Preparation and Characterization of FC Films Coated on PET Substrates by RF Magnetron Sputtering

Mei-lin Huang[1, 2] , *Sheng-guo* Lu [1*], and *Wen-qin* Du[2]

[1]Guangdong Provincial Research Center on Smart Materials and Energy Conversion Devices, Guangdong Provincial Key Laboratory of Functional Soft Condensed Matter, School of Materials and Energy, Guangdong University of Technology, Guangzhou 510006, Guangdong, China
[2]Guangdong College Research Center of Functional Textile Engineering and Technology, School of Textile and Clothing, Wuyi University, Jiangmen 529020, Guangdong, China

Abstract. Fluorocarbon (FC) films were prepared on polyethylene terephthalate (PET) plates and PET fabrics respectively by a radio-frequency (RF) magnetron sputtering technique using polytetrafluoroethylene (PTFE) as a target. Scanning electron microscope and X-ray photoelectron spectroscopy were used to investigate the morphology, structure and composition of the obtained FC films. The hydrophobicity and uvioresistant properties of the FC film coated fabric were studied. The results show that the FC films were successfully deposited on the PET substrates by a RF magnetron sputtering. The deposited films are made up of four components -CF3, -CF2-, CF- and -C-. The proportions of the four components and surface morphologies of the deposited films vary with the sputtering conditions. Compared with the original fabric samples, the hydrophobicity of the FC film coated fabrics is quite good and improved significantly.

1 Introduction

Magnetron sputtering has been taken into account as a very effective deposition method for growing thin films. It is easy to prepare high-purity and good uniformity films because of its many advantages, e.g., multiple targets, ease of sputtering parameter control, large film area, and good adhesion. The magnetron sputtering technology can be used to prepare super-hard films, wearable and corrosion-resistant films, superconducting films, magnetic films, optical films, and other specific films [1]. It has been using the magnetron sputtering to deposit thin films on fabric to obtain functionalities, such as wettability, hydrophilicity, hydrophobicity, antibacterial property, and uvioresistant property [2-9].

There have been lots of studies on the coating of fluorocarbon films by using magnetron sputtering to achieve various properties [10-15]. Nevertheless, since the properties of the deposited films are greatly affected by the sputtering conditions, e.g., sputtering power, sputtering time, vacuum pressure, substrate temperature, gas flowing rate and other factors

* Corresponding author: sglu@gdut.edu.cn

[16-18], it is of significance to investigate the relationship between the sputtering conditions and the properties of the films coated on the textiles directly.

In this work, the fluorocarbon (FC) films were deposited on the polyethylene terephthalate (PET) plates and PET fabrics, respectively, by a RF magnetron sputtering technology using polytetrafluoroethylene (PTFE) as a target. The influences of different sputtering conditions on the properties of FC films and coated fabrics were studied especially the impact of substrate's temperature. The compositions, morphologies, and microstructures of the films were analysed by a scanning electron microscope (SEM) and an X-ray photoelectron spectroscopy (XPS). The hydrophobicity of the FC film coated fabric were also tested and discussed.

2 Experimental details

2.1 Materials

The substrate material is a kind of PET fabric, which was cut into circular sample with 6 cm in diameter, and washed in acetone and ethanol successively, then vibrated for 10 min by an ultrasonic cleaner, and then repeat for several times using deionized water, and dried finally.

The PET thick plates, which have the same chemical composition as the PET fabric, with 0.35 mm in thickness, were also used as substrates, because it is easier to investigate the morphology, configuration and structure of the films deposited on the PET plates.

The target material used was commercial PTFE plates.

2.2 Deposition of FC films

The RF magnetron sputtering apparatus (Model FJL450E) with a 13.56 MHz power supply was used in the experiments. The FC films were deposited consecutively on PET plates and PET fabrics using PTFE as a target under conditions shown in Table 1. The distance between the target and the substrate was set as 60 mm, with a base pressure of 4×10-4 Pa, sputtering working pressure of 0.5 Pa, and argon (Ar) gas flowing rate of 40 ml/min.

