Ensemble Laser Diffraction for Online Measurement of Fiber Diameter Distribution During the Melt Blowing Process

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Abstract

Online measurements of the fiber diameter distribution during a melt blowing process were taken using a new laser diffraction technique. This technique measured both the attenuation of the fibers as well as entanglement of the fibers into bundles at large distances from the die. A pilot scale unit with a 20.3 cm (8 inch) slot die was used for the studies. Commercial polypropylene polymer was used. Both the spinline attenuation and fiber bundling were measured as a function of position both below and across the die face.

Keywords

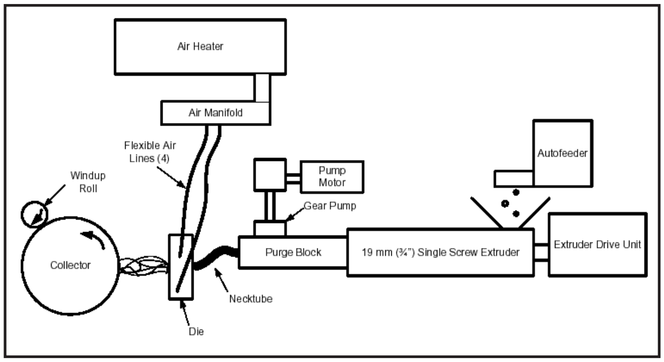
melt blowing, fiber spinning, nonwovens, fiber diameter distribution, ensemble laser diffraction

Introduction

Melt blowing is a process where heated air jets are used to attenuate molten polymer streams into fibers. These fibers are then collected for either direct use or further processing. A schematic of melt blowing equipment is shown in *Figure* 1. The key piece of equipment is the die, below which the heated air and polymer make contact. Because the fibers are directly laid down as a web on the collection device (with no weaving or knitting required), the material is classified as a nonwoven. Nonwovens are a large and growing industry: in 1997 nonwovens producers in the United States had sales in excess of 3.8 billion U.S. dollars (Marlow-Ferguson, 2001). Melt blown fibers are commonly used for filtration, personal hygiene, and absorption applications.

Melt blown fibers exhibit a statistical distribution in their diameters due to the somewhat chaotic process of high-speed attenuation with air. Since size distribution largely determines the finished web properties, the measurement and control of size distribution is of great industrial importance. Ensemble laser diffraction (ELD) provides a method for measuring fiber diameter distribution both while the fiber formation process is still taking place (online) and after the fiber has been formed (offline). Online measurements are important for understanding, controlling and modeling the melt blowing process. The online measurement capability provided by ELD can only currently be matched using either laser Doppler velocimetry (LDV) or high-speed photography (and related particle imaging techniques). Both LDV and photographic techniques have limitations in their application to the melt

Figure 1
DIAGRAM OF THE MELT BLOWING PROCESS USED IN THIS WORK.



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blowing process. In LDV, the measuring volumes, which are typically 1 mm in diameter, are difficult to effectively target on a rapidly vibrating fiber stream. Since LDV only measures the velocity of the fiber, diameter data must be acquired through mass balances. These mass balance calculations require a model for the polymer density that makes the use of LDV a somewhat empirical technique for analyzing multi-hole melt blowing. The work of Bansal and Shambaugh (1996) describes the process of determining polymer density of a filament during the fiber formation process. High-speed photography has its own limitations that include (a) proper illumination by flash, lasers, or other means; (b) obtaining correct depth of field; and (c) resolving the small diameter fibers from either chemical or digital photographs. Yin et al. (1999) used a high-powered pulse laser to provide the necessary illumination for digital imaging. However, even with this sophisticated equipment, online diameter determination was only possible very near the die.

