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Study on Nanofiber Spinning Using Centrifugal Force —Rotation Speed of Fiber-spinning Disk vs Nanofiber/Microfiber Diameter when Disk Speed is Increased via Gears—

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In recent years, the electrospinning method has been studied extensively. However, there is a disadvantage with respect to the amount of solvent being used for producing nanofibers; most of the solvent will be evaporated into the atmosphere leading to a very earth-unfriendly situation. In this study, we attempt to spin nanofibers/microfibers without using solvents in question; alternatively, physical centrifugal force is used. For that purpose, a new experimental device has been designed and fabricated. As the material spun, we chose polypropylene (PP), which was heated by a rotating disk via an induction heater (IH) and sprayed out from a microsize nozzle onto the disk rotated by centrifugal force. PP fibers are formed when molten PP is released from the nozzle while being simultaneously cooled by air. Fabricated PP fibers are observed by scanning electron microscopy (SEM). The maximum relative centrifugal force of a rotating disk was 67000 G in a previous report in which a belt driving system was employed [Z. Shichang and H. Noguchi: Proceedings of Seikei-Kakou Annual Meeting (2012) 181]. In the present work, the rotation speed of the disk is increased by a gear driving system, in which the centrifugal force of the rotating disk is increased to 100000 G, for the centrifugal melt spinning equipment. Eventually, we applied the present nanofiber/microfibers as a good material for fabricating thin filters for collecting, sensing, and subsequent evaluation of the characteristics of PM2.5 generated in the exhaust gas of gasoline-fueled automobiles because of their superior resistance to various chemicals.

1. Introduction

In recent years, several noteworthy nanofiber fabrication methods such as self-assembly, phase separation, and electrospinning methods have been developed.(1–6) The last method involves high-voltage application to a polymer solution or molten polymer to obtain extremely thin fibers, and has been extensively studied because the method can produce nanofibers relatively easily, thus leading to their practical use in the fabrication of nonwoven fabrics. However, electrospinning cannot produce polypropylene (PP) fibers because PP cannot be dissolved in a solvent.

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Given these circumstances, we have developed a new method of spinning molten plastic nanofibers/microfibers using physical centrifugal force, which not only eliminates the use of indispensable yet very earth-unfriendly organic solvents for the electrospinning method, but also enables a very low cost production of fibers with excellent antichemical properties. In this new method, a small-diameter aperture is first drilled into a disk, and a plastic pellet is charged within the disk. Then, the disk is heated with an induction heater (IH) to melt the plastic pellet. Then, the disk is rotated at a high speed using centrifugal force to obtain energized and molten nanofibers/microfibers that are being cooled while being spun; thus, finally, the desired nanofibers are obtained. Furthermore, this new method is very attractive from an industrial viewpoint, compared with other conventional spinning methods, because it continuously provides spun fibers, which is desirable for mass production.

In an actual experiment, the most important factor in the molten spinning of nanofibers is fiber diameter, which is dependent on the viscosity of the molten plastic pellet, centrifugal force, and nozzle diameter. Furthermore, in this study, the PP resin pellet, which is difficult to dissolve by the electrospinning method, is chosen as the test material.

2. Target of the Present Study

We previously reported on the centrifugally rotated equipment, in which pulley belts are used to rotate the disk. However, this structure caused marked disk vibration when the motor was driven at a high rotation speed owing to the rattling of pulley belts.[7] Thus, in the present study, we separated the motor shaft from the disk rotating at a high speed. Although our previous equipment could stably yield thin 3-μm-diameter spun fibers if rattling did not occur, improvement is desired to obtain thinner nanofibers.

Accordingly, in the present work, we fabricated a disk to be centrifugally rotated at a very high speed using gears instead of a belt system, thus enabling us to obtain submicron-diameter fibers stably. Eventually, we increased the peripheral disk rotation speed up to near-sonic speed, and the results of this increased rotation speed will also be reported in the following.

3. Experimental Equipment

In the following, we will describe in detail the process of fabricating the vitally important experimental equipment for forming submicron-diameter spun fibers by centrifugally driven rotation of a disk.

