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Evaluation of the interfacial bonding between fibrous substrate and sputter coated copper

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Abstract

The sputter coatings provide new approaches to the functionalization of textile materials. One of the key issues in the use of sputter coatings onto textiles is the interfacial adhesion between the coated layer and the fiber substrate. The interfacial bonding between polypropylene (PP) fibrous substrate and sputter coated copper was investigated and discussed by abrasion test and peel-off test in this study. It was found that the plasma pretreatment and heating during the sputter coating process obviously improved the adhesion of the coating layer to the PP fibrous substrate. The mechanism of the interfacial adhesion between copper and PP substrate was also examined by atomic force microscopy. The AFM observations revealed the surface and interfacial structures of the PP fibers.
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1. Introduction

Textile materials have been increasingly used in many industries ranging from automobile, medical and health care to aerospace [1]. For all these technical applications it is desirable to produce such textile materials with specially designed surface features to meet various needs. Various techniques have been developed to functionalize textile materials [2–4]. In recent years, physical vapor deposition (PVD) [5] has been applied to modify textile materials due to its inherent merits, such as environmental friendly, various functions and solvent-free process. Sputter coating is one of the most commonly used techniques in PVD, which has been widely used in glass, ceramic and micro-electronic industries [6].

Sputter coating produces very thin metallic or ceramic coatings on to a wide range of substrates, which can be either metallic or non-metallic in different forms. Sputter coating has also been used to coat textile materials for technical applications [7,8]. The sputtered atoms have a high energy and when they impinge on any surface, they form a surface coating. The adhesion between the coated layer and the substrate plays a very important role in various applications of the sputter coated materials [9]. Plasma treatment has used to enhance the coating adhesion on the polymer substrates [10]. Plasma treatment has also been increasingly used to modify polypropylene materials [11].

In this study, the interfacial bonding between fibrous substrate and sputter coated copper was investigated and discussed by abrasion test and peel-off test. The effects of plasma pretreatment and heating during the sputter coating process were examined and the mechanism of the interfacial adhesion between copper and polymer substrate was also studied by atomic force microscopy.

2. Experimental

2.1. Materials preparation

The material used in this study was polypropylene (PP) spunbonded nonwoven with a mass per unit area of 75±2 g/m^2. The

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Table 1
Details of the sample preparation

Sample	Plasma pretreatment (s)	Heating during sputtering (°C)	Cu coating (nm)
1	No	No	No
2	No	Room temperature	100
3	30	Room temperature	100
4	60	Room temperature	100
5	90	Room temperature	100
6	No	40	100
7	No	80	100
8	30	40	100

samples of the nonwoven material were washed with ethanol and rinsed with distilled water before the sputter coatings. After washing, the samples were dried in an oven at 40 °C.

The plasma pretreatment of the PP nonwoven samples was performed in a HD-1A vertical laboratory plasma treatment machine to investigate the effect of the pretreatment on the interfacial adhesion of the coating layer. Oxygen was used as the treatment gas. The treatment conditions were set at a pressure of 15 Pa with a power of 50 W.

A magnetron sputter coating system JZCK-420B was used to deposit a nanolayer on the materials. A high-purity Cu target (99.999%) was mounted on the cathode, and the nonwoven sample was placed on the anode with a side facing the target. The DC (direct current) power used for Cu sputtering was adjusted to 50 W. Argon (99.999%) was used as the bombardment gas. The sputtering pressure was set at 0.9 Pa. The thickness of the deposition layer was measured using a coating thickness detector (FTM-V) fixed in the sputtering chamber. The effect of the heating during the sputtering process on the interfacial adhesion of the coated layer was also investigated by using different temperatures. The combination of the plasma treatment and heating was also tried for comparison in this study. The details of the samples prepared in this study are listed in Table 1.

2.2. Abrasion test

The abrasion test was performed on a Zweigle G552 abrasion tester to study the surface and interfacial properties. Abrasion load was 30 g. The size of the sample was 20 cm × 1.0 cm. All the tests were performed at 20 ± 2 °C and 65 ± 2% RH (relative humidity). Each test was carried out three times and the average values were reported.