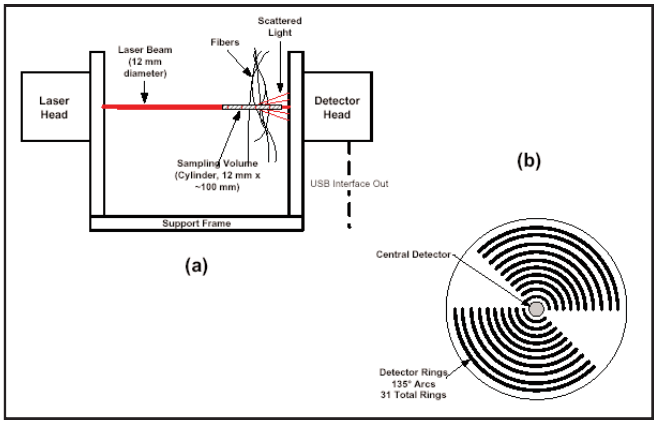
The work described herein applies ELD to the online measurement of fiber diameter during the melt blowing process. This ELD method provides data that are difficult to obtain through other methods. Furthermore, ELD provides fiber distributions in near real-time, without interfering with the fiber formation process.

EXPERIMENTAL METHODS

Ensemble Laser Diffraction

Ensemble laser diffraction (ELD) works by passing a collimated laser beam through a group of fibers and measuring the scattering of the transmitted light. The radial scattering profile is directly related to the diameter distribution of the fibers present within the sampling volume. The sampling volume for the FibrSizr unit (Powerscope, Inc., Minneapolis, MN) is a cylinder that is 12 millimeters in diameter and up to 200 millimeters in length. The sample, either a fiber mat or fiber stream, can lie in almost any orientation relative to the sampling volume. *Figure* 2 illustrates the use of the FibrSizr unit. The scattering of the light is measured using a central sensor and a series of concentric sensor arcs. Primarily based on the Mie scattering model (Mie, 1908), the forward scattering of the light is used to measure the fiber diameter distribution of a given sample. Similar technology has already been applied to particle sizing applications (Black et al., 1996). The calculation scheme based on the scattering data is iterative; this iterative process is depicted in *Figure 3*. The raw scattering data are taken at the detector, and then the data are sent to a computer for analysis. A multiple regression technique is used to compute what fiber diameter distribution would produce the observed laser scattering. Since this method is based on the accuracy of the scattering model, calibration is necessary. The manufacturer of the unit conducted an extensive calibration of the scattering model wherein the ELD measurements were verified by both scanning electron microscopy (SEM) and optical microscopy; see Fandrey and Naqwi (2003). These researchers found that the laser scattering technique gave very good agreement with both SEM and optical measurements.

Figure 2
THE FIBRSIZR UNIT:
A) OVERALL DIAGRAM OF THE UNIT;
B) DETECTOR ARRAY



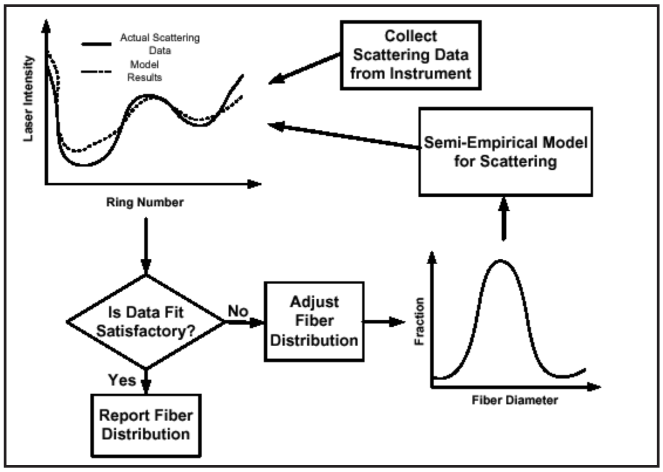


Figure 3
THE CALCULATION SCHEME USED FOR DETERMINING FIBER DIAMETER DISTRIBUTIONS

Die and Extruder Unit

The experiments were conducted on a pilot scale melt blowing line at the 3M Nonwovens Technology Center in St. Paul, Minnesota. An eight-inch wide slot die of drilled design was used in this line; see *Figure 4*. The die was oriented horizontally such that the fiber curtain was parallel to the ground. (The fiber curtain is the assembly of fibers that travel between the die and the collector.) The die had 101 capillaries spread evenly across the central 4 inches of the die; air flowed through the entire 8 inches of the die width. Electrical heating was used to control the die temperature. Each polymer capillary had a diameter of 0.015 inches. Compressed air for blowing was routed first through an electric heater, and then through a four hose manifold, and finally to the die itself. Airflow was measured using Pitot sensors in the air lines, and airflow was controlled with a pressure regulator. Polymer pellets were melted and pressurized using a 19 mm ($^3/_4$ inch) Brabender® extruder. The molten polymer was then fed to a

