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To cite this article: Xinqiu Hong *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **439** 042011

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Dry-wet spinning of PVA fiber with high strength and high Young's modulus

Xinqiu Hong^{1,2}, Liming Zou^{1*}, Jiongxin Zhao¹, Chunlai Li¹, Longdan Cong¹

¹ State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, Donghua University, Shanghai, 201620, China

² College of Material and Textile Engineering, Jiaying University, Jiaying, Zhejiang, 314001, China

* E-mail: lmzou@dhu.edu.cn

Abstract. In this report, polyvinyl alcohol (PVA) fibers with high strength and high Young's modulus were prepared by dry-wet spinning, and they were characterized by FT-IR, XRD, TGA and XQ-1A yarn strength tester. The XRD measurement showed that PVA fiber had a single crystal type. The TGA results indicated the heat resistance of PVA fiber was better than that of PVA. The average tensile strength and Young's modulus of PVA fibers were 1.73 GPa and 40.02 GPa, respectively, their strength were high enough to meet the requirements for the use of civil and architectural.

1. Introduction

Civil and architectural materials are the material foundations of civil and architectural engineering, they are important cornerstones of promoting and guaranteeing social develop steadily [1]. Cement concrete, mortar and asphalt mixture are important components of civil and architectural materials [2], but they have obvious brittleness, easy cracking, poor durability and other problems [3], these cause serious deterioration of performance before the expected service life of civil and architectural engineering and affect the normal use [4]. At present, the international consistent method is adding high-performance fibers into cement concrete, mortar and asphalt mixture to improve their crack resistance and toughness [5].

High-performance polyvinyl alcohol (PVA) fiber has the advantages of high Young's modulus, strong adhesion, good dispersion and low price [6], so PVA fiber has a good application prospect in civil and architectural engineering materials [7]. However, the breaking strength of commercial PVA fibers is generally less than 1.6 GPa, that is not high enough to meet the requirements of civil and architectural engineering. Therefore, it is a great significance to research and develop PVA fiber with high strength and high Young's modulus, which can be industrialized.

In this paper, high-performance PVA fibers with break strength greater than 1.6 GPa were prepared by dry-wet spinning process by adjusting the concentration of spinning dope, the temperature of coagulation bath and multi-stage drafting.

2. Experimental

2.1. Materials

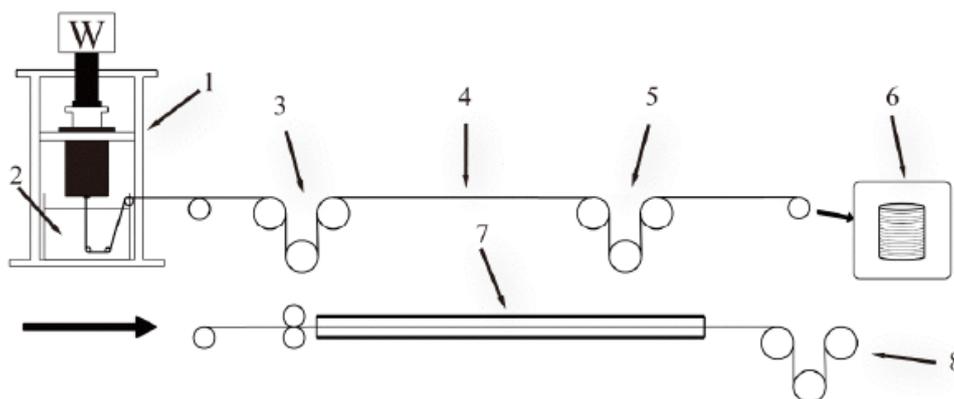


PVA (2400 of DP, 99% of alcoholysis) was received from Sinopec Sichuan Vinylon Works Group. Dimethyl sulfoxide was purchased from Shanghai Ling Feng Chemical Reagent Co., Ltd. All the other reagents were supplied from Sinopharm Chemical Reagent Co., Ltd. All reagents were analytical grade and used as received.

2.2. Fiber spinning

2.2.1. Preparation of PVA spinning dope. 20.0 g PVA and 80.0 g DMSO were put into a 250 mL single mouth flask containing a blender. After swelling for 1 hour at around 50 °C, the mixture was subjected to further stirring for 3 hours at around 100 °C. The desired PVA spinning dope was obtained after defoaming in an oven at around 80 °C for 24 hours.

2.2.2. Fiber spinning. PVA fiber was spun by self-designed spinning equipment (scheme 1). The preparation process of PVA fiber was as follows: (1) the spinning solution was added to the extruder and extruded through the spinneret at 70 °C by circulating water to form a fine flow of the fiber; (2) the fibers flowed through a 10 mm air layer, then flowed into the coagulation bath with anhydrous ethanol; (3) the fiber was drawn in the air at room temperature after it flowed out of the coagulation bath; (4) The air-stretched fiber was fully extracted in anhydrous methanol, and dried in an oven at around 50 °C; (5) The fiber was stretched in heat pipe at around 220 °C.