2.3. Peel-off test

The peel-off test was performed on a Zwick universal materials testing machine to examine the interfacial adhesion of the coated layer. The test speed was set at 200 mm/min in this study. The initial distance was 10 mm. The tape used was 3M600 test adhesive tape. The test samples were cut into 7 cm × 2.5 cm for the peel-off test. The samples were pressed with a load of 400 g for 12 h before the peel-off test. All the tests were performed at 20 ± 2 °C and 65 ± 2% RH (relative humidity). Each test was carried out three times and the average values were reported.

2.4. AFM observation

The surface and interfacial structures of the sputter coated fibers were observed by atomic force microscopy (AFM). The AFM used was a Benyuan CSPM 4000 Atomic Force Microscope (AFM). Scanning was carried out in contact mode AFM with a silicon cantilever. Scanning size was 5000 nm × 5000 nm and all images were obtained at ambient conditions.

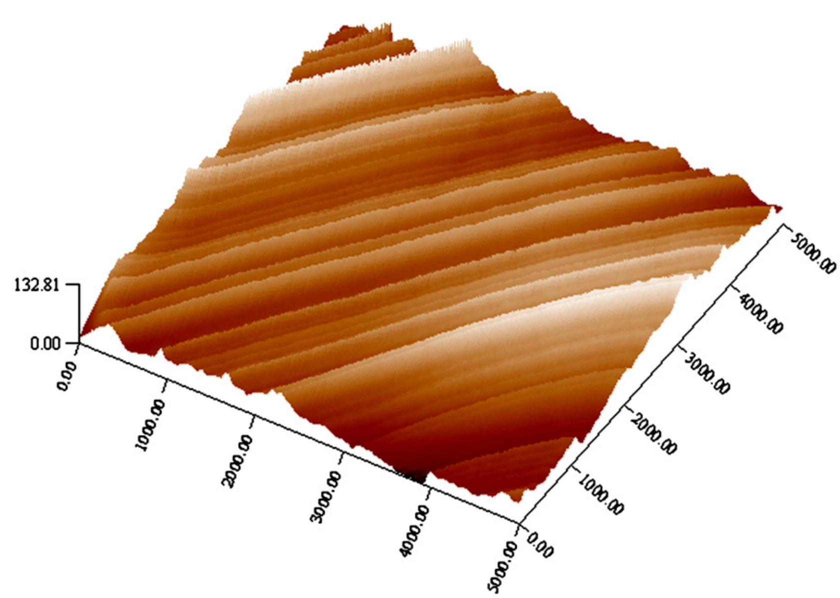

Fig. 1. Surface morphology of the PP fiber (Sample 1).

3. Results and discussion

3.1. Surface morphology

The surface morphology of the original PP fiber in the nonwoven is presented in Fig. 1. The AFM image clearly reveals the groove-like surface of the PP fiber in the nonwoven. The groove structures are the fibrils formed during the spinning process [12]. It can be seen that the groove structures are stretched along the axis direction.

The series of images in Fig. 2 demonstrate the effect of oxygen plasma treatment on the surface morphology of the PP fibers in the

