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AN ENSEMBLE LASER DIFFRACTION TECHNIQUE FOR ON-LINE CHARACTERIZATION OF ELECTROSPUN FIBERS

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ABSTRACT

An ensemble laser diffraction (ELD) technique has been developed for the measurement of fiber size distributions in nonwovens. This technique enables in-process real-time measurements, as well as quick off-line measurements. ELD is advantageous over imaging techniques because it does not search for individual in-focus fibers for measurements. Instead, it collects the combined signal of a collection of fibers (typically several hundred) in the path of a laser beam for the measurement. Further, the optical system of ELD is designed to measure the light scattering patterns of fibers regardless of their locations and orientations.

Earlier work has shown that fiber size distributions can be measured down to roughly 1.5 um mean size using a violet laser diode. Recent work has focused on using ELD to measure the fibers produced by two different electrospinning systems. This work (along with other measurements) has shown that it is possible to measure fiber mean sizes down to approximately 0.6 um. Future hardware changes should allow for measuring of mean sizes down to 0.2 um.

INTRODUCTION

Fiber diameter is one of the most important physical properties affecting nonwoven's performance. Previous work has shown how the ELD technology can be applied to accurate sizing of both offline samples and also how it can be applied to online processes for real-time measurement of fiber size distributions. Several improvements to the system have been incorporated. Two major improvements have been the use of a violet laser diode for measurement of smaller fiber diameters, and changes to the receiving head of the instrument which allow for sizing of fibers that are further distances from the optics. Both of these changes allow the ELD technology to be applied to a broader set of production methods and fibers.

HARDWARE

Figure 1 shows a schematic of the hardware involved in measuring the diffraction pattern from a group of fibers. A laser source produces a collimated beam of light, which is then scattered by fibers. The diffracted light hits a lens, which focuses the light onto a custom twenty-eight ring detector. This ring detector provides a measurement of the light scattering as a function of angle. The unscattered light is focused down and passes through a hole in the ring detector. A transmission detector located behind the ring detector measures the fraction of light that is not scattered by fibers. Figure 2 shows the custom ring detector. The hole at the center of the detector through which the unscattered light passes can be seen, as well as several of the larger rings.

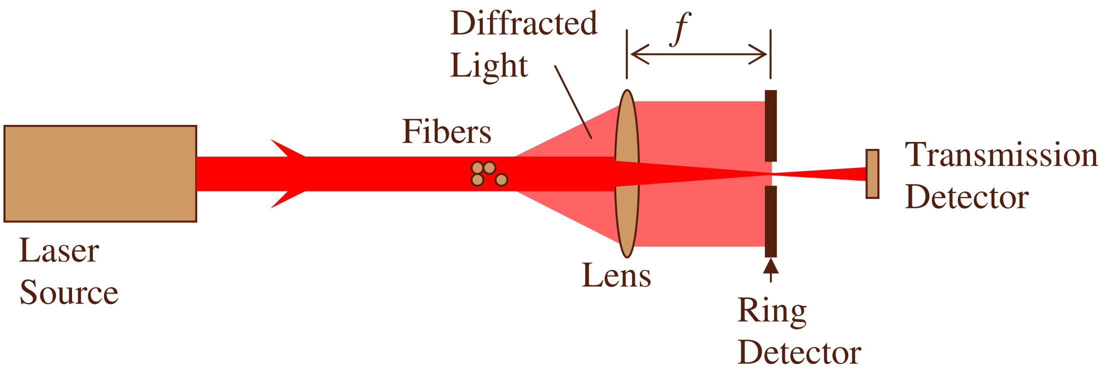

Figure 1: Schematic of ELD Hardware

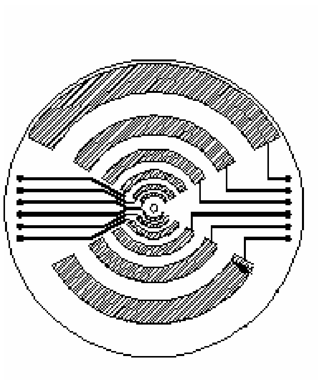

Figure 2: Schematic of the Ring Detector

MEASUREMENTS MADE ON FIRST ELECTROSPINNING APPARATUS

Figure 3 shows the physical layout of the electrospinning apparatus and the ELD hardware. In electrospinning, a conductive polymer solution is forced through a needle tip. A high voltage is applied between this needle tip and a ground plate which is located some distance away from the needle. The electric field forces the polymer solution into a narrow cone, and pulls the solution from the needle tip towards the ground plate. This process forms fibers of very small diameters, and creates a fiber web on the grounding plate.

Two sets of data were taken. Each of the two sets of data had a slightly different needle tip to ground plate distance (dimension B from Figure 3) and also a slightly different needle tip to receiver distance (dimension C from Figure 3).

