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Diffuse plasma treatment of polyamide 66 fabric in atmospheric pressure air

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A B S T R A C T

The polyamide 66 (PA66) fabrics are hard to be colored or glued in industrial production due to the poor hydrophily. Diffuse plasma is a kind of non-thermal plasma generated at atmospheric pressure in air. This paper proposes that large-scale diffuse plasma generated between wire electrodes can be employed for improving the hydrophily of PA66 fabrics. A repetitive nanosecond-pulse diffuse-discharge reactor using a cylindrical wire electrode configuration is presented, which can generate large-scale non-thermal plasmas steadily at atmospheric pressure without any barrier dielectric. Then the reactor is used to treat PA66 fabrics in different discharge conditions. The hydrophilicity property of modified PA66 is measured by wicking test method. The modified PA66 is also analyzed by atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS) to prove the surface changes in physical microstructure and chemical functional groups, respectively. What's more, the effects of treatment time and treatment frequency on surface modification are investigated and discussed.

1. Introduction

The polyamide 66 (PA66) is an important synthetic with high strength and impact resistance, and has been broadly used as artificial fibers in many industry fields [1–4]. However, the poor hydrophily makes it difficult to be colored or glued in industrial production [5]. Therefore, PA66 fiber/fabric often needs to be preprocessed. The chemical treatment is a kind of widely used pre-treatment method. However, large amount of waste water with diverse toxic chemicals may be produced during the chemical treatment. This will probably lead to high environmental cost.

In recent years, plasma treatment has gained the recognition due to the environment-friendly property [6–9]. A. Nakahira et al. found that the interfacial shear strength of glass fiber would be intensified by low temperature plasma with short treatment time [10]. Using unipolar nanosecond pulses, two typical discharges exhibiting homogeneous and filamentary modes were applied to modify the surface of PET films by Zhang et al. [11] Their report showed that the surface roughness increased and the contact angle lowered. Lai et al. studied on hydrophilicity of polymer surface improved by plasma treatment [12], they founded that C=O, C—O, COOH and C—NH2 appeared after the plasma treatment, what's more, the carbonyl group was the key factor to the improvement of the hydrophilicity. Up to now, most of the plasma treatment methods were processed at low pressure or/and inert gas environment to prevent plasma transformation from non-thermal mode to thermal mode. As the development of pulsed power technology, the repetitive nanosecond-pulse discharge becomes one of the most promising methods to generate steady non-thermal plasmas at atmospheric pressure in air [13,14]. Furthermore, the atmospheric pressure nanosecond pulse air diffuse discharge (ANPADD), as a special discharge form, have attracted many attentions for its low power and high chemical reaction property [15–17].

Usually, ANPADD is applied in the company of the dielectric barrier discharge (DBD) [18]. However, the barrier dielectric in the DBD may lead to plasma pollution, and it is hard to produce large-volume plasmas [19]. The size of the treated materials is limited in the discharge direction. In previous studies, large-volume diffuse plasmas have been obtained using wire electrodes and repetitive nanosecond pulses at atmospheric pressure in air without barrier dielectric [20,21]. This made it possible to investigate the related applications of the diffuse plasmas.

In this paper, we would report the experiment results that the hydrophily of polyamide fabrics are modified through atmospheric-pressure air diffuse plasmas which are generated in a cylindrical discharge chamber. This designed chamber could be continuously rotated to get homogeneous surface modification. Effects of treatment time and pulse repetition frequency on

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surface features are investigated and discussed. In addition, through analyzing and comparing under different discharge conditions, it is proved that adding treatment time can achieve the same treatment effect while the requirement of pulse repetition frequency could be reduced.

2. Experimental

2.1. Materials

As for analysis samples, the mesh number of the processed PA66 fabrics is 400 (38 μm pore size). All the samples are sterilized by alcohol firstly, and then cleaned up by the deionized water. At last, they are dried in vacuum drying device before the plasma treatment.

2.2. Experimental setup of the diffuse discharge

The schematic of experimental setup is shown in Fig. 1, which can be divided into a repetitive nanosecond pulse generator, a match electrical circuit, a cylinder reactor and the measurement devices. As shown in Fig. 1(a), it is the front view of the setup. The colors of orange and blue stand for the high-voltage (H.V.) and grounding pole, respectively. A 500 Ω resistor is connected to negative high-voltage pole to control the discharge current value.

