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Fabrication of Switchable Superhydrophobic Nonwoven Fabrics via Cosputtering

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Superhydrophobic materials have generated worldwide interest due to their wonderful array of properties and applications. In this paper, a combination of micro- and nano-scale structure was fabricated on a polypropylene (PP) nonwoven fabric through cosputtering of polytetrafluoroethylene (PTFE) and TiO_2 . The effects of PTFE and TiO_2 sputter coating on surface morphology and chemical properties were characterized using atomic force microscopy (AFM), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). The wettability of the fabric was characterized through measuring the surface contact angle by a dynamic sessile analysis (DSA) technique. The contact angle of the coated fabric showed significant superhydrophobic property. The experimental results also revealed that contact angle of the coated fabric could decreased from 151.2° to 31.5° under the irradiation of UV light.

Keywords: Superhydrophobic, Cosputtering, TiO₂, PTFE.

1. INTRODUCTION

Superhydrophobic surfaces with special liquid–solid adhesion have attracted significant interest due to their great importance in material science, biotechnology and microfluidic systems.^{1,2} Surface free energy is an intrinsic property of the material that can be controlled by chemical modification. In textile applications, several studies revealed that hydrophobic surface of clothing and garment could be achieved through the coating of fluorocarbon.^{3–5} In recent years, many researchers revealed the interdependence between surface roughness, reduced particle adhesion and water repellency based on experimental data obtained on microscopically smooth and rough plants.^{6–8} Inspired by these findings, various research groups have developed the superhydrophobic materials through controlling the surface free energy and surface roughness.^{9, 10}

Functional surfaces with controlled wetting properties, which can respond to external stimuli, have attracted huge interest of the scientific community due to their wide range of potential applications, including smart textiles, microfluidic devices and self cleaning surfaces.¹¹ Now, there have been numerous reports on the reversible wettability between hydrophobicity and hydrophilicity.^{12, 13} In this study, a switchable superhydrophobic nonwoven fabric was achieved by cosputtering of PTFE and TiO₂. This surface tenability leads to a switchable liquid wetting surface for water, from 151.2° to 31.5°, under UV irradiation.

2. EXPERIMENTAL DETAILS

2.1. Materials and Preparation

The nonwoven fabric (PP, 140 g/m²) used in this study was purchased from a local fabric store (Wuxi, China). The deposition was performed in a magnetron sputtering arrangement (JZCK-420B, Juzhi Co., Ltd.) including a molecular vacuum pump, RF-DC power source and a control unit. Two pure sputtering targets (99.99%) of PTFE and TiO₂ mounted on the cathode and argon, 99.99% purity as the bombardment gas, were used in this study. The substrate was located 100 mm from the magnetron. Different pressures were adopted to investigate the shape and structure of cosputtering materials formed on the fabric. The sputter coating was performed with 30 minutes for each sample.

2.2. Surface Characterization

Morphology observation was performed employing a <u>CSPM-4400 atomic force microscopy (AFM)</u> which is one of the most effective tools to examine the microstructures of materials. In this study, scanning was carried out in contact mode AFM using a silicon nitride cantilever CSC11 with a nominal force constant of 0.35 Nm⁻¹. All images were obtained at ambient conditions.

A SEM instrument (S-4800, HITACHI, Japan) was used to observe the interfacial structure between the substrate and PTFE-TiO₂ coating. The EDX unit connected to the SEM microscope was used to determine the chemical contents of elements presented in the surface of coated fabrics.

DSA100 apparatus produced by KRUSS Company was employed to measure the contact angle. De-ionized water was dropped onto the sample from a needle on a microsyringe during testing. A picture of the drop was taken a few seconds after the

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drop set onto the fabric. Static contact angles can be calculated by the software through analyzing the shape of the drop.

To investigate the switchable wettability of coated fabrics, a self-made UV light generator was used in this paper.

3. RESULTS AND DISCUSSION

3.1. Surface Morphology and Nanostructure

The SEM image reveals the morphology of the PP fiber surface of the coated and uncoated nonwoven fabric, as presented in Figure 1. The images magnified 300 and 3000 times show some micro-structures on the fibers after cosputtering in Figure 1(b). The coating multilayer is so thin that more detailed nanostructure can't be observed through the SEM image.