3.1 Overview of experimental equipment

Figure 1 shows an overview of the newly fabricated fiber-spinning equipment including four pillars, a blower to air-cool the motor, and a wire-net collector for collecting the formed fibers. The main structural key points here are as follows. First, a high-speed motor is used as a power supply with which the rotation speed is changed by controlling the impressed voltage. Second, chromium–molybdenum steel SCM440 (JIS) is used to fabricate two disks

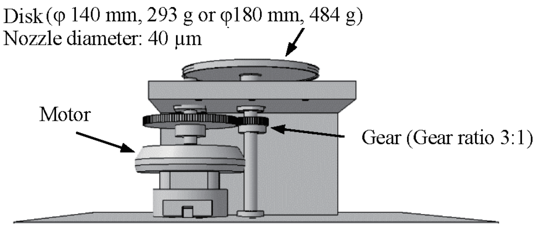

Fig. 1. Overview of fiber-spinning equipment (weight: 20 kgf).

of different diameters that must be sufficiently durable against high rotation speeds. Third, a metal gear system was used to increase the disk rotation speed, and finally, the total weight of the equipment was suppressed to 20 kgf to prevent its rattling.

3.2 Power supply motor

Table 1 shows the specifications of the motor of the spinning equipment; Nichidou-kougyou's extra vacuum cleaner motor NVC-S35L was used.

3.3 Fabrication of gear system

In this study, two kinds of induction-hardened gear were used: the number of gears was 120 and the number of pinions was 40, thus giving a multiplying gear ratio of 3:1.

3.4 Ceramic ball bearing

Since the present heated disk is rotated at extremely high speeds to continuously yield polypropylene resin micron-scale-diameter fibers, it is imperative to provide a rattling-suppression method by which the shaft of the disk can endure high temperatures and/or centrifugal force so as to maintain a high precision of the said shaft. Thus, in this study, we chose ceramic bearings to support the shaft. Silicon nitride ceramic ball bearings (KOYO high-temperature-clean-pro bearing®, Catalogue No. SE6203ZZSTPRBC3YS) were used.

3.5 Fabrication of disk

Figure 2(a) shows a top view of a disk made of JIS SCM440 installed in the equipment shown in Fig. 1. Since this is a vital element for the present experiment to be successful, we requested first-grade Japanese lathe Meistersinger craftsmen to fabricate them; the eccentricity of the disk could be suppressed to within 4 µm after assembly.

Figure 2(b) shows that the 500-µm-thick wall was reduced to 200 µm thickness using a hand grinder (Leutor®) applied from inside of the disk. Subsequently, a nozzle with an aperture of

Table 1
Motor specifications.

Maker	Nichido-kougyo
Rated voltage	100 V
Rated power consumption	1200 W
Rated frequency	50 Hz
Maximum speed	35000 min^{-1}

