

# Poly(ethylene-2,6-naphthalate) microfiber prepared by carbon dioxide laser-thinning method

Akihiro Suzuki \*, Masaya Tojyo

*Interdisciplinary Graduate of School of Medicine and Engineering, University of Yamanashi, Takeda-4, Kofu 400-8511, Japan*

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## Abstract

Poly(ethylene-2,6-naphthalate) (PEN) microfiber was continuously obtained by using a carbon dioxide (CO<sub>2</sub>) laser-thinning method. As a winding speed increased, the fiber diameter decreased, and its birefringence increased. When the PEN microfiber, obtained by irradiating the laser operated at a power density of 9.15 W cm<sup>-2</sup> to the original fiber supplied at 0.33 m min<sup>-1</sup>, was wound up at 1594 m min<sup>-1</sup>, the obtained microfiber had a diameter of 2.8 μm, a birefringence of 0.174, tensile modulus of 5.4 GPa, and a tensile strength of 0.36 GPa.

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## 1. Introduction

Microfibers are very valuable from the viewpoint of industrial and medical materials and are now manufactured with highly skilled techniques, such as a conjugate spinning (requiring a highly complex spinneret), islands-in-a-sea fiber spinning, melt blowing, and flash spinning [1–4].

A laser-thinning method developed by us could easily produce microfibers by irradiating a continuous-wave carbon dioxide (CO<sub>2</sub>) laser to fibers, such as PET [5], nylon 6 [6], nylon 66 [7], i-PP [8], and poly(L-lactic acid) [9] fibers. The apparatus used for the laser-thinning method could wind up monofilament microfibers in the winding speed range of 100–2500 m min<sup>-1</sup>. The CO<sub>2</sub> laser-thinning can be

applied to almost all thermoplastic polymers, and then their microfibers can be easily obtained.

Poly(ethylene-2,6-naphthalate) (PEN) is one of high temperature semicrystalline polymers that combine the properties of superior chemical resistance, flame resistance, and mechanical strength. Its mechanical properties, glass transition temperature, and melting point are higher than those of PET.

It is the purpose this paper to optimize the condition for producing a PEN microfiber and to discuss the superstructure and mechanical properties of PEN microfibers obtained.

## 2. Experimental

### 2.1. Material

The original fiber used in this study was an as-spun PEN fiber supplied by Teijin Ltd. and had

\* Corresponding author.

E-mail address: [a-suzuki@yamanashi.ac.jp](mailto:a-suzuki@yamanashi.ac.jp) (A. Suzuki).



Fig. 1. Wide-angle X-ray diffraction pattern of PEN original fiber.

a diameter of 230  $\mu\text{m}$ , a birefringence of  $0.18 \times 10^{-3}$ , and an intrinsic viscosity of  $0.63 \text{ dL g}^{-1}$ . The original fiber was amorphous and isotropic as shown in Fig. 1.

## 2.2. Measurements

The SEM micrograph of microfiber was taken with a JSM6060LV (JEOL Tokyo, Japan) with an acceleration voltage of 4 kV. Before the observation, the sample was coated with gold using a sputter coater. The fiber diameter was obtained by using an imaging analyzer.

A birefringence was measured with a polarizing microscope equipped with a Berek compensator (Olympus Optical Co., Ltd., Tokyo, Japan).

A wide angle X-ray diffraction (WAXD) image of the microfiber was taken with an imaging-plate (IP) film and an IP detector R-AXIS DS3C (Rigaku Co., Akishima Japan). The IP film was attached to a X-ray generator (Rigaku Co., Akishima, Japan) operated at 40 kV and 35 mA. The radiation was Ni-filtered Cu  $K\alpha$ . The sample-to-IP film distance was 65 mm. The microfibers were exposed for 30 min to the X-ray beam from a pinhole collimator with a diameter of 1.0 mm.

The differential scanning calorimetry (DSC) measurement was carried out using a THERM PLUS 2 DSC 8230C calorimeter (Rigaku Co., Akishima, Japan). The DSC scans were performed in the temperature range of 25–300  $^{\circ}\text{C}$ , using a heating rate of 10  $^{\circ}\text{C min}^{-1}$ . All DSC experiments were carried out under a nitrogen purge. The DSC instrument was calibrated with indium.

A thermal shrinkage was measured with a THERM PLUS TMA8310 (Rigaku Co., Akishima,

Japan) at a heating rate of 5  $^{\circ}\text{C min}^{-1}$ . The measurements were performed in the temperature range from 25 to 270  $^{\circ}\text{C}$ . The specimens (15 mm long) were given a very small tension (5 mN) to stretch the specimen tightly.

Tensile properties were measured at 23  $^{\circ}\text{C}$  and a relative humidity of 50% with EZ Graph (Shimadzu Co. Ltd., Kyoto Japan). A gauge length of 20 mm and elongation rate of 10 mm  $\text{min}^{-1}$  were used. The experimental results are the average of 10 measurements.

## 2.3. CO<sub>2</sub> laser-thinning apparatus

The CO<sub>2</sub> laser-thinning apparatus used to continuously produce the microfiber consisted of spools supplying and winding the fiber, a continuous-wave CO<sub>2</sub>-laser emitter, a system supplying the fiber, an optical system, and a traverse as shown in Fig. 2. The continuous-wave CO<sub>2</sub> laser emitted at 10.6  $\mu\text{m}$ , and the laser beam was a 4.0 mm diameter spot. The optical system to circularly irradiate the laser to the original fiber was composed of four mirrors. The laser power was measured with a power meter during the laser-irradiation. It was necessary to supply the fiber to a laser-irradiation point at a constant speed to prepare the microfiber in a stable manner. The supplying system pulled the original fiber from the supplying spool and supplied it to the laser-irradiation point at a constant speed. The supplying

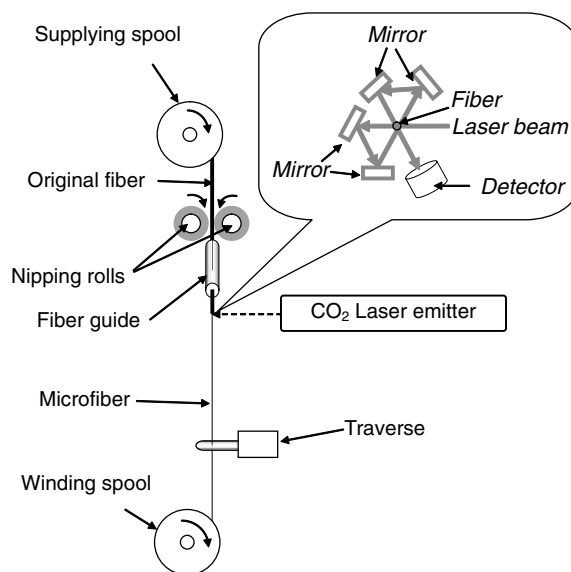


Fig. 2. CO<sub>2</sub> laser-thinning apparatus used for producing the microfiber.

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