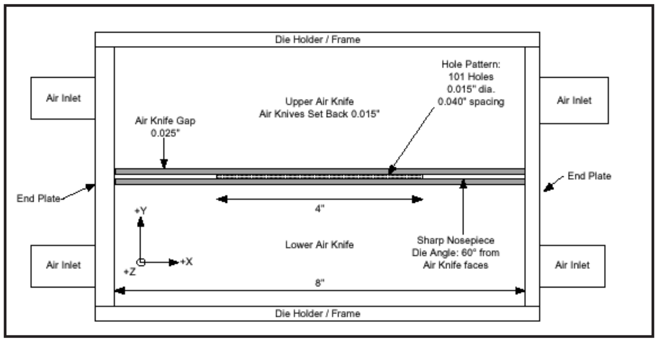


Figure 4
VIEW OF THE FACE OF THE MELT BLOWING DIE. THE +Z DIRECTION IS PERPENDICULAR TO THE FACE, AND THE ORIGIN OF THE COORDINATE SYSTEM IS AT THE CENTER OF THE DIE FACE

gear pump that provided accurate polymer flow control. Polymer flow was measured by timed collection and weighing of fibers from the die. Fibers were collected on a solid drum collector that was placed 1 meter from the die face. The polymer used for the experiments was Fina Dypro® 3860 isotactic polypropylene with a nominal melt flow index of 100.

Operating Conditions

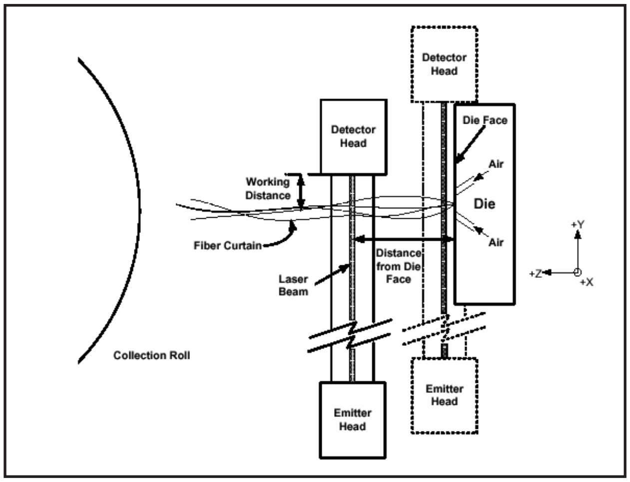
Line operating conditions were chosen to be representative of normal melt blowing. The polymer rates used were 1.13, 2.27, and 4.54 kg/hr (2.5, 5, and 10 lb/hr). Airflow rates used were 2500 and 3900 standard liters per minute (SLPM.) The die temperature was held at 300°C, while the air heater was set to 420°C. There were significant air temperature losses in the air lines leading to the die. The air heater temperature was set such that the air temperature at the die was approximately 300°C. The die configuration did not allow for direct measurements of the air temperature inside the die. However, measurements were taken of the air temperature of the jets at the die discharge; this temperature was 300°C.