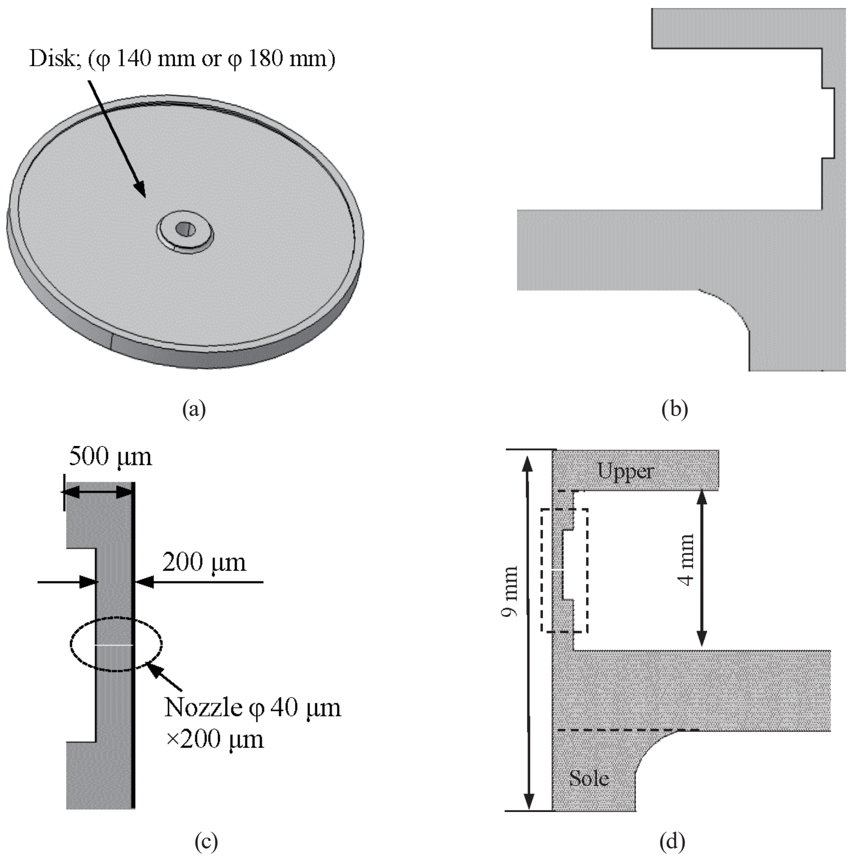

Fig. 2. Structure of the disk. (a) Top view of fiber-spinning disk, (b) detailed view of the disk, (c) enlarged view of the nozzle, and (d) cross-sectional view of the disk.

⌀40 µm was pierced through the wall using a microdrill having a cutting edge diameter of 40 µm and a cutting edge length of 300 µm. An enlarged view of this nozzle portion is shown in Fig. 2(c).

From our experience in the previous study, we noted that maintaining the balance of the disk to suppress rattling and thus maintain the horizontal attitude of the disk is very important. Thus, we chose to provide a "sole" under the disk, as shown in Fig. 2(d), so that the upper and lower portions of the disk have the same weight.

4. Experimental Procedure

We insert 2 g of PP pellets into the disk and then switch on the IH to heat the entire disk to 180 °C or higher to melt the PP pellets. After the pellets become molten, the disk is rotated while recording the temperature of the disk via an infrared-ray thermometer. Then, the molten PP solution is accelerated simultaneously with the rotating disk making it adhere onto the disk. When the PP solution reaches a constant speed, the solution comes out of the nozzle and finally cools to below the ambient temperature to solidify into fibers. Eventually, microfibers are formed, and when the fiber-spinning speed becomes equal to the disk peripheral speed, the formed microfibers are extruded from the disk at the peripheral speed of the disk. The extruded solid nanofibers are observed by scanning electron microscopy (SEM).

5. Experimental Results

5.1 Performance evaluation of the present disk without IH

Figure 3 shows the relationship between the impressed AC voltage (set at 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 V) and the motor rotation speed under a no-load condition. Figure 4 shows the relationship between the impressed AC voltage set at the same values as above and the motor rotation speed under the condition of loading ⌀140 and ⌀180 mm disks. Figure 5 shows the relationship between the motor speed N (rotations per min) and the disk circumferential speed V ($=\pi dN/60$; d is disk diameter). Figure 6 shows the relationship between the angular velocity ω or frequency of the disk and the centrifugal force G ($=r\omega^2$, r is the radius of the disk) acting onto it in the rotating equipment shown in Fig. 7 for which the IH was not yet used.

Although not shown, a centrifugal force of 4750 G could not produce microfibers; the reason might be that such a centrifugal force is smaller than the surface tension of molten PP liquid. In contrast, when the centrifugal force was 18121 G, we successfully produced spun microfibers.

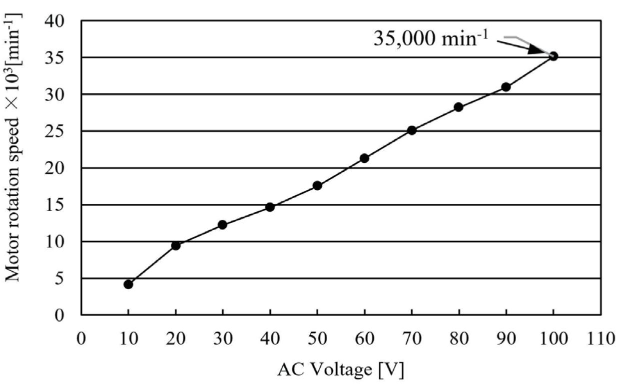

Fig. 3. Input voltage and rotation speed of motor.

