Surface Modification of Polyimide Fibers by Oxygen Plasma Treatment

*You Wen（温友）, Xiangsheng Meng, Jingling Yan, Zhen Wang（王震）**

Polymer Composites Engineering Laboratory, Changchun Institute of Applied Chemistry（中科院长春应化所）, Chinese Academy of Science, Changchun Jilin, 130022, China）

** Corresponding author, Email: wangzhen@ciac.ac.cn*

1. Introduction

Aromatic polyimide (PI) fibers have great potential applications as reinforcements in the composite industry due to their low density, good mechanical and thermal properties, and excellent chemical resistance [1]. The interfacial properties between fiber and matrix play a crucial role in the overall performance of fiber-reinforced composites. Good interfacial adhesion facilitates the efficient load transfer from matrix to fibers, which can reduce internal stress concentration and improve overall composite mechanical properties. However, the surface of the PI fiber is relatively smooth and chemically inert, which lead to very poor interfacial adhesion between fiber and matrix [2-3]. Therefore, it is of great scientific and practical significance to modify the surface of PI fibers. In this work, we report the oxygen plasma modification of PI fiber.

2. Results and discussion

The surface properties of polyimide fibers were greatly impacted by their surface composition and roughness. As listed in Tables 1 and 2, the ratio of oxygen to carbon atoms (O/C), and the concentrations of the oxygen and nitrogen atoms, and –C–O– and –C–N– bonds increased, while the concentrations of the carbon atom and –C–C– bond decreased after the oxygen plasma treatment. The introduction of C-O and C-N bonds, and other functional groups can improve the adhesion between fibers and resins. The surface morphologies of pristine and treated fibers were characterized by scanning electron microscope (SEM), and the results were compared in Figure 1. The pristine PI fiber was smooth and 'clean'. However, spots, protuberances, and concave points were found on the fiber surface after exposed to oxygen plasma. The dynamic contact angles and surface free energy were determined to further evaluate the effect of plasma treatment on the surface properties of PI fibers. As listed in the table 3, after 120 W plasma treatment, the total surface free energy and its polar components of treated PI fiber increased from 37.28 to 70.63, and from 18.10 to 45.66 mN/m, respectively. These results indicated that the wettability and interfacial adhesion strength between fibers and resin matrix were greatly enhanced. The oxygen plasma treatment can incorporate the oxygen-rich groups onto fiber surface, which gave rise to the dipolar interaction and augmented the polar component. In addition, due to the effects of bombardment and etching by plasma treatment, the contact area and surface roughness were enlarged, which was beneficial for the dipolar and dispersion interaction.

The single-fiber tensile strength and interfacial shear strength were also measured for the pristine and treated PI fibers. As shown in Figure 2, treated PI fiber maintained 97% of its original tensile strength, while the interfacial shear strength of treated fiber was around 30% higher than that of pristine one. The increase of interfacial shear strength can be attributed to dipolar and dispersion interaction, as well as the increase of surface roughness and the contact area.

3. Conclusions

In summary, oxygen plasma treatment was investigated as a new technique for the surface modification of PI fibers. The results demonstrated that the adhesion between PI fibers and epoxy

resins, which was indicated by the increase of the surface free energy and interfacial shear strength, was greatly improved by oxygen plasma treatment. Furthermore, the treated fiber maintained 97% of its original tensile strength. Therefore, the treated fibers are more suitable for the preparation of high performance composites. This study provides new insights on how to modify PI fiber while maintaining its mechanical properties.

Acknowledgement Financial support from the National Science Foundation of China (NO. 51173178 and 21304093) is gratefully acknowledged.

References

1. Zhang Q, Luo W, Gao L, Chen D, Ding M, *J Appl Polym Sci*, **92**: 1653 (2004).
2. Brown J, Chappell P, George G, *J Mater Sci Lett*, **12**: 754(1993).
3. Tamargo-Martinez K, Martinez-Alonso A, Villar-Rodil S, Paredes J, Montes-Moran M, Tascon J, *J Chromatogr A*, **1218**: 3781 (2011).

Table 1. Atomic compositions of untreated and treated PI fibers.