Fiber Diameter Measurements

The laser scattering unit used for the ELD measurements in our experiments was the Powerscope Fibrsizr. The unit consisted of a laser and a detector attached to a "U" frame and mounted on a movable stand; see *Figure 5*. Raw scattering data were transferred to a computer for analysis through a USB interface. Scattering data fitting and analysis were performed using included proprietary software. The instrument was located such that the detector head was as close as possible to the fiber stream without interfering with the process. The distance between the detector and the fibers is the working distance. This working distance (measured in the y direction in *Figures 4 and 5*) must be kept as small as possible in order to improve the accuracy of the measurements. Since the distance between the detector and the emitter is about 1 meter, there are not any clearance problems associated with the emitter interfering with the fibers. At large working distances, the ELD technique will not resolve fibers of the smallest diameters. However, when only large diameter fibers are present in the sample, the working distance can be increased. At large distances from the die (large +z positions as shown in *Figures 4 and 5*), the working distance used was approximately 7.6 cm (3 inches). This working distance was small enough to resolve fiber diameters down to the instrument's lower limit of 1.5 microns. Due to physical clearance issues around the die, when the distance from the die (z position) was less than 8.9 cm (3.5 inches), the working distance was increased to approximately 20 cm (8 inches), which was just sufficient to clear the die; see the dotted lines in *Figure 5*. With the increased working distance, measurements were taken as close as 0.64 cm (0.25 inches) from the die (z position); the die face itself interfered with closer measurements. Measurements taken at z = 8.9 cm (3.5 inches) from the die – using both 7.6 and 20 cm working distances – showed no difference due to using a larger working distance. The compensating factor is that, near the die, the fibers have larger diameters, and, hence, there is no need to measure smaller diameters.

Measurements were taken in two directions. First, the fiber diameter profile was measured by moving the ELD assembly in the z direction while keeping the assembly in the x=0 plane (i.e., the center of both the die face and the fiber curtain). These data provided both a measure of fiber attenuation and an approximation of fiber bundling. Second, measurements were taken across the width of the die (in the x direction) in order to determine the uniformity of the fiber diameter profiles across the width of the die.

Figure 5
ORIENTATION OF FIBRSIZR UNIT RELATIVE TO THE MELT BLOWING PROCESS
THE DISTANCE BETWEEN THE EMITTER AND COLLECTOR IS ABOUT 1 METER, AND THE WORKING DISTANCE IS OPTIMALLY ABOUT 7.6 CM. HOWEVER, NEAR THE DIE FACE, THE WORKING DISTANCE MUST BE INCREASED TO ABOUT 20 CM BECAUSE OF CLEARANCE PROBLEMS (THE DOTTED LINES SHOW THE FIBRSIZR™ UNIT IN THIS SITUATION).



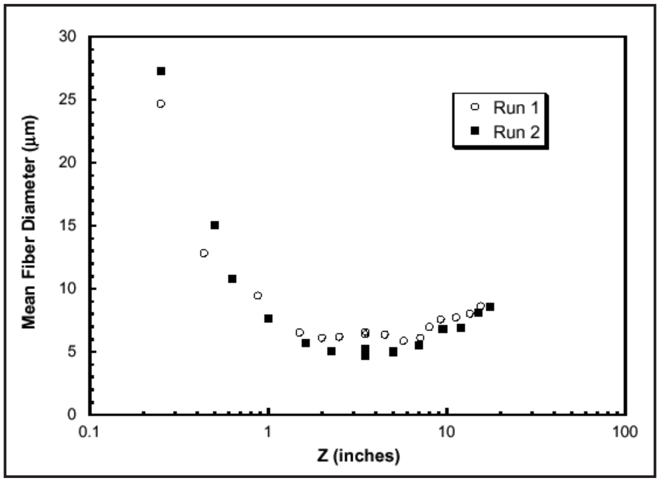


Figure 6
MEAN DIAMETER PROFILES OBTAINED FROM TWO SUCCESSIVE RUNS AT 2.27 KG/HR (5 LB/HR) POLYMER FLOW, 300°C DIE TEMPERATURE, AND 2500 SLPM AIRFLOW