Table 1. The sputtering conditions for deposition of FC films

Sample	Substrate	Sputtering power (W)	Substrate temperature (oC)	Sputtering time (min)
#1	PET plate/fabric	150	40	30
#2		150	60	30
#3		150	80	30
#4		210	40	120
#5		210	70	120
#6		210	100	120

2.3 Characterization

The morphologies were observed by a scanning electron microscope (SEM, JEOJ-JSM-840). The chemical compositions and structures of the deposited films were analyzed using

XPS (VG ESCA LABS5 spectrometer), in which the monochromatic Al Ka radiation was used as the X-ray source. The XPS curves were studied using a conventional analytical procedure [19,20]. The samples' hydrophobicity before and after the sputtering was tested by a static contact angle measurement (JC2000A) with conventional water droplet method and by a water repellency tester (Y(B)813) with standard GB/T4745-1997.

3 Results and discussion

3.1 Surface morphology and structure

The SEM images of the films deposited on PET plates of Sample #1 - #6 are shown in Fig. 1. Results indicate that the surface morphology of the deposited films varies with the sputtering conditions. As can be observed in Fig. 1 (a) - (c), the surfaces of Sample #1 - #3 look like mountains, with concaves and convex, grooves and buckling. The number of grains were less and the surface is not smooth because the sputtering time is shorter and the growth of film insufficient. The grain sizes was ranged from 10 nm to 1 m. There are wrinkles appeared on the surfaces of three PET plates, and the higher the substrate temperature, the worse the wrinkles are. It is obvious that the substrate temperature has less effect on the film formation but more effect on the PET plates themselves. In brief, after sputtering for 30 min in power of 150 W, the FC grains are dispersively deposited on the substrate and the FC film still has not formed.

In Fig. 1 (d) - (f) for Samples #4 - #6, results indicate that after sputtering for 120 min at 210 W, the film's thickness increases with the increased substrate temperature, which makes the surface smooth. With further deposition, contraction crack emerges and the surface becomes fragile and flaws form eventually. When the substrate temperature increases from 40 to 100°C, the surfaces of films become more compact, dense and uniform. The investigations indicate that either the sputtering time of 120 min is too long, or the sputtering power of 210 W is too high, hence the impact of substrate temperature is not obvious. Therefore, the sputtering at a relatively lower substrate temperature is better.

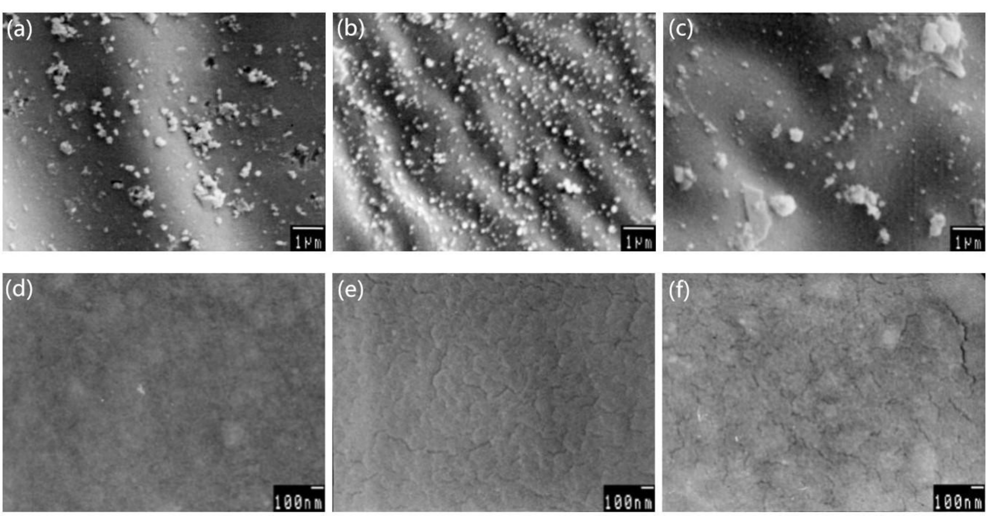

Fig. 1. SEM images of (a)150 W,40°C, 30min; (b)150W,60°C, 30min;(c) 150W,80°C, 30min; (d) 210W,40°C, 120min;(e) 210W,70°C, 120min; (f) 210W,100°C, 120min

3.2 Composition analysis and hydrophobicity study

The composition and chemical structure of the deposited films were determined from C1s spectrum which was resolved into four sub-bands -CF3, -CF2-, CF- and -C-. The software XPSPEAK was used to fit the XPS curves of C1s spectrum and obtain the position, area and FWHM (full width at half maximum) of the peaks of -CF3, -CF2-, CF- and -C-, respectively. The proportions of four components, F/C atom ratio and the static contact angle of Samples #1 - #6 are given in Table 2. A typical C1s spectrum of fluorocarbon film was shown in Fig. 2.