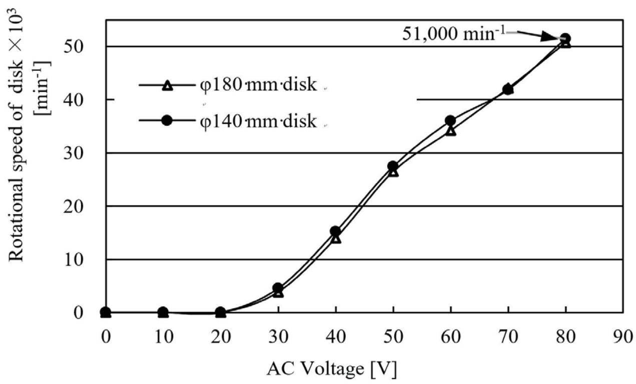

Fig. 4. Input voltage and rotation speed of the disk.

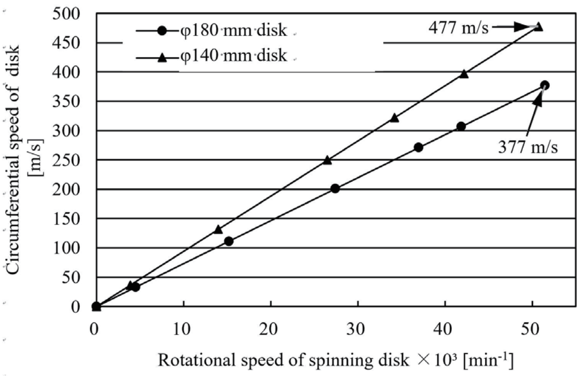

Fig. 5. Rotation and circumferential speed of disk.

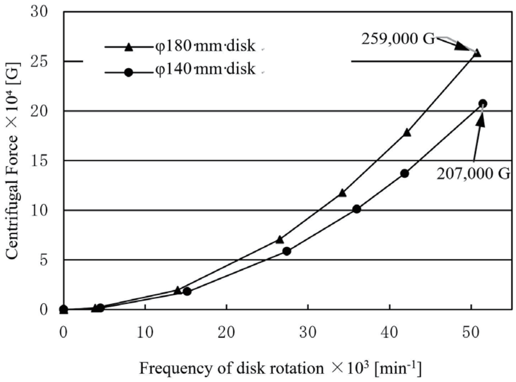

Fig. 6. Rotation speed/frequency ω of the disk and centrifugal force.

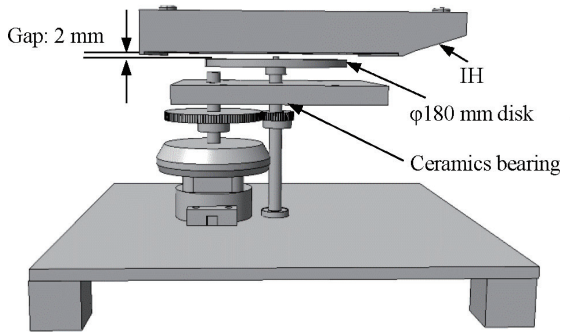

Fig. 7. Whole view of the present spinning equipment with IH.

5.2 Detailed description of disk and IH

In the present experiment, we chose an inexpensive and temperature-controllable electromagnetic IH available on the market (Koizumi-seiki Co., Ltd; Catalogue No. KIH1402R, consumption power = 300–1400 W) to achieve uniform heating of the disk. We also increased the disk diameter from the previous 100 to 140 mm or more, because a disk with a diameter of 100 mm could not be recognized by the IH as the target to heat.

As the next step in the experiment, since we wanted to determine the necessary electric power for heating the disk, we investigated the relationship of the distance/gap between the IH and the disk with the power consumption, and show the results in Fig. 8. In the figure, we can see a decrease in power consumption in proportion to the gap size.

Figure 9 shows the relationship between the rotation speed of the disk heated by the IH and the temperature of the disk at a gap of 2 mm. We can clearly see that the temperature of the disk can be maintained at 200 °C or more even while the disk rotates.