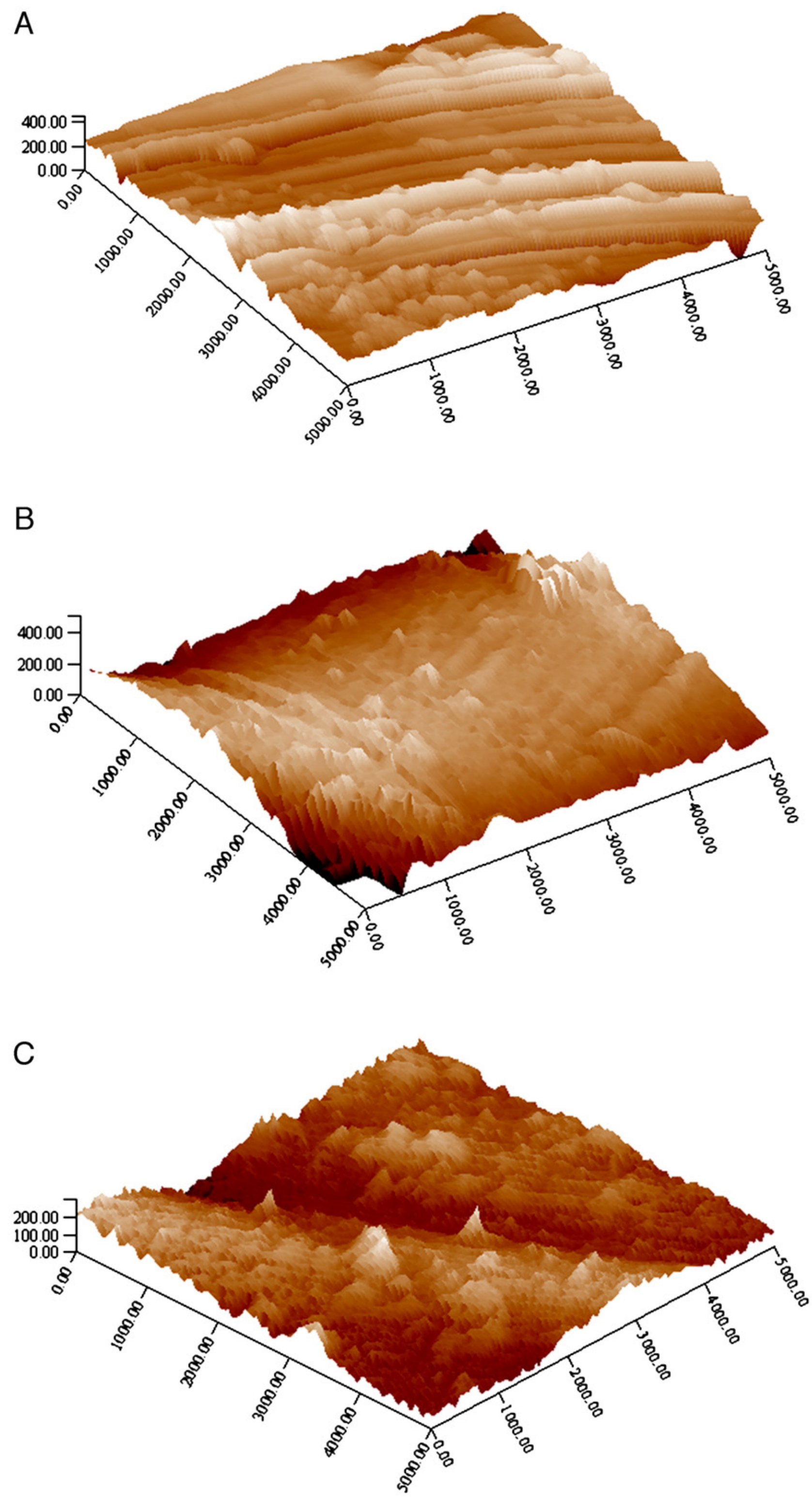

Fig. 2. Surface morphology of the plasma treated PP fibers: (A) Sample 3; (B) Sample 4; (C) Sample 5.