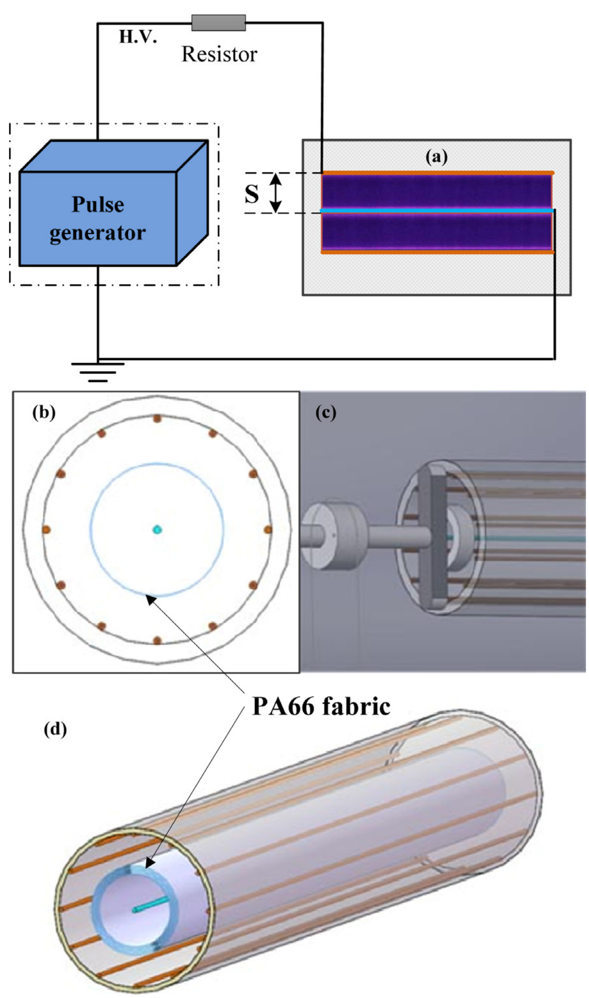

Fig. 1. Schematic diagram of the experimental setup. (a) The front view of the setup (The colors of orange and blue stand for the high-voltage and grounding pole, respectively). (b) The side view of a cylindrical electrode configuration (The blue circle represents the material to be processed). (c) The 3D image of the supporting structure. (d) The 3D image of the position of the polyamide fabrics being processed.

Fig. 1(b) shows the side view of the cylindrical electrode configuration. Similarly, the orange and blue spots stand for the connections with the high-voltage and grounding pole, respectively. According to the previous researches [14,15,22], the short rise-time and narrow pulse-width are necessary to the diffuse discharge. As for the pulse generator in Fig. 1, the pulse rise-time and peak-voltage are around 30 ns and −70 kV here, respectively. The full width at half maximum (FWHM) is ~750 ns. The pulse repetition frequency can vary from 1 Hz to 500 Hz. Additionally, to achieve more uniform and larger volume diffuse plasmas [23], 12 pieces of high-voltage pole are surrounded the grounding pole, and the distance between high-voltage pole and the grounding pole is 4.0 cm, i.e., S is 4.0 cm. All these poles are made of straight bare copper wires. The selected copper wires are 1.0 mm in diameter and 20.0 cm in length. All the wires are parallel to each other.

The 3D view images of the cylindrical electrode configuration are shown in Fig. 1(c) and (d). It can be seen that an independent insulation supporting structure is installed on both sides of the cylinder, which is used to fix the relative positions of the high-voltage pole, the polyamide fabrics and the grounding. Meanwhile, the supporting structure might be rotatable at a certain speed, which would obtain more uniform treatment results. By the way, both the pulse generator and the reactor should be conjunctly grounded. The blue circles in Fig. 1(b) and (d) represent the PA66 fabrics being processed. Therein, the blue circles in Fig. 1(d) shows that layers of PA66 fabrics can be treated in the same time.

All of the experiments were at atmospheric pressure in air. The nanosecond pulse frequencies were set at 100, 300 and 500 Hz, respectively. The total treatment times were set at 4 s, 10 s, 20 s and 60 s, respectively. With the time and frequency varies, the corresponding pulse number may change from 400 to 30,000. Through the experiments of controlling variables, the influences of treatment time and pulse repetition frequency during the plasma processing will be studied.

2.3. Measurement for surface analysis

To observe surface microstructure changes of the processed PA66 fabrics, surface analysis were made by the X-ray photoelectron spectroscopy (XPS), using the Japanese Shimadzu-Kratos model AXIS-ULTRA DLD-600W X-ray photoelectron spectrometer with the Bi-anode Al/Mg X-ray source. All XPS-peaks were referred to the C1s signal at a binding energy of approximately 285 eV. Then the data were fitted by Gaussian–Lorentzian curves after removing Shirley background using XPSpeak software. In addition, the test of atomic force microscopy (AFM) was adopted too. The instrument was an atomic force microscope of Japanese Shimadzu model SPM9700.

The wicking effect experiment was made immediately after diffuse plasma treatment. Having the PA66 fibers hung on the retort stand. The water tank was filled with purified water colored red by organic dyestuff. The ends of treating fibers were immersed into the aqueous solution by 5 mm. From the very beginning of the immersion, the wicking time nodes were observed and recorded every rising 5 mm. Each time nodes were recorded until the top of the wicking solution stopped rising, meanwhile, the total wicking time was controlled within 4000 s.

3. Result and discussion

3.1. Mechanism of plasma generation

The side diagram of the cylinder is abstracted in Fig. 2(a). Fig. 2(b) gives the position of the PA66 fabrics being treated by the diffuse plasmas produced through one pair of wire electrodes. The side