Sample	C (%)	N (%)	O (%)	O/C
Untreated	77.84	0.66	21.50	0.28
Treated	69.97	3.66	26.36	0.38

Table 2. Percentages of chemical bonds of treated and untreated PI fibers

Sample	-C-C- (%)	-C-N- (%)	-C-O- (%)	-C=O (%)
Untreated	54.10	27.50	7.86	10.54
Treated	26.91	37.00	25.94	10.16

Table 3. The contact angles and surface free energies of treated and untreated PI fibers.

Samples	Contact angle (^{o})		Surface energy (mN/m)		
	Water	Diiodomethane	γ^p	γ^d	γ_{total}
Untreated	66.65 (2.83)	63.02 (1.94)	18.10	19.18	37.28
Treated	14.83 (1.28)	39.32 (2.04)	45.66	24.97	70.63

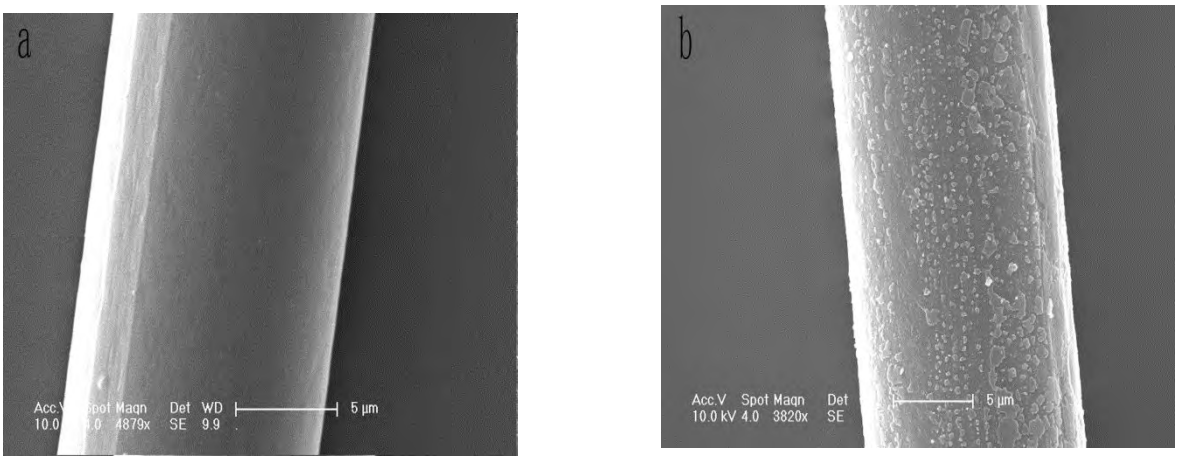
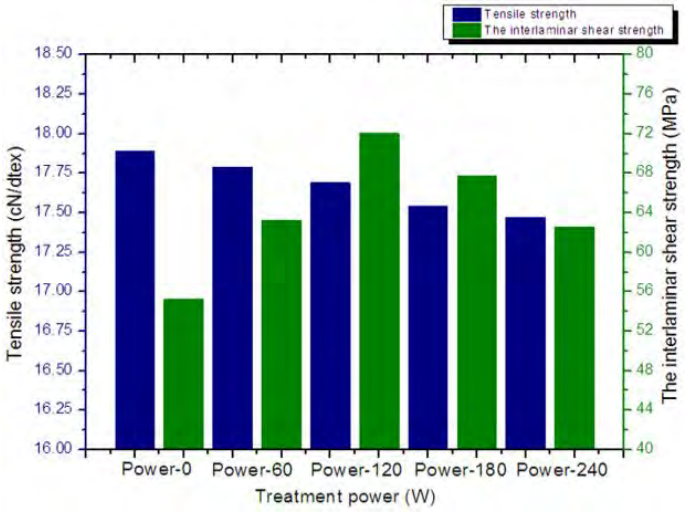


Figure 1. SEM images of treated and untreated PI fibers (a: untreated, b: treated).



.

Figure 2. The comparison of the single-fiber tensile strength and interfacial shear strength of treated and untreated PI