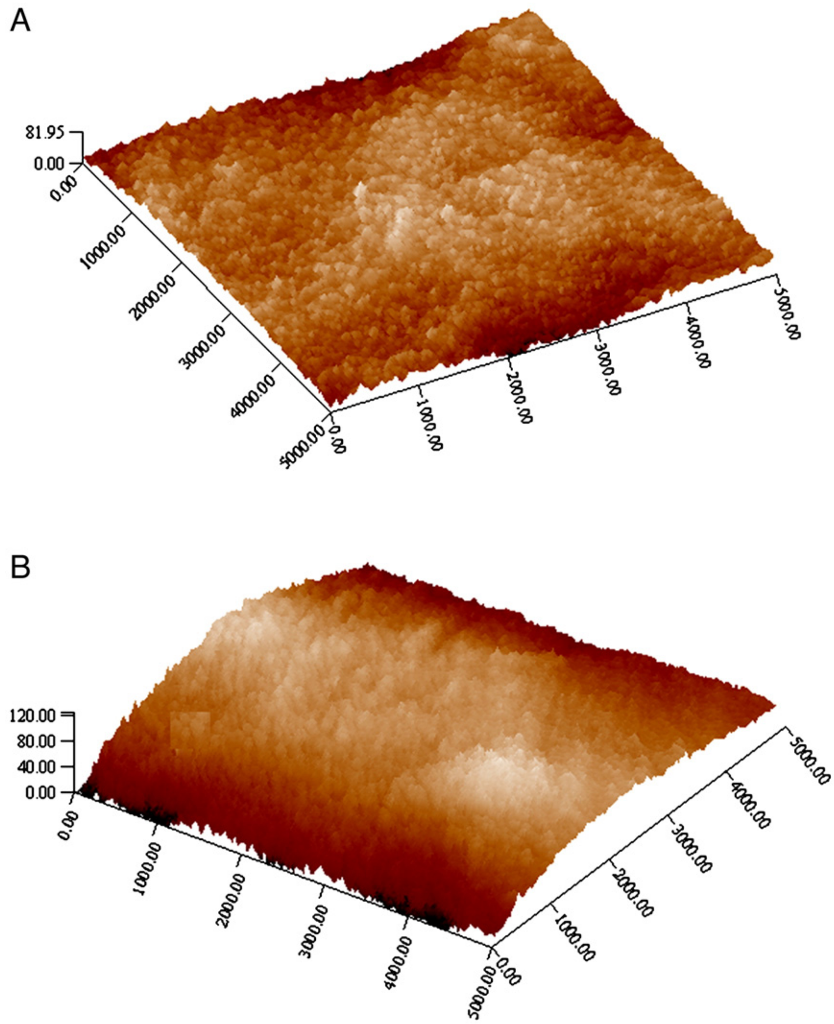

Fig. 3. Surface morphology of Cu coated PP fiber: (A) Sample 2; (B) Sample 3.

nonwoven web. The surface of the PP fiber is obviously roughened after the plasma treatment for 30 s, as shown in Fig. 2(A). The fibril structure is still visible, but some pit-like structures are formed on the fiber surface. The variable sizes of the pits indicate the uneven effect of the surface etching by plasma treatment. Oxygen plasma treatment for 60 s further roughens the PP fiber surface, resulting in the formation of the more pit-like structures on the fiber surface as displayed in Fig. 2(B). The fibril structures are not very clear in this stage, as shown in Fig. 2(B). The further etching by the plasma treatment contributes to the surface roughness. The plasma treatment for 90 s causes the degradation of the fiber surface due to the etching effect, as exhibited in Fig. 2(C) and the fibril structures are not visible anymore.

The sputter coating of Cu significantly alters the surface characteristics of the PP fiber, as indicated in Fig. 3. The images clearly show the formation of nanoclusters covered on the fiber surface. The fibril structures are not visible as the coating with a thickness of 100 nm is formed on the PP fiber surface as illustrated in Fig. 3(A). The surface morphology of plasma treated fiber coated with 100 nm Cu (Fig. 3(B)) shows no difference from that of the untreated PP fiber. This is attributed to the growth of the sputtered copper nanoclusters covering up the fiber surface.

The images in Fig. 4 clearly show the effect of heating during the sputtering process on the surface morphology of the copper coated fibers. The growth of the Cu clusters formed on the PP fiber is observed as the coating temperature is increased to 40 °C. The AFM image in Fig. 4(A) clearly shows the formation of larger clusters on the fibers compared to those on the fiber sputtered at room temperature (Fig. 3(A)). The nanoclusters formed on the fiber surface at room temperature look much more even than those on the fiber surface sputtered at 40 °C, as displayed in Figs. 3(A) and 4(A). The increase in the cluster size is attributed to the collision of the sputtered Cu particles caused by higher energy during heating. The sizes of the clusters are further increased as the coating temperature is increased to 80 °C (Fig. 4(B)). The AFM image in Fig. 4(B) also reveals the rougher surface of the fiber. The image in Fig. 4(C) demonstrates the surface of the Cu coated fibers treated by the combination of plasma treatment and heating. It appears that this surface is very similar to the surface shown in Fig. 4(A).