Scheme 1. Self-designed spinning equipment

(1-extruder; 2-coagulating bath; 3,5,8-roller; 4-stretching in air; 6-extraction; 7-stretching in heat pipe)

2.3. Characterization

Fourier transform infrared spectra (FT-IR) in the range of 500-4000 cm^{-1} were obtained by a Nicolet 8700 Fourier transform infrared spectrometer (Thermo Electron, American), and under the averaging of 32 scans at a resolution of 4 cm^{-1} . X-ray Diffraction (XRD) curves were acquired on a Powder X-ray diffractometer (Bruker, Germany), the scanning angle was 5-90 degrees. Thermogravimetric analyses (TGA) were performed by use of a TG 209 F1 Iris thermogravimetric analyzer (Netzsch, Germany), from room temperature to 550 °C, at a heating rate of 20 °C/min, under continuous nitrogen flow. Tensile strength and Young's modulus were measured on XQ-1A (China) yarn strength tester.

3. Results and discussion

3.1. FT-IR analysis

The FT-IR spectra (figure 1) were used to characterize the functional groups of PVA and PVA fiber. There are associative absorption peaks of -OH at 3374 cm^{-1} and 3371 cm^{-1} in the spectrum of PVA and PVA fiber, respectively. The higher degree association is, the wider absorption peak is, and the lower wave number will be moved, the stronger hydrogen bond is. It means that hydrogen bond of PVA

fiber is stronger than that of PVA. There are stretching vibration peaks of $-CH_2$ at 2940 cm^{-1} and 2939 cm^{-1} in the spectrum of PVA and PVA fiber, respectively.

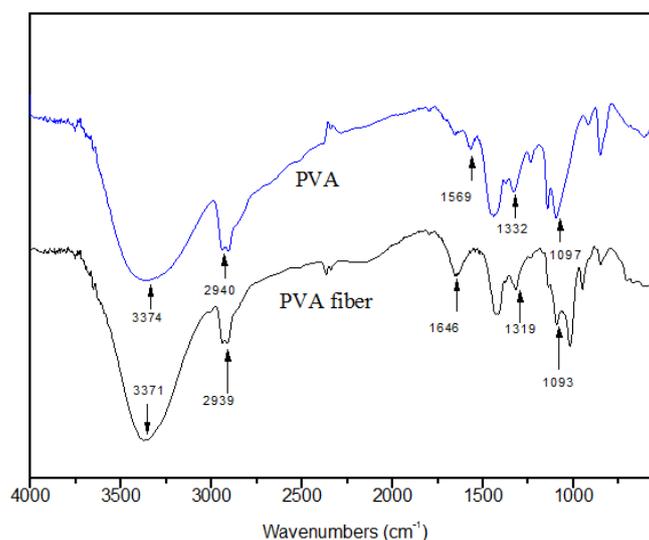


Figure 1. FT-IR spectra of PVA and PVA fiber

3.2. XRD analysis

The XRD curves (figure 2) were used to characterize the crystallization of PVA and PVA fiber. There are obvious diffraction peaks at the diffraction angle of 19.17° in the both curves of PVA and PVA fiber. However, there is another peak at 40.47° in the curve of PVA. These indicate that after spinning and stretching, the crystallinity of PVA fiber increases, and the crystal type also develops toward to a single type.

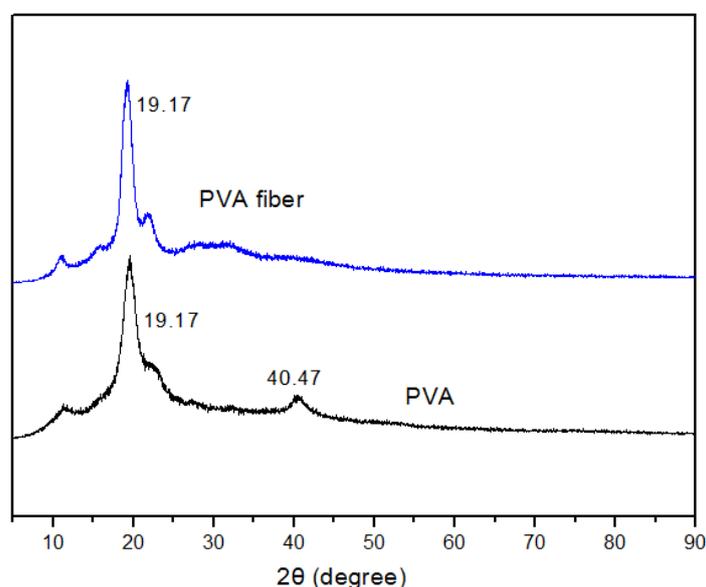


Figure 2. XRD curves of PVA and PVA fiber

3.3. TGA analysis

The thermogravimetric curves of PVA and PVA fiber are divided into three stages: the first stage, before 230°C , the quality of PVA and PVA fiber decrease due to the volatilization of free water and bound water; the second stage, between 250°C and 450°C , the quality decrease sharply due to decomposition of PVA and PVA fiber; the third stage, after 450°C , as decomposition is end, the