The AFM testing is used in this study as a higher magnification microscopy. Compared to the original sample which can be seen from Figure 1(a), the PTFE-TiO₂ coating significantly alters the surface characteristics of the fibers. The details of the cosputtered nanoclusters on the polymer fibers prepared at 0.5, 3.0, and 6.0 Pa argon pressure can be seen from the high magnification AFM images obtained by the 1000 nm scanning, as illustrated in Figures 2(a)-(c), respectively. The AFM images clearly show the roughness of sputtered film generally increases with the working gas pressure. Figure 2(a) shows that the PTFE $-TiO_2$ clusters with an average diameter of 55 nm are formed on the fiber surface where the sputtering is performed at a pressure of 0.5 Pa. As the pressure is increased to 3.0 Pa, larger clusters are deposited on the fiber surface, as indicated in Figure 2(b). It can also be seen from Figure 2(c) that the diameter of the sputtered PTFE-TiO₂ particles enlarges to 350 nm when the working pressure reaches 6.0 Pa. The higher argon pressure increases the collisions, resulting in higher ionization efficiency and higher plasma



Fig. 1. (a) SEM images of uncoated and (b) coated PP fiber surface.



Fig. 2. AFM images of PP fabrics coated under the working pressure of (a) 0.5 Pa (b) 3.0 Pa (c) 6.0 Pa.

density. This may be the main reason for the roughness increase with pressure.

3.2. EDX Analysis

The functionalization of the PP nonwoven surfaces by sputter coatings of PTFE-TiO₂ is also confirmed by EDX analyses. The EDX spectra in Figure 3 shows the PP nonwoven material before and after the cosputter coatings. It can be seen in Figure 3(a) that the surface of the nonwoven material dominantly consists of C and O before the sputter coating. The composition of H in the material is too light to be detected in the EDX analysis. Two clear peaks of F and Ti in the EDX spectrum after sputter depositing can be seen in Figures 3(b) and (c). Compared with the EDX spectra of coating samples, the composition of Ti is obviously increased, while the content of F is significantly reduced with





the increasing of working pressure. The results indicate that high working pressure is conducive to sputter coating of TiO_2 .

3.3. Contact Angles

Surface contact angle of the coated PP fabrics is measured using sessile drop observation. Figure 4(a) shows a water droplet formed on the untreated PP fabric. The surface contact angle of the fabric is 87.2°, in agreement with the polypropylene material to water. The image in Figure 4(b) shows a little increase in contact angle on the PTFE-TiO₂ coated fabric under 0.5 Pa working pressure, which may be ascribed to the function of fluorocarbon groups. It can be seen from Figure 4(c) that the contact angle increases to 151.2° , which reveals that the coating fabric takes on the superhydrophobic property after it was sputtered with 6.0 Pa working pressure. It has been proved that TiO₂ simultaneously possesses photocatalytic and photostimulated superhydrophilic properties.¹⁴ In this study, superhydrophobic property of the coated fabric can be switched to hydrophobic property, as shows in Figures 4(c) and (d). The coated fabrics are shined

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Fig. 4. Surface contact angles of PP fabrics (a) uncoated coated under the working pressure of (b) 0.5 Pa (c) 6 Pa and (d) with UV shining.

under a UV light for 30 minutes. The contact angles of the fabric can be switched from 151.2° to 31.5°. The coated fabric takes on a significantly tunable wettability.

4. CONCLUSION gica

Aug 20 The sputter deposition was proved to be able to modify the surface property of PP nonwoven fabrics, producing the micro-nano structures on the fibers. During sputtering, the working pressure played an important role in creation of $PTFE-TiO_2$ clusters. The higher working pressure could bring the rougher surface morphology, which might be a key factor influencing the changing of contact angle. The EDX testing indicated that high working pressure was benefit to coat the TiO_2 structure. The functional fabric cosputtered under 6.0 working pressure showed the superhydrophobic property which could be easily switched to hydrophilic property through UV light shining.

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