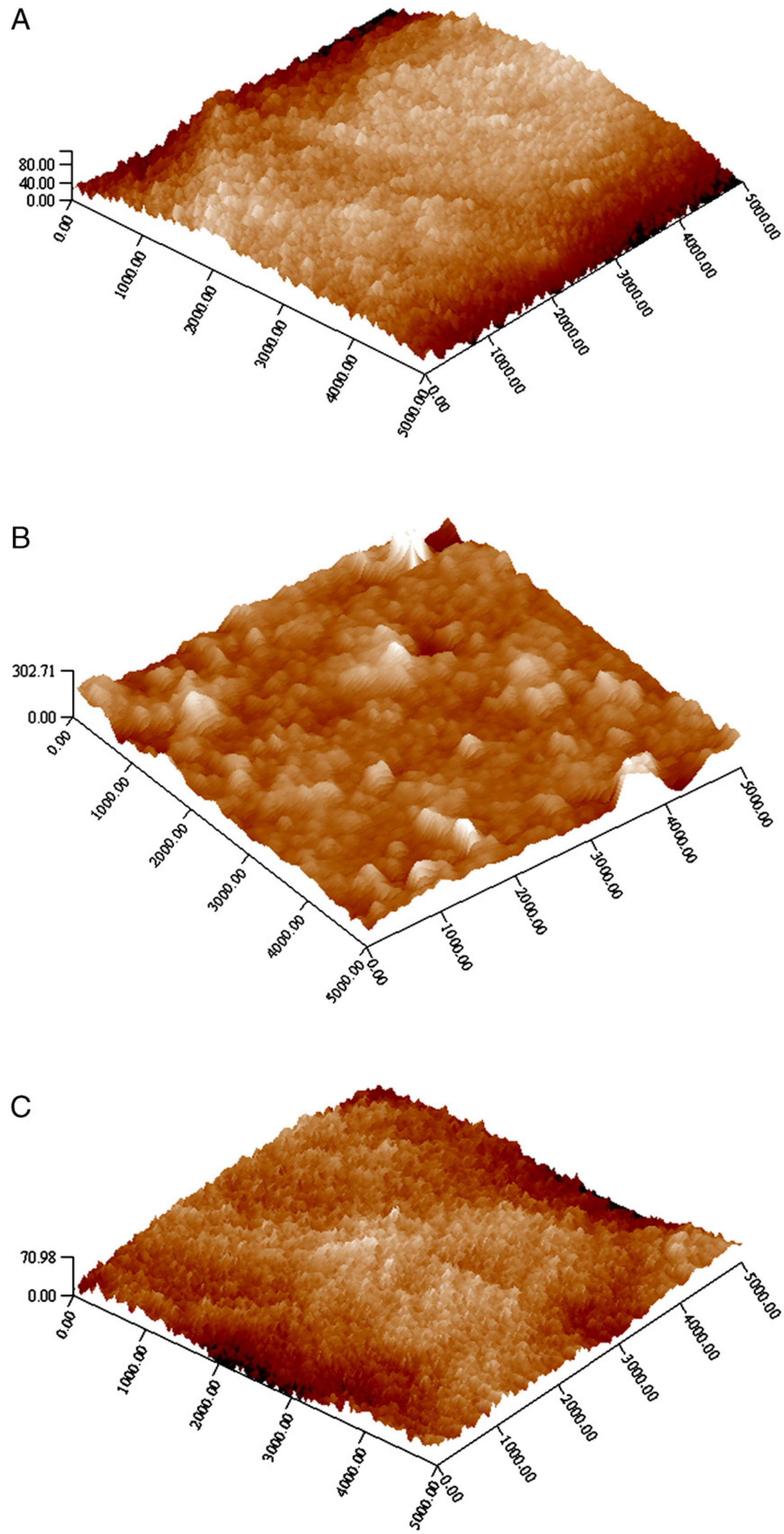

Fig. 4. Surface morphology of Cu coated PP fiber heated during the sputtering: (A) Sample 6; (B) Sample 7, (C) Sample 8.