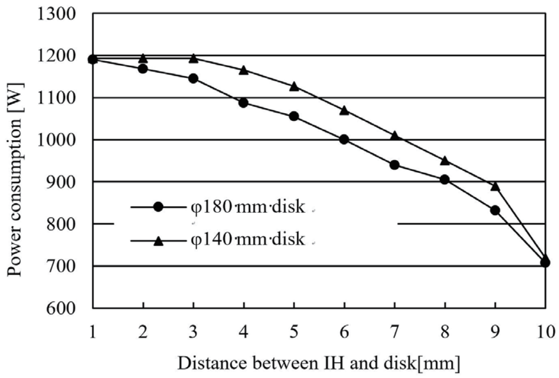

Fig. 8. Relationship of distance/gap between IH and disk with power consumption.

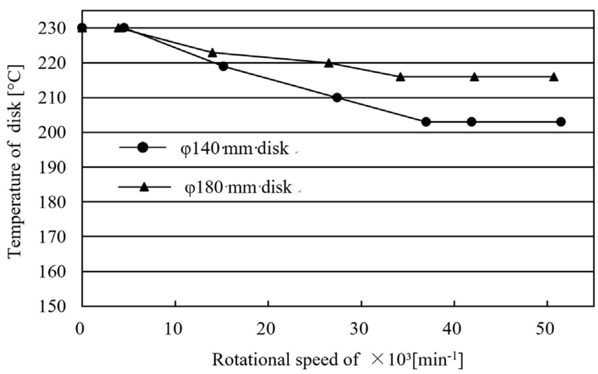

Fig. 9. Relationship between rotation speed/frequency of the disk and temperature of the disk (gap: 2 mm).

Figure 10 shows the relationship between radiated heat expressed as temperature of the disk and the duration of disk rotation after being heated to 200 °C. The disk temperature was measured when the disk was at a standstill at rotations of 30000, 40000, and 50000 min^{-1}. As can be seen clearly, the temperature of the disk abruptly decreases at the start of disk rotation. This suggests that the maximum IH heating power may be necessary to keep the disk temperature sufficiently high while it rotates at a high speed, since the wind flow generated may cool the disk.

5.3 Effects of heating temperature of rotating disk (in Fig. 1) on uniformity of microfiber diameter

Although not shown, in the previous study in which the disk was directly heated with a gas burner flame, not only did the plastic pellets sometimes burn or decompose, but it was also difficult to achieve uniform heating throughout the entire disk. The heating process was further made difficult as the flame itself could be easily extinguished when the disk rotated at an extremely high speed. In contrast, because the disk can be heated uniformly when using the IH shown in Fig. 7, undesirable pyrolysis of the PP pellets did not occur even when the disk was rotated for 30 min. Furthermore, we found that the disk could be maintained at a constant temperature for a long period.

Figure 11 shows a SEM image of the PP microfibers spun using the previous equipment with pulleys at a centrifugal force of 67000 G without rattling. As can be seen, uniform microfibers could be produced. As can be seen in Fig. 12, the average fiber diameter was found to be as small as 3.3 μm, and the fibers themselves were found to be formed uniformly as well.

Figure 13 shows the external appearance of another fiber sample obtained using the new equipment shown in Fig. 1, in which an even higher centrifugal force of 86000 G was applied for a shorter period of time, i.e., 2 min. The calculated average diameter of the fiber was 1.9 μm. However, since in the previous work a metal bearing was employed for the disk shaft, resulting in excessive heat generation and rattling vibration, further production of spinning fibers was difficult.

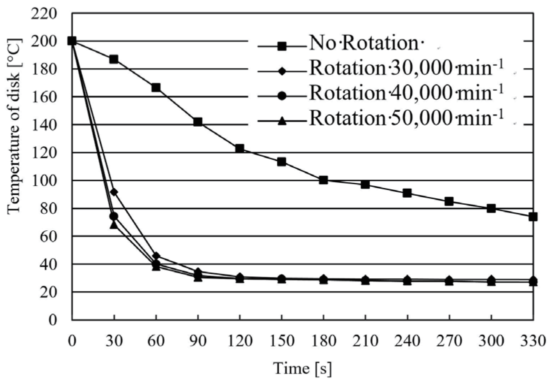

Fig. 10. Relationship between rotation time and temperature of disk (gap: 2 mm).