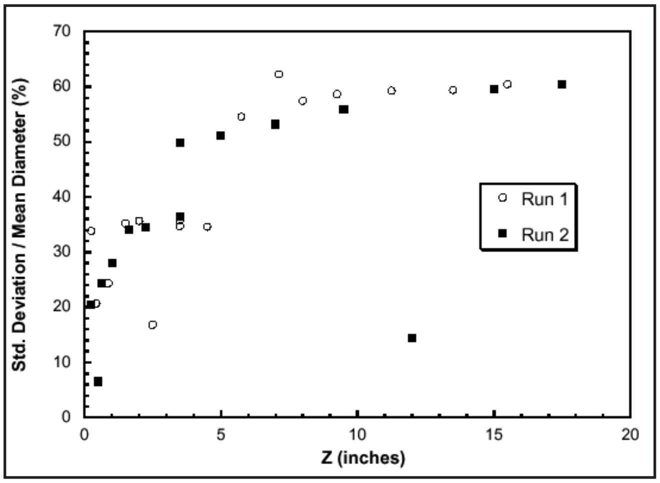


Figure 7
PROFILES OF DIAMETER STANDARD DEVIATION OBTAINED FROM TWO SUCCESSIVE RUNS AT 2.27 KG/HR (5 LB/HR) POLYMER FLOW, 300°C DIE TEMPERATURE, AND 2500 SLPM AIRFLOW

RESULTS AND DISCUSSION

Reproducibility Test

The first set of experiments was designed to evaluate the repeatability of the ELD (ensemble laser diffraction) method. Process flows were held constant at 2.27 kg/hr (5 lb/hr) of polymer and 2500 SLPM of air. Measurements were taken at increasing distances (z positions) from the die face and at the center (x=0 plane) of the fiber curtain. The mean diameter profiles determined by two typical replicate measurements for these process conditions are shown in *Figure* 6, and the standard deviations are shown in *Figure* 7. The standard deviation is an output of the ELD instrument. The standard deviation represents the width of the distribution of all fibers contained in the ELD measuring volume. As *Figure* 6 illustrates, the mean diameter data were highly reproducible from measurement to measurement, with typical differences being less than two microns. *Figure* 7 shows that the standard deviation is also reproducible from run to run (but not to the same degree as mean diameter). One important feature of *Figure* 6 is the apparent increase in mean diameter at distances (z positions) farther than 2.5 inches (6.3 cm) from the die. Since melt blown fibers are always expected to attenuate (have diameter reduction) and then reach constant diameter as the distance from the die increases, the increase shown on *Figure* 6 is attributed to bundling of fibers as they progress away from the die. In comparing *Figure* 6 with typical fiber attenuation profiles (e.g., see Bansal and Shambaugh, 1996), it appears that fiber attenuation has ceased at about z = 2.5 inches. Then, in our multi-hole system, fiber bundling begins to cause the mean diameter to increase. Of course, if bundling begins to take place at z values where attenuation is still occurring, then bundling may compete with fiber attenuation to cause a shift (to a smaller z value) in the minimum diameter shown on *Figure* 6. Fiber bundling is a well-known occurrence in the melt blowing process (Yin et al., 1999). While the ELD technique does detect the bundling of fibers, the technique does not provide a quantitative measurement for the amount of bundling. In *Figure* 7 it appears that the normalized standard deviation of the fiber distribution approaches an asymptote at approximately 10 inches (25 cm) from the die face. However, even through the normalized standard distribution is constant, the mean diameter is still increasing with further bundling as shown in *Figure* 6.

Variations across the Die Face

Measurements were taken across the width of the fiber curtain in order to gauge the uniformity of fiber distribution. End effects play an important role in the formation of melt blown products, and controlling the depth of the end effect is an important part of engineering a melt blowing process. The air flow patterns near the ends of the air slots are different from the air flow patterns near the center of the slots. These differences are collectively referred to as jet end effects. Measurements across the width of the die (in the x direction) were taken at four different distances (z positions) from the die. These results are shown in *Figures 8 and 9* for mean diameter and standard deviation, respectively. Observe that the fiber size is nearly constant across the die face. This suggests that the air end effects do not penetrate far enough from the ends of the air slots to affect the fiber attenuation. Thus, having two inches of fallow space (die length with air slots but no polymer holes) at either end of the die was sufficient to prevent air jet end effects from affecting the fiber attenuation. Without this fallow space, the polymer exiting the holes at the