3.2. Abrasion test

The results of the abrasion test are listed in Table 2. The results clearly reveal the effects of the plasma pretreatment and heating on the abrasion resistance of the Cu coated PP nonwoven. The original PP nonwoven shows the lowest abrasion resistance among the materials tested. The sputter coating of copper significantly improves the abrasion resistance of the PP nonwoven, as presented in Table 2. The higher abrasion resistance is contributed by the nanoclusters of copper formed on the fiber surfaces. It can be seen from Table 2 that plasma pretreatment obviously increases the abrasion resistance of the material, but the pretreatment for 90 s causes the decrease in the abrasion resistance. The plasma pretreatment roughens the fiber surface and facilitates the bonding between the sputtered nanoclusters and polymer. The longer exposure to the plasma for 90 s, however, leads to the decomposition of the fibers as revealed in Fig. 3(C), therefore the abrasion resistance of the material is also weakened to some extent.

Table 2 also reveals the contribution of heating during the sputtering process to the abrasion resistance. It is believed that the heating enhances the bonding between the sputtered nanoclusters and polymer, but the high temperature to about 80 °C may cause the damage to the fibers themselves. The combination of plasma treatment for 30 s and heading at 40 °C enhances the surface abrasion resistance, which contributes to the effects of the surface roughening and particle diffusion.

3.3. Peel-off test

The effects of different processing conditions on the adhesion of the sputtered nanoclusters to the PP fiber substrate were also examined by peel-off test. The results of the test are listed in Table 3. It can be seen from Table 3 that the plasma pretreatment and heating during the sputtering process both contribute to the improvement in the adhesion of the sputtered Cu nanoclusters to the PP nonwoven substrate. The plasma pretreatment roughens the fiber surface as revealed in Fig. 3. The rough surface enhances the adhesion of the sputtered Cu nanoclusters to the PP fibers. The heating at 40 °C obviously improves the adhesion of the coating layer as indicated in Table 3. This is attributed to the diffusion effect of the sputtered nanoclusters, the higher temperature to about 80 °C, however, may cause the deformation of the fibers, leading to the damage to the sputtered layer of copper. It is observed that the combination of plasma treatment for 30 s and heading at 40 °C obviously improves the adhesion of the sputtered Cu nanoclusters to the PP nonwoven substrate. It is believed that the surface roughening by plasma treatment facilitates the interaction between the coated Cu particles and polymer matrix and the heating further enhances the interaction between the coated Cu particles.

Table 2
Results of abrasion test

Sample	1	2	3	4	5	6	7	8
Abrasion resistance (wear cycles)	975	1846	2545	2878	2476	2643	2436	2748

Table 3
Results of peel-off test

Sample	1	2	3	4	5	6	7	8
Peel-off strength (N/cm)	No	1.25	1.55	1.71	1.82	1.77	1.63	1.91

3.4. Interfacial observation

The interfacial bonding structures between the coated clusters and fibers can also be observed by AFM. The images in Fig. 5 show the interfacial structures of the PP fibers after the peel-off test. The interface without plasma pretreatment or heating during the coating shows the separation of the Cu nanoclusters from the fiber surface after the peel-off test, indicating the poor adhesion of the sputtered nanoclusters to the PP fibers, as presented in Fig. 5(A). Some remained nanoclusters are clearly recognized in the image (Fig. 5(A)).

The interface with plasma pretreatment appears much rougher after the peel-off test, compared to that of the untreated fibers. The remained copper clusters are clearly recognized on the fiber surface after the peel-off test, revealing the improved adhesion of the coated layer to the PP fibers. It is believed that the rough surface and functional groups [13] formed on the fiber surface contribute to the better adhesion of the coated material.

The interface with heating during the sputtering coating shows different structures as presented in Fig. 5(C). The sputtered nanoclusters look embedded in the PP fiber matrix, indicating the enhanced adhesion of the coated layer to the PP fibers. It is believed that the heating during the sputtering coating of copper causes the diffusion of the nanoclusters into the fibers, leading to the better adhesion of the coated layer.

4. Conclusion

This study investigated the functionalization of nonwoven by the sputter coating of copper. The surface and interfacial structures were examined by atomic force microscopy. The AFM observations revealed the formation of the functional coating on the PP fibers. The observations by AFM also revealed the interfacial structures affected by the coating conditions. It was found that the plasma pretreatment and heating during the sputter coating process obviously improved the adhesion of the coating layer to the fibrous substrate. The improvement in interfacial adhesion was attributed to the roughening effect by plasma pretreatment and the diffusion effect by heating.

