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Effect of atmospheric pressure plasma treatment condition on adhesion of ramie fibers to polypropylene for composite

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ABSTRACT

In order to improve the interfacial adhesion between hydrophilic ramie fibers and hydrophobic polypropylene (PP) matrices, ramie fibers are modified by atmospheric pressure dielectric barrier discharge (DBD) plasma with our continuous ethanol flow technique in helium environment. A central composite design of experiments with different plasma processing parameter combinations (treatment current, treatment time and ethanol flow rate) is applied to find the most influential parameter and to obtain the best modification effect. Field emission scanning electron microscope (SEM) shows the roughened surfaces of ramie fibers from the treated groups due to plasma etching effect. Dynamic contact angle analysis (DCAA) demonstrates that the wettability of the treated fibers drastically decreases. Microbond pullout test shows that the interfacial shear strength (IFSS) between treated ramie fibers and PP matrices increases significantly. Residual gas analysis (RGA) confirms the creation of ethyl groups during plasma treatment. This study shows that our continuous ethanol flow technique is effective in the plasma modification process, during which the ethanol flow rate is the most influential parameter but all parameters have simultaneous influence on plasma modification effect of ramie fibers.

1. Introduction

As a result of increasing concerns in environmental protection and energy saving, vegetable fibers have been considered as potential alternative reinforcement fibers to man-made synthetic fibers in composites. Among these vegetable fibers, ramie fibers, known as "Chinese plant", are abundant in China [1], which have low cost, low density, similar specific strength and modulus compared to those of glass fibers [2]. On the other hand, ramie fibers also have disadvantages such as poor compatibility with hydrophobic polymer matrices [3], which limits its application as reinforcement in composites.

For many years, various chemical methods have been used to modify the composite interface to increase the compatibility between cellulose fibers and polymer matrices [3–10]. Although these methods are more or less effective, the ever more stringent legislation will little by little lead to the substitution of traditional wet-chemical techniques [11]. In recent years, some new energy-efficient techniques have been developed in cellulose modification [12–22], among which atmospheric plasma treatment has attracted much attention as a clean and high-efficient surface modification technology. Compared with low pressure plasma devices, the atmospheric pressure plasma systems do not require complicated and expensive vacuum system and therefore can be operated at low costs [23–25].

From our previous study, ethanol-soaked ramie fibers exhibited hydrophobicity and rougher surfaces after plasma treatment, enhancing interfacial adhesion between the fibers and polypropylene (PP) matrices [26]. Although ethanol pretreatment plus plasma treatment have been effective in surface modification of ramie fibers [27], one of the major drawbacks of this technology is the rapid evaporation of ethanol molecules before and during plasma treatment, making the treatment process less reproducible and controllable.

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Table 1
The plasma treatment parameters of the 15 treated groups.

Run	Factor 1 current (mA)	Factor 2 flow (sccm)	Factor 3 time (min)
1	10	5	5
2	10	5	9
3	11.8	3.2	2.6
4	8.2	6.8	2.6
5	10	2	5
6	11.8	6.8	7.4
7	8.2	6.8	7.4
8	8.2	3.2	7.4
9	10	8	5
10	13	5	5
11	7	5	5
12	11.8	3.2	7.4
13	10	5	1
14	8.2	3.2	2.6
15	11.8	6.8	2.6

In this paper, a mixture of ethanol vapor and helium gas with controllable composition was used as plasma treatment environment in a sealed dielectric barrier discharge (DBD) installation. A central composite design of experiments with treatment current (mA), treatment time (min) and ethanol flow rate (sccm) as variables was applied to study the joint influence of simultaneous interaction of the experimental parameters on the fiber surface modification effect.

The topology, chemical composition and wettability of the ramie fiber surfaces were characterized by field emission scanning electron microscopy (FESEM), X-ray photoelectron spectroscopy (XPS) and dynamic contact angle analysis (DCAA), respectively. The interfacial shear strength (IFSS) between ramie fibers and PP matrices was measured by microbond pullout test. The chemical composition in plasma treatment chamber was investigated by residual gas analysis (RGA).

2. Experimental

2.1. Materials

Ramie fibers were supplied by Mahua Technology Co., Ltd. (Suzhou, China) in the form of degumming fibers with a tensile modulus of 25 GPa. The diameters of the single fibers ranged from 15 to 40 μm. The PP matrix was provided by Shanghai Great Eastern Garden Chemical Fiber Co. Ltd. (Shanghai, China) in the form of a yarn composed of monofilaments with a Young's modulus of 1.75 GPa and a single fiber diameter around 50 um. The ethanol with 99.7% purity was obtained from Sigma-Aldrich Co., (Milwaukee, WI).

