

Poly(ethylene terephthalate) nanofibers prepared by CO₂ laser supersonic drawing

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ABSTRACT

Poly(ethylene terephthalate) (PET) nanofibers were prepared by irradiating a PET fiber with radiation from a carbon dioxide (CO₂) laser while drawing it at supersonic velocities. A supersonic jet was generated by blowing air into a vacuum chamber through the fiber injection orifice. The flow velocity from the orifice was estimated by computer simulation; the fastest flow velocity was calculated to be 401 m s⁻¹ at a chamber pressure of 6 kPa. A nanofiber obtained using a laser power of 8 W and a chamber pressure of 6 kPa had an average diameter of 193 nm and a draw ratio of about 900,000. This technique is a novel method for producing nanofibers.

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1. Introduction

Nanofibers have been produced by electrospinning [1–9], melt electrospinning [10–12], sea-island-type conjugated melt spinning, single-orifice melt blowing, [13] and jet blowing [14]. Out of these methods, electrospinning is the most widely used for preparing nanofibers. Poly(ethylene terephthalate) (PET) nanofibers have been prepared by electrospinning [15] and PET/nylon 6 sea-island conjugated melt spinning [16]. Electrospun PET nanofibers were prepared by spraying a solution of PET–trifluoroacetic acid onto a metallic collector under a high voltage. A PET nanofiber with a diameter of less than 100 nm was formed by PET/nylon 6 sea-island conjugated melt spinning together with laser-heated flow drawing. In this method, the melt-spun fiber was drawn at a draw ratio of 174 by laser heating, and the PET nanofiber was formed by removing the nylon 6 sea component from the drawn conjugated melt-spun fiber.

Recently, we proposed a new approach for producing nanofibers. It involves irradiating a fiber with radiation from a carbon dioxide (CO₂) laser while drawing it at a supersonic velocity. A supersonic jet was generated by blowing air into a vacuum chamber through the orifice used to inject the fiber into the vacuum chamber. The adiabatic expansion of air across the orifice

cools the jet. The fiber is instantly melted by the high-power laser beam that irradiates the cold supersonic jet. It is then tremendously deformed by the shear force generated by the supersonic flow, and ultradrawn to a draw ratio of the order of 10⁵. We named this preparation method *CO₂-laser supersonic drawing*. Supersonic drawing has already been applied to poly(L-lactic acid) (PLLA) [17] and poly(ethylene-2,6-naphthalate) [18], and in both cases nanofibers were obtained without using any solvent or without removing the second component. The nanofiber obtained by supersonic drawing is basically endless nanofiber because a fiber supplied at a constant speed was continuously laser-irradiated.

The CO₂ laser has been applied to drawing and annealing of various fibers because it can rapidly and uniformly heat fibers. We used the CO₂ laser to draw and anneal fibers and to prepare microfibers. A CO₂ laser-thinning method producing microfibers was previously applied to PET [19], PLLA [20,21], nylon 6 [22], isotactic polypropylene [23] fibers, and their microfibers were obtained, but no fiber of 1 μm or less in diameter was produced by the CO₂ laser-thinning method. Other groups also proposed drawing methods by using the CO₂ laser, but the CO₂ laser drawing methods including our method could not produce nanofibers by using only CO₂ laser-irradiation to fiber [24–26]. On the other hand, the CO₂-laser supersonic drawing can easily prepare various nanofibers by only CO₂ laser-irradiation without combining any process.

In this paper, we prepare PET nanofibers by CO₂-laser supersonic drawing.

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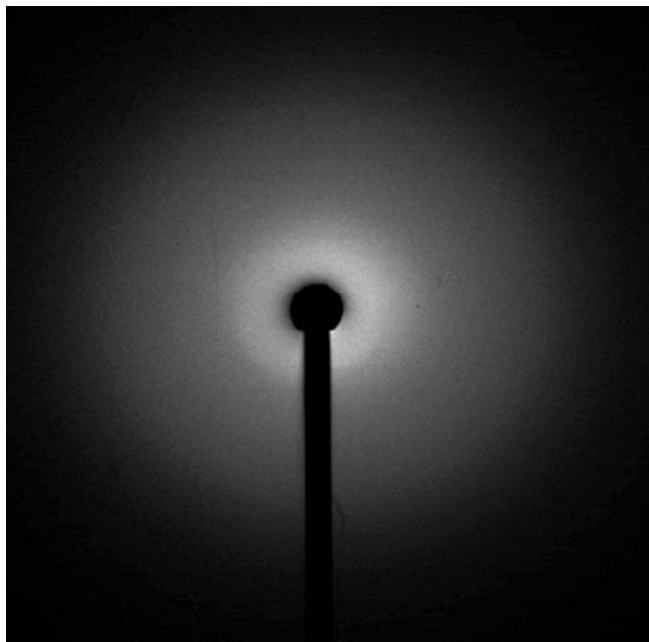


Fig. 1. Wide-angle X-ray diffraction pattern of the original PET fiber.