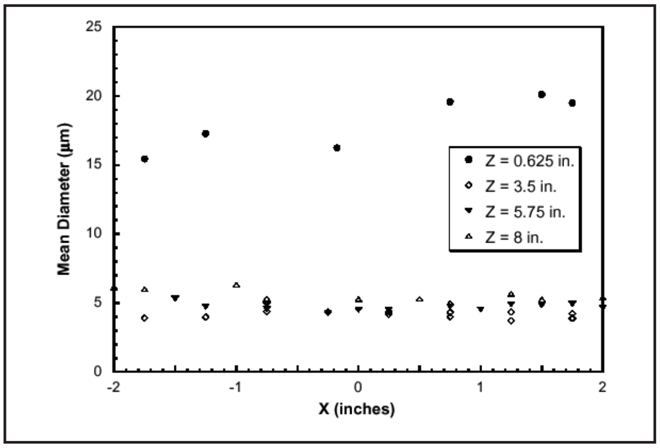


Figure 8
VARIATION OF FIBER DIAMETER IN THE X DIRECTION FOR DIFFERENT Z LEVELS AND Y = 0. THE RUN CONDITIONS WERE 2.27 KG/HR (5 LB/HR) POLYMER FLOW, 300°C DIE TEMPERATURE, AND 2500 SLPM AIRFLOW

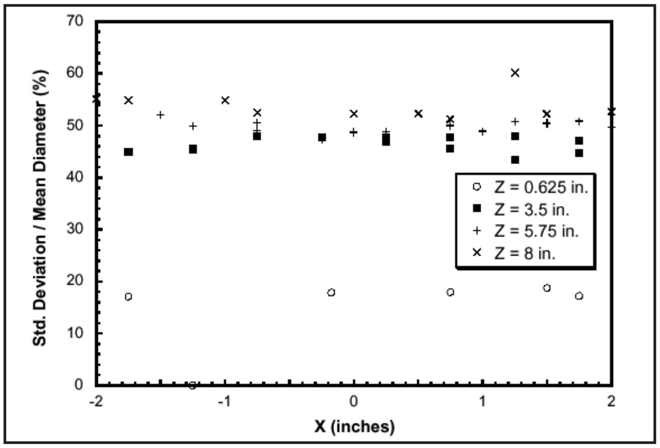


Figure 9
DIAMETER STANDARD DEVIATION VARIATION IN THE X DIRECTION FOR DIFFERENT Z LEVELS AND Y = 0. THE RUN CONDITIONS WERE 2.27 KG/HR (5 LB/HR) POLYMER FLOW, 300°C DIE TEMPERATURE, AND 2500 SLPM AIRFLOW.

ends of the die would have been exposed to a different air field than the polymer exiting the holes that are nearer to the die center. Since the air field is what makes melt blowing work, then a different air field would produce different fiber attenuation, different bundling, etc. (Fiber uniformity would decrease.)

Spinline Profiles

To gauge the sensitivity of the ELD instrument to process changes, three measurements of the center fiber diameter profile (measurements along the z axis at x=0 and y=0) were taken at different polymer flow rates and air flow rates. The three tested polymer flow rates (2.5 lb/hr, 5 lb/hr, and 10 lb/hr) cover most of the operating range of this die at 300°C and 2500 SLPM airflow using Fina Dypro 3860 isotactic polypropylene. The mean diameter is shown in *Figure 10*, and the standard deviation is shown in *Figure 11*. The lower the polymer flowrate, the smaller the fiber diameter, as is expected. All three cases also show the mean diameter growth due to bundling.

Figure 11, the standard deviation graph, shows that, at large distances from the die face, the normalized standard deviation approaches an asymptotic value. The standard deviation becomes approximately 60% of the measured mean fiber

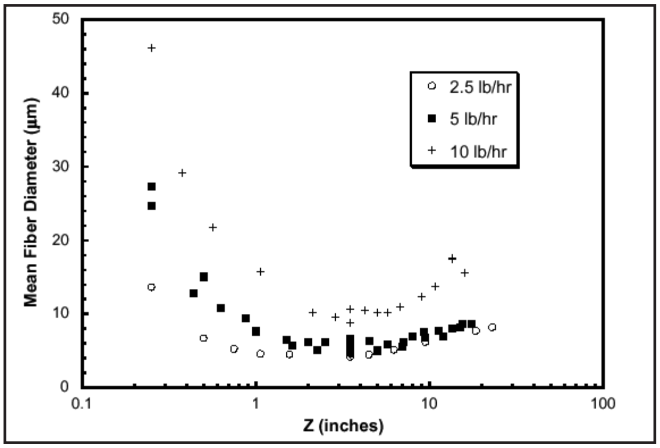


Figure 10
MEAN DIAMETER PROFILES AS A FUNCTION OF Z AND FOR VARYING POLYMER FLOW RATES. THE DIE TEMPERATURE WAS 300°C AND THE AIRFLOW WAS 2500 SLPM

