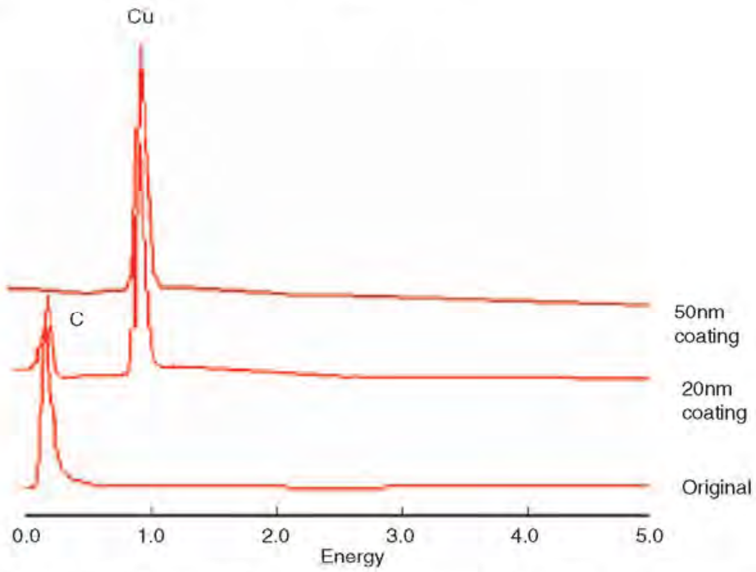

FIGURE 2. EDX spectra of the nonwoven material before and after Cu coating.

Optical Properties

Figure 3 shows the transmittance of the UV and visible light through the nonwoven coated with different thickness of Cu clusters. The uncoated nonwoven showed the transmittance of about 60% in the range of 300–600 nm. This means the uncoated PP nonwoven had a good transmittance of visible light. The transmittance dropped gradually from 60% to about 15% in the range from 300 to 200 nm, indicating the UV shielding effect of the material. The Cu sputter coating significantly altered the optical properties of the PP nonwoven as displayed in Figure 3. The transmittance of visible light reduced considerably to <20% and <10% for UV light as the thickness of the coating was 20 nm. The increase in coating thickness led to the further decrease in transmittance in both UV and visible light range, as revealed in Figure 3. The transmittance dropped to <5% as the coating increased to 100 nm, showing very high light shielding properties.

Electrical Resistance

The results of the electrical conductivity test for the materials are given in Table 1. The uncoated PP nonwoven had a very high surface resistance,

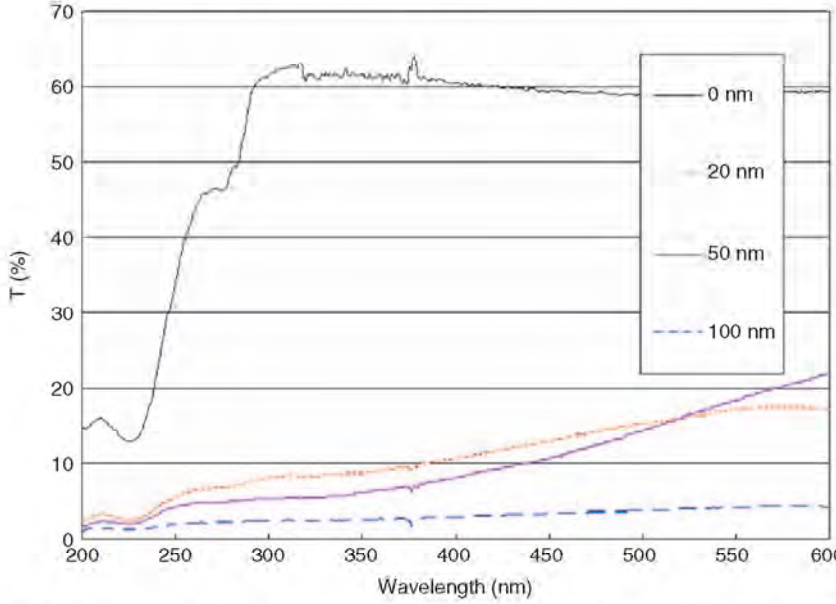

FIGURE 3. Optical properties of the nonwoven material before and after Cu coating.

Table 1. Results of electrical resistance tests.

Coating thickness (nm)	Average resistance (Ω)
0 nm	Out of range (over 10^6)
20	156.72
50	23.57
100	7.85

but the Cu sputter coating reduced the surface resistance significantly as shown in the table. The test results clearly showed that the resistance of the material decreased as the film thickness increased. The surface resistance dropped to about 150 Ω, as the coating had a thickness of 20 nm. The thinner coating formed looser cluster structures, which could generate electric potential barriers, resulting in the higher resistance. The resistance further reduced to about 23 Ω, as the coating increased to 50 nm. Table 1 indicates that the resistance dropped to about 8 Ω, as the coating was added to 100 nm. The increase of the coating thickness led to the formation of compact and improved coverage of the Cu clusters on the fiber surface, resulting in better conductivity.

CONCLUSIONS

This study has explored the construction of Cu nanocomposite textiles using Cu sputter coating. The sputter coating formed nanosized clusters scattered or covered on the fiber surface subject to the sputtering times. Sputtering time affected the grain sizes of Cu clusters. As sputtering time was extended, the grain sizes of the Cu clusters were increased and the coating layer became more compact. The surface electrical and optical properties of the substrate were improved as the sputtering time is increased. The experiments were carried out using small samples, but the process can be made continuous by using rolled feeding system.

The functional Cu nanocomposite textiles offer great potentials for a wide range of applications, such as anti-static, visible light, and UV shielding.

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BIOGRAPHY

Dr Qufu Wei is professor of textiles science and engineering and director of the Functional Textiles Research Centre at Southern Yangtze University in 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 between 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.