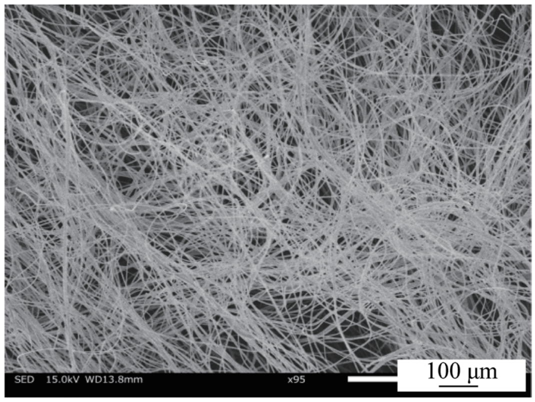

Fig. 11. SEM image of PP microfibers melt spun from the ⌀40 µm spinning nozzle in Fig. 7. (Duration: 15 min, electric power: 800 W)

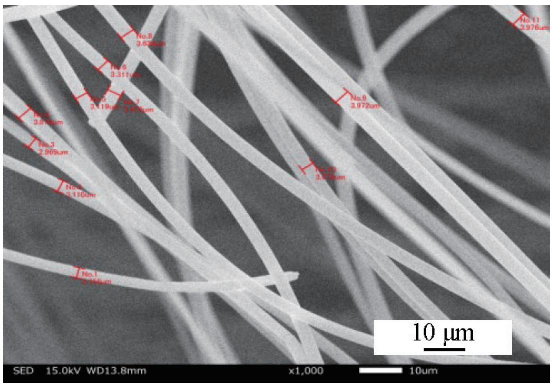

Fig. 12. (Color online) Enlarged SEM image of PP microfibers shown in Fig. 11.

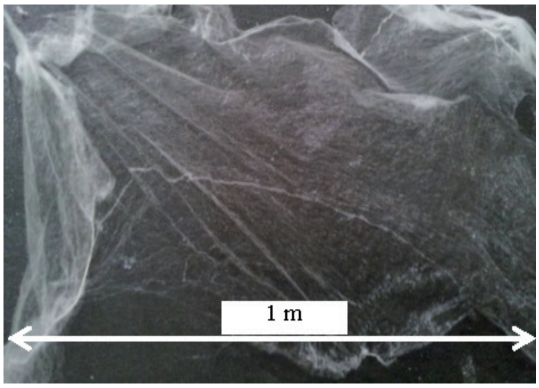

Fig. 13. Top view of microfibers (average diameter: 1.9 µm) melt spun at 86000 G for 2 min (Disk temperature: 200 °C).

5.4 Effects of circumferential speed of rotating disk with gear system and ⌀140 and ⌀180 mm disk on microfiber diameter

Figure 14 shows the relationship between the circumferential speed of the disk and the diameter of fibers produced. As shown clearly in the figure, even at the same disk circumferential speed, a larger-diameter disk (⌀180 mm) yielded a smaller fiber diameter. It can be assumed that this 180-mm-diameter disk can be rotated at a subsonic or sonic speed to yield submicron-diameter fibers.

Figure 15 shows a SEM image of the centrifugally spun nanofibers using the ⌀180 mm disk rotated at 50714 min^{-1} (circumferential speed: 477 m/s, centrifugal force: 259000 G). The average diameter of spun fibers was calculated to be 595 nm.

In melt spinning, the fabricated fiber diameter is normally around 10–15 µm; thus, it will be necessary to increase the yarn drawing speed to reduce the fiber diameter. Furthermore, the

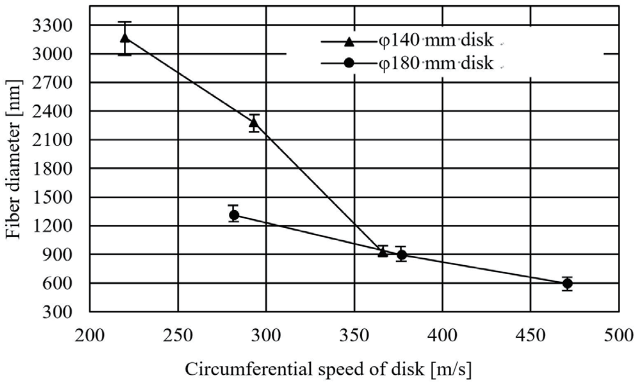

Fig. 14. Circumferential disk speed vs fiber diameter.