Acknowledgements

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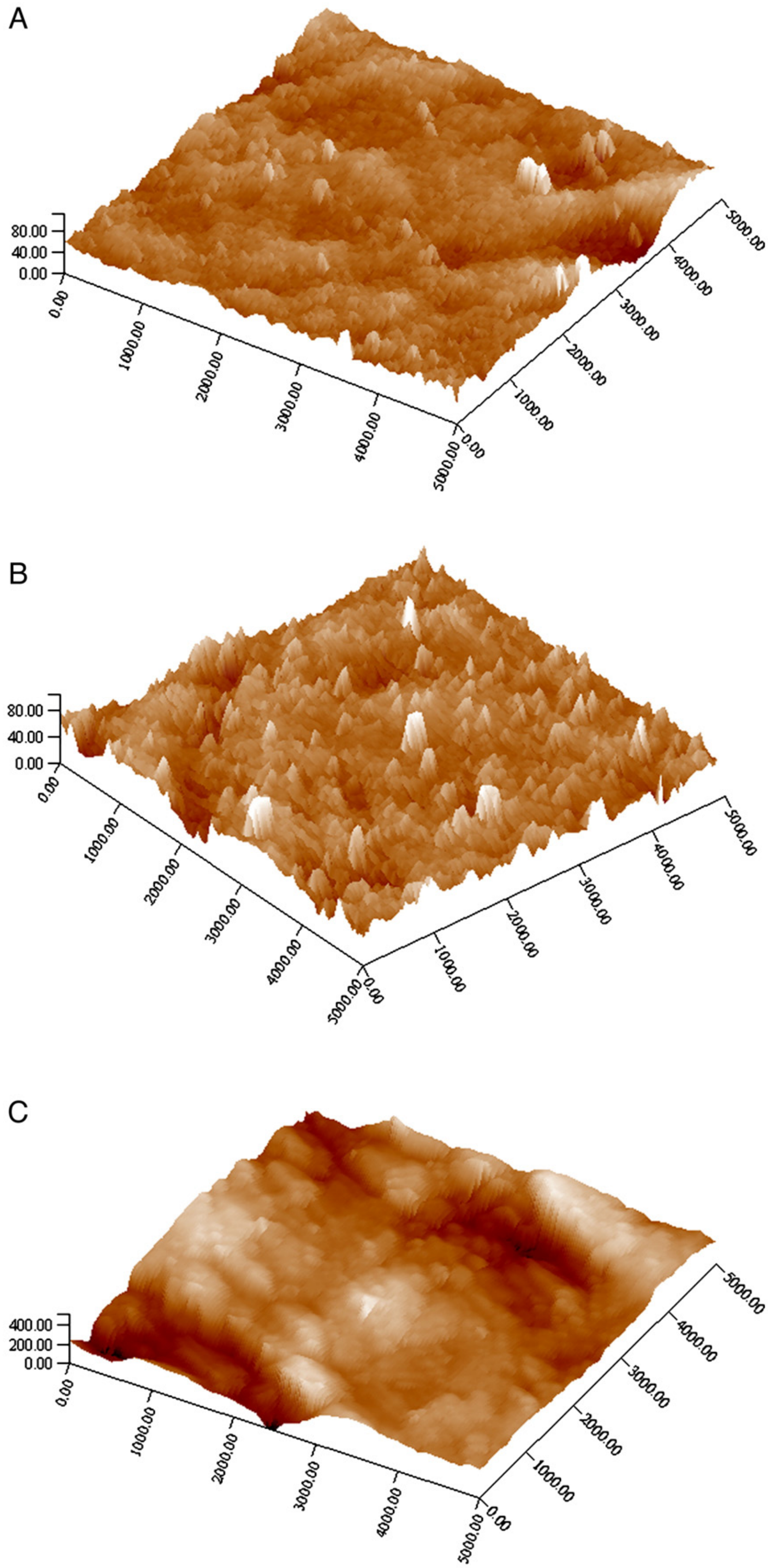

Fig. 5. Interfacial structures observed in AFM: (A) Sample 2; (B) Sample 3; (C) Sample 6.

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