



Influence of pore size on penetration of surface modification into woven fabric treated with atmospheric pressure plasma jet

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ABSTRACT

One of the biggest difference between atmospheric pressure plasma jet (APPJ) and other plasma surface treatment is that only one side (top) of substrate is contacted with plasma jet at atmospheric pressure while in other plasmas two sides (top and bottom) of substrate are both contacted with plasma. The modification of the bottom side of woven fabric treated by APPJ is largely dependent on the penetration of active species in plasma jet, which is accordingly affected by plasma parameters and the structure of materials. In order to investigate the influence of pore size on penetration of surface modification into woven fabric treated with APPJ, four kinds of polyester woven fabrics with different pore sizes were used as the model porous medium. Two groups of parallel polyester fibers are respectively and tightly pasted on the top and bottom side of each fabric. Penetration of plasma effects through the pores was detected by changes in contact angle on the bottom side before and after APPJ treatment. It was further demonstrated by changes in surface morphology and chemical composition using atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS) analyses. The degree of penetration of APPJ surface modification was increased with the increasing pore size. Complete penetration was realized in fabric with pore size larger than 200 μm and nearly no penetration was found in fabric with the pore size smaller than 10 μm . This is attributed to more active species in plasma jet diffusing through the larger pores in fabric. Those species can reach the bottom side without losing their modifying ability during the movement process. Therefore the pore size might be a more important factor affecting penetration of APPJ surface modification into woven fabric.

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1. Introduction

Plasma technology is popularly used as a surface modification technique in the pre-treatment and finishing process of materials. It is known that only the surface of nonporous materials within several nanometers is penetrated by plasma treatment [1]. For porous materials, however, plasma effect is not only limited to the top side but also affects surfaces of deeper layers through pores. According to previous studies on plasma treatments of porous materials, plasma effect can penetrate through the porous substrates. In the study of Masuoka et al. [2], a substance was found to be grafted in the interior and on top surfaces of the polypropylene membrane after low-temperature plasma treatment. Krentsel et al. [3–5] reported the penetration of the CF_4 and C_2F_4 plasma in the filter papers. Johansson et al. [6] indicated that species formed in low-temperature plasma penetrated through a microporous polycarbonate membrane. Geyer et al. [7] proved that process pressure had an important effect on the

penetration of plasma effect through the textile layers at medium pressure. Plasma assisted surface coatings on porous materials was reported by Mukhopadhyay [8,9].

In these studies, the filter papers or nonwoven fabrics were treated at low or medium pressure to investigate the plasma penetration. For those materials, the space between the fibers was much larger than that of microporous woven fabric. For smaller porous structures, the penetration of atmospheric pressure plasma jet surface modification into microporous woven fabric remains to be investigated. If the interior structure of a porous substrate can be treated by atmospheric pressure plasma jet, the utilization of plasma technique will be greatly expanded.

The interaction between active chemical species and substrate surface due to the collision between plasma and polymer surface leads to the breakage of molecules chain, the morphological change and the formation of new functional groups. At the same time, the active species in plasma will easily lose their energy once they have reacted with the material surface [1,10]. The chemically active species produced in APPJ are able to move to the reaction place on the textile fiber surface and modify textile surface molecules by chemical reaction after impinging upon the surface. It is assumed that active species react or recombine after several collisions with gas particles and fiber surface. Some active

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species in plasma jet reacting with the surface of textile fibers will deactivate the reaction site. Other new active species are able to penetrate through pores even if they hit the deactivated surface site again [11]. The smaller the pore size, the larger is the actual surface area of fabric and the more the reaction site is on the fabric surface. Thereby, the lifetime of more active species in plasma jet that collided with the fabric surface is reduced (more active species in plasma jet collide with the fabric surface reducing their lifetime), and as a result, the species cannot reach the reaction sites inside the voluminous fabric. Moreover, with the increasing pore size, the number of collision and chemical reaction between active species in plasma jet and fiber surface is decreased when those active species penetrate through the pores. And more active particles can penetrate into textile structure and reach the bottom side with their modifying ability. It can be concluded that pore size is an important factor affecting the penetration of APPJ surface modification into the woven fabric.

In this paper, penetration of APPJ surface modification into textile structure in the case of hydrophilisation of polyester fibers was investigated. The pore size is considered as the most important factor. Polyester fibers pasted on the top and bottom sides of woven polyester fabrics with different pore size were used as the model system. Penetration of plasma effect through the pores was detected by the changes in hydrophilicity of fiber surface, morphological roughness, chemical composition as well as fiber tensile strength. These properties were characterized by optical contact angle test, atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS), and single fiber tensile test.