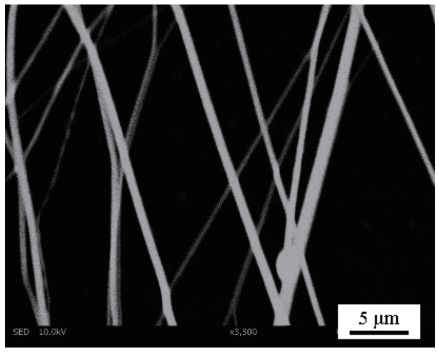

Fig. 15. Perspective view of nanofiber (average diameter: 595 nm) melt spun at 259000G (circumferential disk speed of 477 m/s) for 5 min.

nozzle aperture diameter normally cannot be made smaller than 50 μm because, in that case, the strength of the nozzle itself may decrease. Typically, the yarn speed is from as low as 5 m/min to as high as 50 m/min at most. However, the yarn speed of the present study is at around 500 m/min, leading to the realization of nanofibers without sacrificing the strength of the nozzle itself.

6. Conclusions

We successfully spun PP microfibers without the need for an environmentally unfriendly organic solvent. A centrifugally driven disk rotated continuously at an extremely high circumferential speed, with its temperature controlled using a commercially available IH. Continuous spinning operation could be performed for more than 30 min.

The following results were obtained.

(1) By devising a fiber-spinning disk structure that not only prevented the rattling of the disk, allowing a marked increase in rotation speed, but also enabled stable heating of the disk even at extremely high rotation speeds, we realized the stable production of microfibers of approximately 3 μm diameter.

(2) The above structure was achieved partly by rotating the disk with a gear-driven system instead of pulley belts so as to prevent the undesirable rattling of the disk, and partly by employing an IH that enabled the uniform heating of the whole disk even when the disk was rotated at extremely high circumferential speeds.

(3) With the combination of the countermeasures described above, we realized a maximum centrifugal force of 259000 G at a circumferential disk speed of 477 m/s, while the disk temperature was maintained at 200 °C.

(4) Eventually, submicron PP nanofibers with an average diameter of 595 nm were obtained, which was the initial target of our present study.

(5) The said PP-related nanofibers, which have superior resistance to chemicals, can be used to make thin filters for collecting, sensing, and subsequent evaluation of the characteristics of PM2.5 generated in the exhaust gas of gasoline-fueled automobiles. Because the flow resistance through the filter is low leading to good capture of PM2.5 as the filter sensor; the sensor could be efficiently subjected to chemical analysis.

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References

1 F. Rombaldoni, K. Mahmood, A. Varesano, M. Bianchetto Songia, A. Aluigi, C. Vineis, and G. Mazzuchetti: Surf. Coat Technol. **216** (2013) 178.
2 Y. Yamashita, F. Ko, A. Tanaka, and H. Miyake: J. Text. Eng. **53** (2007) 137.
3 S. Ito, J. Nakamura, A. Obata, T. Kasuga, and H. Hirata: Phosphorus Res. Bull. **26** (2012) 109.
4 Z. K. Nagy, A. Balogh, B. Démuth, H. Pataki, T. Vigh, B. Szabó, K. Molnár, B. T. Schmidt, P. Horák, G. Marosi, G. Verreck, I. Van Assche, and M. E. Brewster: Int. J. Pharm. **480** (2015) 137.
5 Y. Kishimoto, T. Kobashi, H. Morikawa, and Y. Tamada: J. Silk Sci. Technol. Jpn. **25** (2017) 49.
6 A. Balogha, A. Domokosa, B. Farkasa, A. Farkasa, Z. Rapia, D. Kissb, Z. Nyiric, Z. Ekec, G. Szarkad, R. Örkényia, B. Mátravölgyia, F. Faigla, G. Marosia, and Z. Kristóf Nagya: Chem. Eng. J. **350** (2018) 290.
7 M. Shahhosseininiaa, S. Bazgirb, and M. D. Jouparic: Mater. Sci. Eng., C **91** (2018) 502.

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