2. Experimental

The PET fiber used in the present study was prepared from a commercial-grade PET pellet by using a lab melt spinning machine. The as-spun PET fiber had a diameter of $183\ \mu\text{m}$ and a birefringence of 0.27×10^{-3} . It was almost amorphous and isotropic (see Fig. 1). Number- and weight-average molecular weights of this fiber were 3570 and 8380, respectively.

The morphology of the produced nanofiber was determined by scanning electron microscopy (SEM) (JSM-6060LV, JEOL Ltd., Japan) using an accelerating voltage of 10 kV. Before SEM observation, the

samples were coated with gold using a sputter coater. The average diameter and the diameter distribution of the nanofiber were measured using an imaging analyzer. The average diameter of a fiber was determined by averaging the diameters measured at 100 locations in a webbed fiber.

The weight-average molecular weight (M_w) and number-average molecular weight (M_n) were measured by gel permeation chromatography (GPC-14, Waters Co.). GPC analysis was performed using two 30 cm gel columns (Shodex HFIP-806M). The measurements were carried out at a column temperature of $40\ ^\circ\text{C}$ in hexafluoroisopropanol and a flow rate of $0.5\ \text{mL/min}$. A differential refractive-index detector was used as the detector.

Differential scanning calorimetry (DSC) measurements were conducted using a calorimeter (Therm Plus 2 DSC 8230C, Rigaku Co.). The DSC scans were performed within the temperature range $25\text{--}280\ ^\circ\text{C}$ at 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 using indium.

Fig. 2 shows the apparatus used for the CO_2 -laser supersonic drawing. It consists of a spool to supply the fiber, a continuous-wave CO_2 laser with an output wavelength of $10.6\ \mu\text{m}$ and a maximum power of 8 W, an acrylic vacuum chamber with Zn–Se windows and a 0.5-mm diameter orifice for injecting the fiber, a power meter, a movable platen, and a vacuum pump. The vacuum chamber was placed on the movable platen that consists of a micro-alignment stage and a laboratory jack that can be moved parallel to the Y and Z axes allowing the laser irradiation point on the fiber to be finely adjusted.

The velocity distribution from the orifice and the force exerted on the fiber in the air jet were estimated by performing fluid analysis using a three-dimensional (3D) finite element method (FEM) with ANSYS® CFZ 11.0 software.

3. Results and discussion

When performing CO_2 -laser supersonic drawing using an air jet, it is very important to determine the velocity distribution in the air

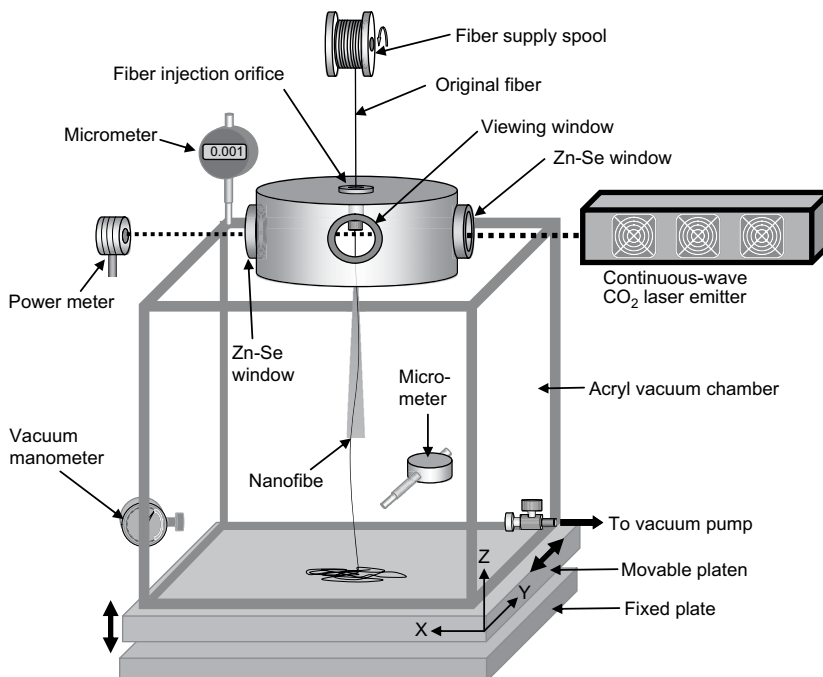


Fig. 2. Schematic diagram of apparatus used for CO_2 -laser supersonic drawing.

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