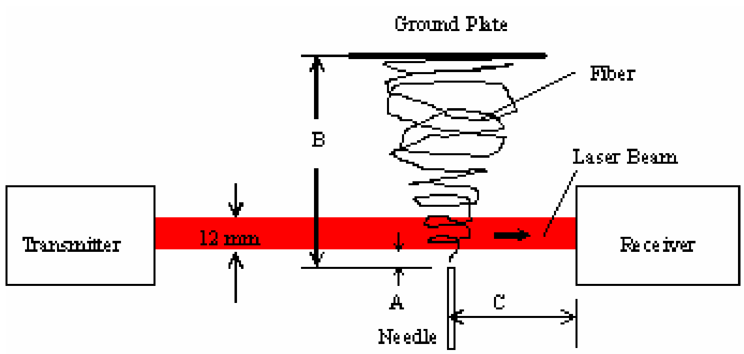

Figure 3: Physical Arrangement of Electrospinning Device and ELD Hardware

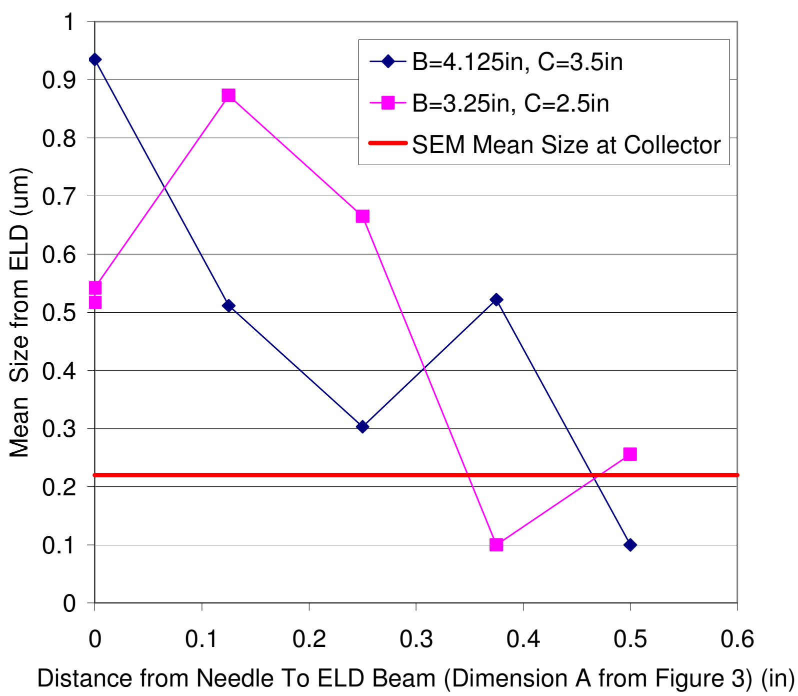

Figure 4: Mean Size Results For First Electrospinning Setup

The overall trends suggest that the fibers are decreasing in mean size as they move away from the needle tip. This is what would be physically expected to occur. Of course the uncertainty in the mean sizes measured will be quite large at less than 1.0 um, but still there is likely to be useful information in the results.

Fiber Orientation

By looking at the intensity of scattering between the horizontal and vertical configurations (rotating the ELD apparatus by 90 degrees), information on the orientation of the fibers can be discovered. This is because of the wedge shaped nature of the custom photodetector in the ELD apparatus (see Figure 2). As shown in Figure 5, the scattering was much stronger in the vertical configuration when measurements were made very close to the needle tip. This means that close to the needle tip the fibers are predominantly oriented perpendicular to the needle. Further away from needle tip the scattering is roughly equal between the horizontal and vertical configurations. This shows that further away from the tip the fibers are more randomly oriented. This seems to be in agreement with previous work (Kowalewski et al., 2003).