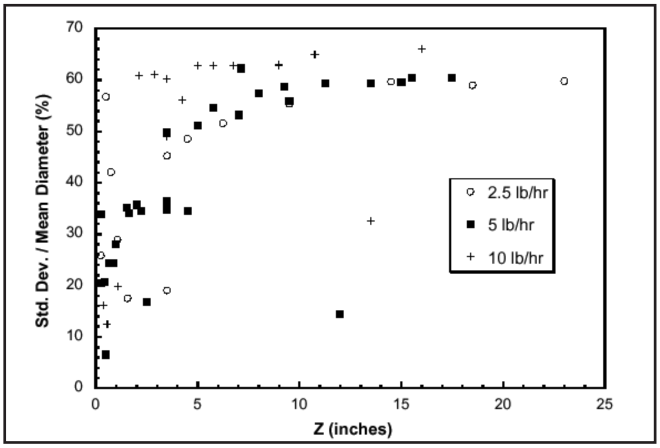


Figure 11
DIAMETER STANDARD DEVIATION VARIATION AS A FUNCTION OF Z AND FOR VARYING POLYMER FLOW RATES. THE DIE TEMPERATURE WAS 300°C AND THE AIRFLOW WAS 2500 SLPM

diameter, regardless of the polymer flow rate.

Figure 12 shows the mean diameter profiles for two different airflow rates. As is typical in melt blowing processes, the higher airflow rate produces finer fibers. Also, the distance at which attenuation is complete is shorter for higher airflow rates. As for the bundling effect at high z values, both airflow rates produce about the same percent increase in fiber size (versus the minimum fiber diameter at the flow rate in question).

Conclusion

Ensemble laser diffraction, or ELD, is a new technology for measuring fiber diameter distributions. This technique provides quick distribution measurements for both on-line and off-line operation. However, the ELD technique is based on a semi-empirical model for light diffraction though multiple fibers. This semi-empirical nature of this model may require calibration and verification for new applications. For the melt blowing application discussed in this paper, the ELD technique was calibrated against microscopic (off-line) measurements of fiber diameter and bundle size. The chief advantages of ELD include (a) the large sampling volume compared to conventional LDV, (b) the nearly instantaneous response time, and (c) the ability to measure both moving and stationary fibers (i.e., on-line and off-line capability).

The mean fiber diameter showed a minimum along the spinline length. This minimum separates the attenuation region of the spinline from the bundling region. Also, the 8 inch die - with a center 4 inch zone for the fibers – had minimal variation of fiber attenuation rate across the width of the fiber curtain.

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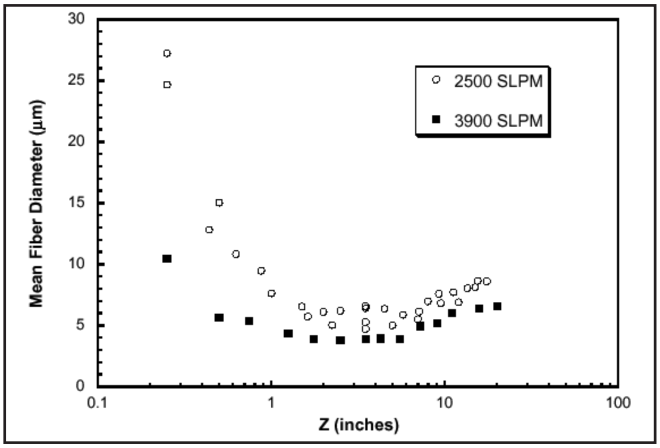

Figure 12
MEAN DIAMETER PROFILES FOR TWO DIFFERENT AIRFLOW RATES. THE POLYMER FLOW RATE WAS 2.27 KG/HR (5 LB/HR) AND THE DIE TEMPERATURE WAS 300°C

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