SUBMICRON POLYPROPYLENE FIBERS FROM THE MELT

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Introduction

Electrospinning was invented more than a century ago [1]. Thermoplastic synthetic polymers were synthesized more than a half century ago. A number of research efforts have been directed towards developing fine fibers – with diameters less than a micrometer – from the melt [2-13]; however, success has been limited especially for commodity polymers with melting temperatures above about 100°C. Successful processing of thermoplastic semicrystalline polymer into useful fiber requires control of a number of parameters, arguably most importantly the temperature of the fiber as it emerges from the spinneret. It comes as no surprise, therefore, that our efforts to melt electrospin fine fibers, which lose heat even more quickly than large fibers, required a heated "quench" chamber. Our apparatus, which produced fibers with mean diameters of PP less than a micrometer, used coaxial needles, with heated air flowing slowly in the annual region between the needles, coupled with heated air flowing laminarly downward, in the direction of spinning. The heated air presumably delayed quiescent crystallization of the charged jet by facilitating self-attenuation of the low viscosity polymer melt in the substantial electric field.

Experimental

Materials

Our work featured polypropylene, PP, a commodity polymer used in a plethora of applications; 80,000 – 85,000 g/mole molecular weight polypropylene was supplied by Exxon Mobil. The PP we used was designed for meltblowing, so it has a low melt viscosity. The melt flow rate is 1.2 kg/10 min at 230°C.

Apparatus

We designed and constructed an apparatus for melt electrospinning thermoplastic polymers. PP was the first polymer we attempted to electrospin with a melting temperature above 100°C; the observed melting temperature of PP is about 165°C.

The apparatus had a source of dry air, which was split into two streams. Each stream had a flow control valve and a resistance heater set a distance from the electrospinning unit so not to disturb the electric field between needle and plate. One line of hot air was used to melt the polymer. Some of that hot air was bled into the annulus between the concentric needles, sizes 14 and 18. We could meltblow with the air; however, in the experiments and results described in this paper, the air flow was insufficient to blow a drop of molten PP off the needle tip in the absence of an applied electric field. The hot air flowing through the annulus kept the viscosity of the PP sufficiently low that it traveled down the lumen.

The other stream of hot air led to the quench chamber. The air was introduced into the square cross–sectional chamber from the top through a diffuser. Hot air, cooling as it moved downward in a laminar fashion, provided the desired temperature profile. Fig. 1 is a schematic of the apparatus' design.

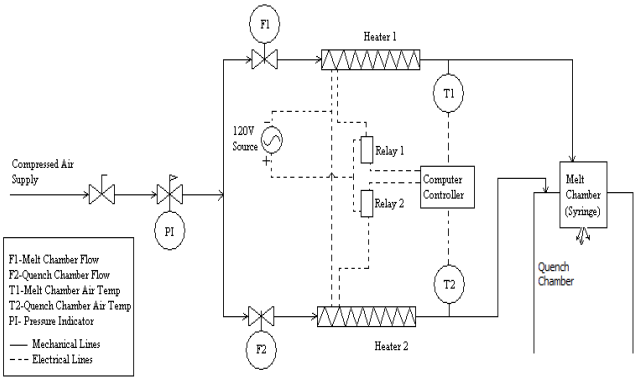

Fig. 1: Piping and Instrumentation Diagram [14]

The power supply, syringe pump and other components were typical of electrospinning apparatuses [7]. We estimate that the freeze point,

110°C, the quiescent crystallization temperature of PP, was perhaps about 7 cm below the needle tip, well after the jet had turned sideways from the poorly-named "whipping" instability. Thus, the jet had much more time to attenuate in the melt than did melt electrospun jets expelled into room temperature air. We assume, but did not measure, that the fibers we collected had low molecular orientation.

Procedures

The experimental conditions for the four trials are shown in Table I. These were our only trials, so they do not necessarily represent optimal conditions; they were simply our best guesses at the outset based largely on meltblowing and electrospinning experience.

Table I: PP Electrospinning Parameters

	Trial 1	Trial 2	Trial 3	Trial 4
Syringe Control Temp. (°C)	190	190	190	190
Quench Control Temp. (°C)	125	125	125	125
Air Flow to Syringe (SCFH)	25	25	25	25
Air Flow to Quench (SCFH)	25	25	25	25
Collection Distance (m)	0.076	0.063	0.076	0.063
Applied Voltage (kV)	15	15	17	17
Nominal Field (V/m)	197	236	223	268

Results and Discussion

The results of the trials are summarized in Table II. Three of the four trials lead to mean fiber diameters less than a micrometer.

Table II. Electrospun PP Fiber Data

	Trial 1	Trial 2	Trial 3	Trial 4
	(µm)	(µm)	(µm)	(µm)
Mean Fiber Diameter	0.82	0.92	0.83	1.02
Std. Dev.	0.25	0.19	0.17	0.22

A representative SEM of the web from trial 4 is shown in Fig. 2. The rather uniform fibers have smooth surfaces.

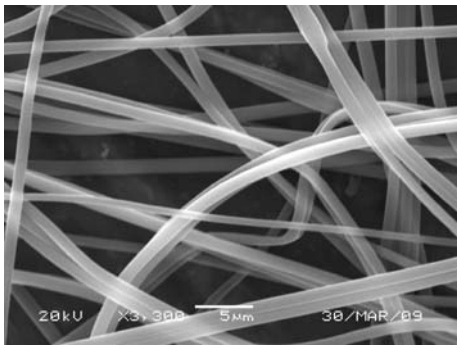

Fig. 2: SEM of Melt Electrospun PP from Trial 4

Conclusions

We have successfully melt electrospun PP into fibers with a mean diameter of less than one micrometer. Concentric needles allowed the polymer melt to remain fluid in the capillary until jet ejection. Delayed quenching insured the jet was sufficiently warm that viscosity remained low and crystallization was delayed, facilitating attenuation.

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