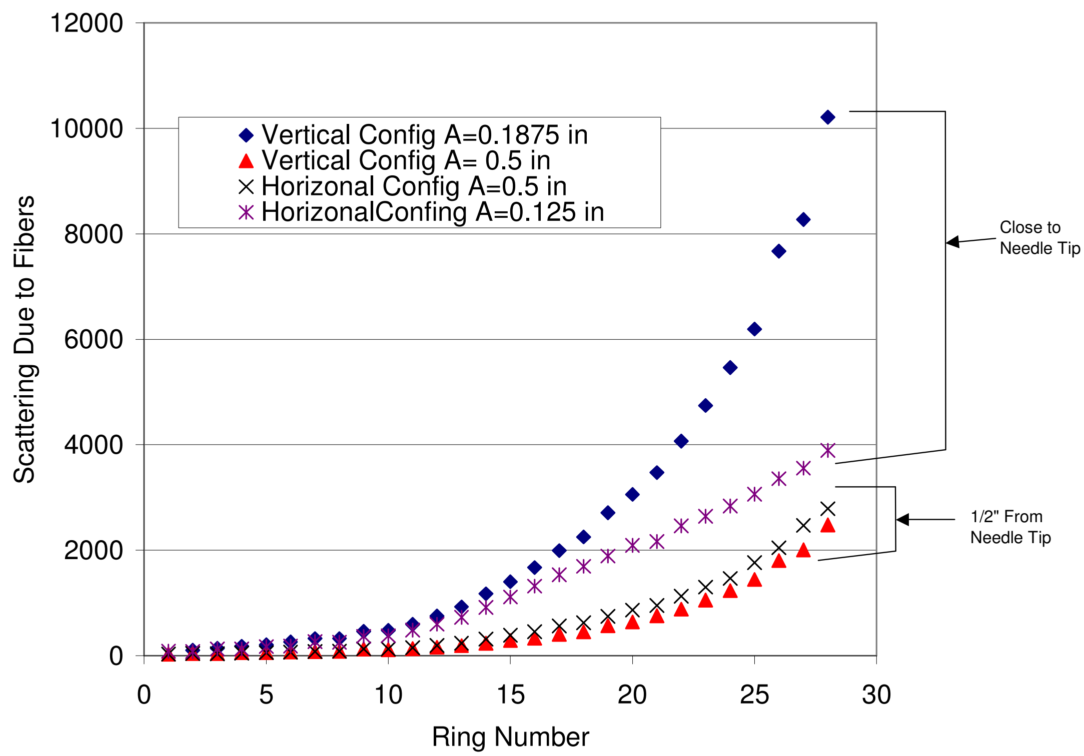

Figure 5: Intensity of Scattered Light between the Horizontal and Vertical Configurations

MEASUREMENTS MADE ON SECOND ELECTROSPINNING APPARATUS

Measurements were taken on a second electrospinning apparatus, very similar to the previous setup (see Figure 3). In this case four different polymer solutions were used to produce four different sizes of fibers. A sample of the fibers produced by each solution was taken for offline sizing using optical microscopy. A comparison of the results (see

Figure 6) shows that the ELD technique measured the mean size quite accurately, as compared with microscopy.

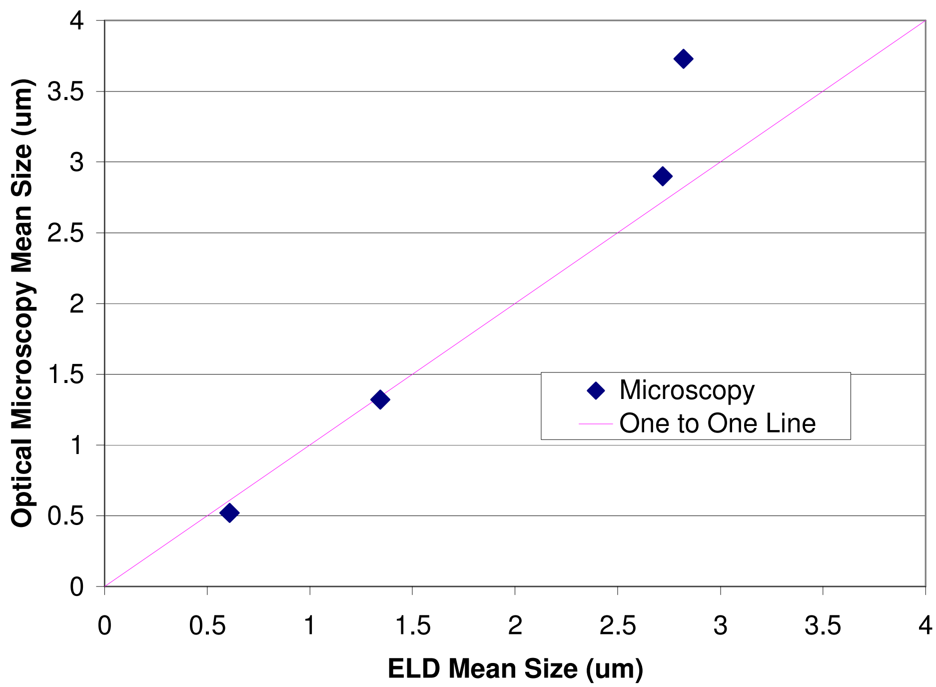

Figure 6: Comparison of Mean Size Data for Second Electrospinning System

FUTURE WORK

The emphasis on future work will be to reduce the minimum fiber size that can be accurately sized by the ELD technique. We currently use a wavelength of 405 nm, so our goal for ELD instrument is to reach the lower limit of 100 nm in fiber diameter. Fortunately, this lower limit is adequate for many of the submicron fiberizing processes. In the proposed arrangement, sensitivity to fine fibers is improved by collecting light over larger scattering angles than the present device. A convenient way to estimate the lower size limit of a particular configuration is to examine how the centroid of the scattering pattern reaches an asymptotic value with decreasing fiber diameter. Below 1 μm, the scattering pattern of the existing ELD system does not exhibit any peak or valley, but its centroid moves outward a bit in the submicron range and can be used to estimate the mean fiber size. For this purpose, centroid of the scattering pattern is defined as