quality of PVA and PVA fiber remain stable. In the second stage, the decomposition curve of PVA fiber laged behind that of PVA, because PVA fiber has higher crystallinity and better orientation than PVA, which make the thermal properties of PVA fiber better than PVA.

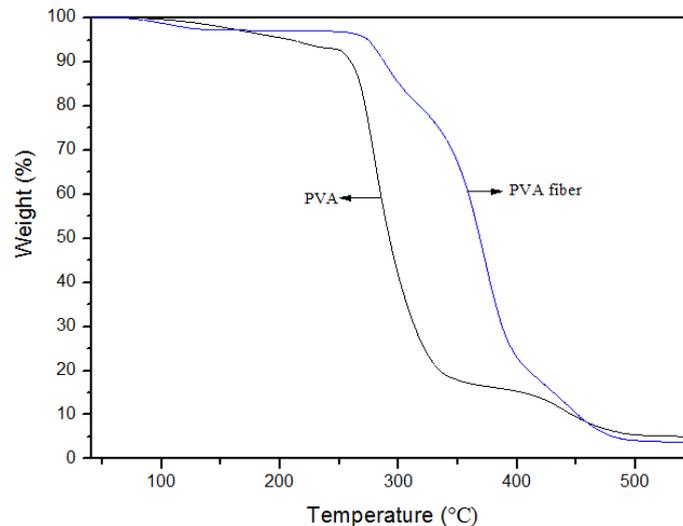


Figure 3. TGA curves of PVA and PVA fiber

3.4. Tensile strength

The tensile curves of PVA fibers are shown in figure 4, the average tensile strength and Young's modulus are shown in table 1. The average tensile strength and Young's modulus of PVA fibers are 1.73 GPa and 40.02 GPa, respectively. The results greatly exceed PVA/MWCNTs 20 wt% composite fiber, which was spun by gel spinning, their tensile strength and Young's modulus are 0.21 GPa and 10.98 GPa, respectively [8].

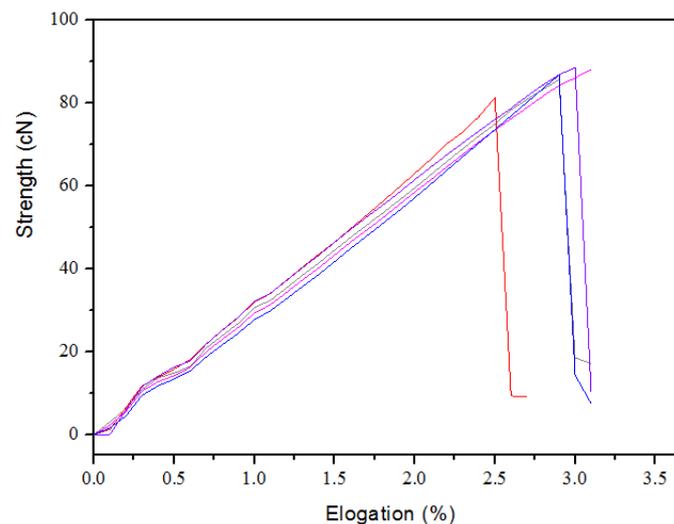


Figure 4. Tensile curves of PVA fibers

Table 1. Mechanical properties of PVA fiber

Sample	Tensile strength (GPa)	Young's modulus (GPa)	Elongation (%)
PVA fiber	1.73	40.02	2.88

4. Conclusion

In summary, PVA fiber with high strength and high Young's modulus were prepared by dry-wet spinning process. The XRD measurement shows that the crystal type of PVA fiber is a single type. The TGA results indicate that the heat resistance of PVA fiber is better than that of PVA. More significantly, the average tensile strength and Young's modulus of PVA fibers are much higher than PVA/MWCNTs composite fibers, their strength are high enough to meet the requirements for the use of civil and architectural.

Acknowledgments

The authors would like to acknowledge the research center for analysis & measurement of Donghua University. This work is supported by National Key R&D Program of China (2016YFB0303200).

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