2.2. Sample preparation and plasma treatment

Before plasma treatment, all ramie fibers were cleaned with acetone and dried in a vacuum oven. Then the fibers were randomly divided into three groups: the control or untreated group containing fibers without any treatment, the DBD treated only group containing fibers only treated with helium (Purity ≥ 99.999%) plasma for 5 min, the DBD plus ethanol treated group containing fibers exposed to helium and ethanol plasma. In DBD plus ethanol treated group, preliminary experiments have been used to select the ranges of plasma treatment parameters. 15 treated groups with different combinations of treatment current, treatment time, and ethanol flow rate were selected according to a three-level factorial central composite design in order to evaluate the joint influence of the plasma treatment parameters on the modification effect of ramie fiber surfaces. Other parameters were kept

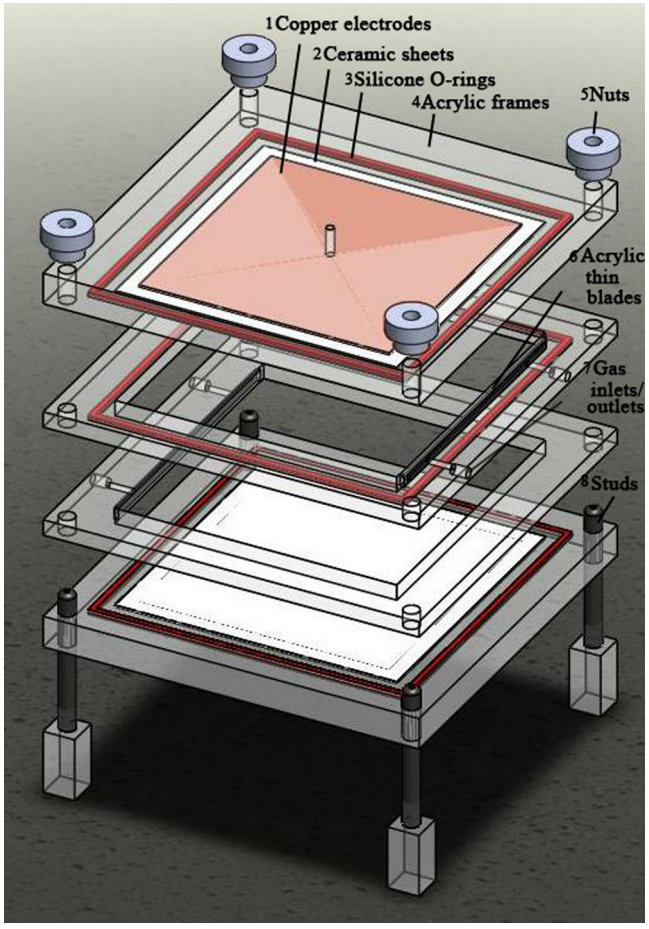

Fig. 1. Exploded view of DBD sealed reactor.

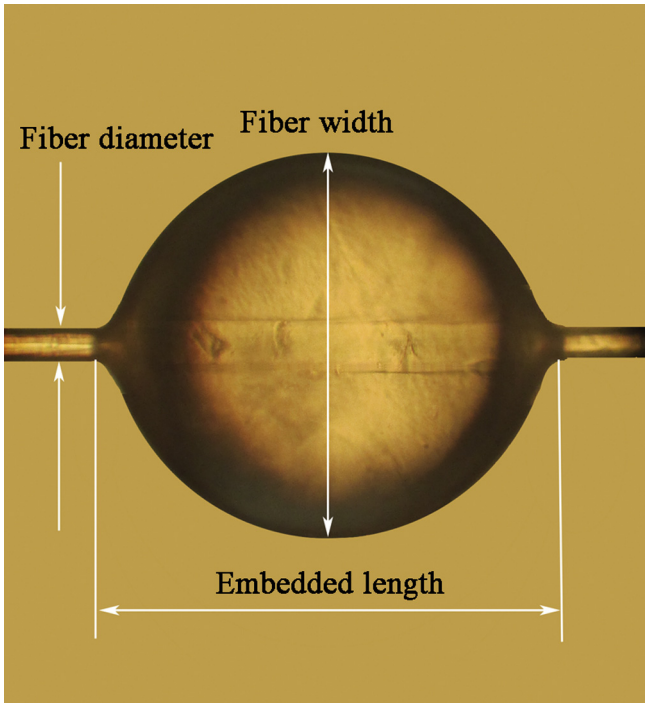

Fig. 2. Polarized light microscope photograph of a micro bead for the microbond pullout test.