Table 2 Proportions of four components, F/C atom ratio and static contact angle of the FC films

Sample	Substrate	0	$-CF_2-$	-CF-	-C-	F/C ratio	Static contact angle
		(%)					(degree)
#1	PET plate	5.31	18.25	63.51	12.92	1.16	96.9
#2		19.34	5.40	25.92	49.34	0.95	94.6
#3		5.01	13.97	10.75	70.27	0.54	93.4
#4	PET fabric	12.67	12.23	52.74	22.36	1.15	127.5
#5		27.54	46.09	0.07	26.29	1.75	125.2
#6		3.00	27.61	44.86	24.53	1.09	130.3

Zhang et al. observed that the F/C ratio decreases with the increasing sputtering power, and increases with the increasing working pressure [20], the F/C ratio is affected by the sputtering power and working pressure significantly. the influence of substrate temperature on the properties of fluorocarbon films was mainly studied. Table 2 shows that the F/C ratio decreases with the increasing substrate temperature on PET plate, but an opposite result was obtained on the fabric substrate.

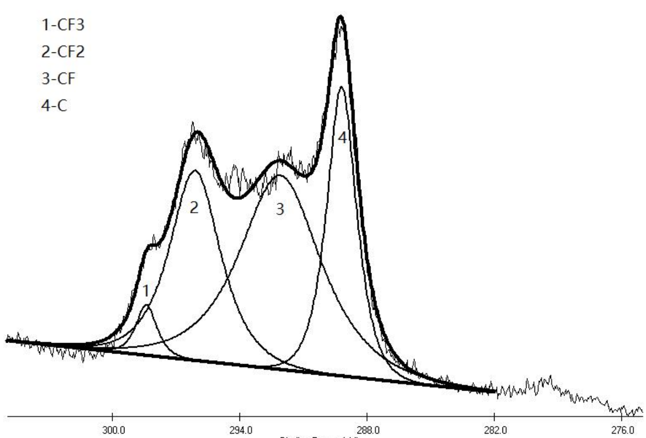

Fig. 2. A typical C1s spectrum of fluorocarbon film

In general, the smaller the F/C ratio, the weaker hydrophobicity of the film is [21]. Because fluorine atoms are hydrophobic groups, less fluorine atoms lead to a smaller F/C ratio, a smaller static contact angle and weaker hydrophobicity. This is true according to the results for Samples #1 - #3, in which the FC films are coated on PET plates. The solid surface is hydrophobic when the static contact angle is larger than 90 degrees, and in this case, the water drop is easy to be moved away the solid surface but not easy to wet the solid. It is slightly increased in the static contact angle of the coated PET plates because there was just particle clusters but no film formed on the surface shown in Fig.s 1 (a), (b) and (c). It seems that the substrate temperature has a slight influence on the F/C ratio and also the static contact angle.

On the contrary, the smaller the F/C ratio, the bigger the static contact angle of the coated fabric Sample #4 - #6 is. After sputtering for 120 min, the static contact angle turns to a value above 120 degrees. It means that the sample's surface changes by sputtering and the deposited films exhibit high hydrophobic properties. The F/C atoms were deposited on the surface and the surface's morphology and roughness change accordingly. At the same time, yarn weaving structure, holes, gaps and fiber microstructure all have impacts on the values of static contact angle.

4 Conclusion

The FC films were successfully coated on the PET substrate by RF magnetron sputtering. The composition of the films is made up of four components -CF3, -CF2-, CF- and -C-. The results indicate that the proportions of the four components and surface morphology of the films change with the different sputtering conditions, leading to changes in the static contact angle. The hydrophobicity of fabrics were improved after coating the FC films.

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