2. Experimental

2.1. Materials

The polyester fibers were provided by Yizheng Chemical Company (Jiangsu, China) with an average diameter of 60 μm . Four kinds of polyester fabrics with the same fabric density of 76×68 were selected. The nominal pore sizes are 10 μm , 60 μm , 100 μm and 200 μm respectively, which were referred to as Fabric 1, Fabric 2, Fabric 3 and Fabric 4 in this experiment.

2.2. Sample preparation

Before plasma treatments, the polyester fibers and fabrics were cleaned in acetone for 30 min to remove the residues and dried in a vacuum oven. Then the cleaned fibers were flatly pasted on the top and bottom sides of the washed fabric (Fig. 1), which was later placed on the substrate conveying belt vertical to the plasma jet. The layout of the fibers and the selected test sample were close to a pore as much as possible.

2.3. Plasma treatment

Plasma treatment was carried out in an atmospheric pressure plasma jet (APPJ) apparatus manufactured by Surfex Technologies (California,

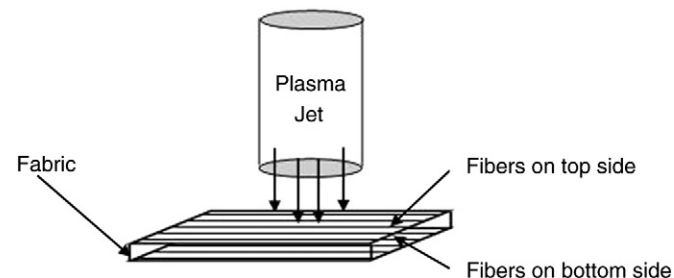


Fig. 1. Pasted fibers on the top and bottom sides of fabric for APPJ penetration studies.

USA) at 65% RH. The substrate conveying belt system was fabricated to move the treated samples at a constant speed. This device employed a capacitively coupled electrode design and produces a stable discharge at atmospheric pressure with 13.56 MHz radio frequency. The plasma jet system with a round nozzle mounted above the conveying belt covered an active area of $1^2 \times 3.14 \text{ mm}^2$. The samples moved underneath the plasma jet at a speed of 3 mm/s, resulting in an equivalent stationary treatment time under the plasma jet for 2.67 s. The flow rate of helium and oxygen was 20 L/min and 0.2 L/min respectively. The power was set at 30 W. After plasma treatments, the pasted fibers on two sides were carefully separated for other properties measurements.

2.4. Hydrophilicity measurement

The hydrophilicity was evaluated by measuring the contact angle between fiber and water. The sessile drop method was applied on Micro Optical Angle Measurement OCA40 (Dataphysics, Germany) by observing the digital images of the distilled water droplet of 50 nL on fiber surface.

2.5. Surface morphology analysis

The topographical studies on the fiber surfaces were carried out using Ultra-high Vacuum Scanning Tunneling Microscopy (Omicron, Germany). Tapping mode was used to prevent significant deterioration of fiber surface. Each AFM image was analyzed in terms of surface average roughness (Ra) and root-mean-square (Rms) of 0.25 $\mu\text{m} \times 0.25 \mu\text{m}$ area on the surface of samples [12].

2.6. Surface chemical composition analysis

The surface chemical composition of the fibers was analyzed by XPS measurements on Thermo ESCALAB 250 system equipped with a Mg Ka X-ray source having a pass energy of 1253.6 eV. The analysis was carried out under UHV conditions (10^{-9} – 10^{-10} Torr). The power was 300 W and spectra were taken at 90° .

2.7. Single fiber tensile test

In order to find out if the plasma treatment could have any adverse effect on single fiber tensile strength, the tenacity of the fibers was tested and compared. The single fiber tensile test was carried out on a XQ-1 Fiber Tensile Testing Machine at a gauge length of 10 mm and a cross-head speed of 10 mm/min under standard conditions (20 $^\circ\text{C}$ and 65% RH). A Nikon Elips E400 P01 reflected light microscope was used to measure the fiber diameter.

2.8. Statistical analysis

One-way analysis of variance (ANOVA) and Tukey's Pair-wise Multiple Comparison were employed to analyze test results of average roughness, contact angle and tensile strength [13,14]. A P-value smaller than 0.05 was considered significant in all analysis.

3. Results and discussion

3.1. Morphological observation

Images of the control and the treated polyester fiber surface observed by AFM on $1 \times 1 \mu\text{m}^2$ area are presented in Fig. 2. The original polyester fiber has a relatively flat and uniform surface. However the pinnacle-like structure can be observed in the AFM images of treated samples because the APPJ treatment creates micropits similarly reported by Poletti et al. [12,15]. The density, depth and size of micropits on the bottom side of fabric increase with the increasing pore size of fabric.

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