$$\theta_c = \frac{\sum_{i=1}^{28} \theta_i P_i}{\sum_{i=1}^{28} P_i}, \qquad (1)$$

where i denotes the ring number of the scattering detector (i=1 being the innermost ring and i=28 the outermost). The symbols θ_i and P_i represent the mean scattering angle and the scattered power pertaining to the ith ring. Figure 7 shows the centroid location as a function of the fiber diameter. Centroid locations for the largest scattering angle of 7 and 35º are compared. They pertain to the existing ELD system and the proposed ELD system respectively. The centroid of the scattering pattern reaches about 99% of the asymptotic value at 0.5 µm for the current system and 100 nm for the proposed arrangement.

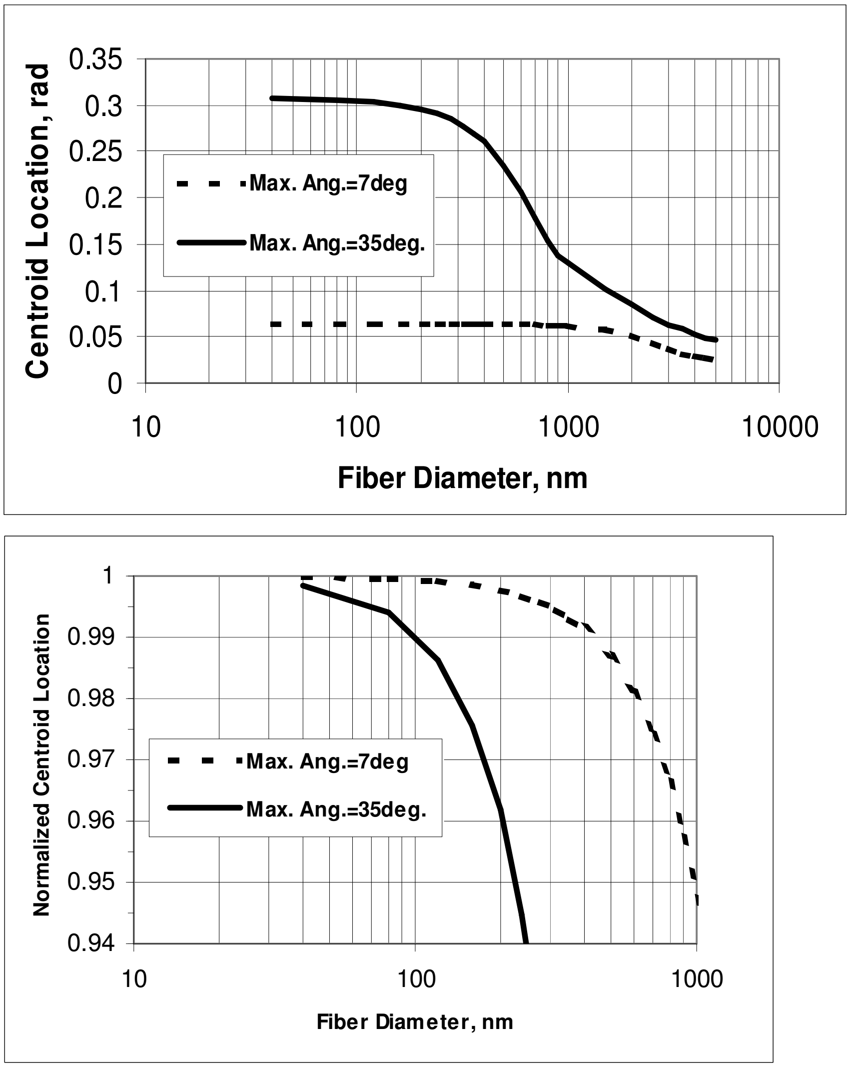

Figure 7: Location of the scattering centroid as a function of fiber size

The maximum scattering angle of 35º may be accomplished in a practical system by using a high numerical aperture lens (diameter of ~150 mm for a focal length of 100 mm) to collect the scattered light. Also a large ring detector needs to be incorporated.

The above lens has an exceptionally large numerical aperture and may involve several elements. Nonetheless, the lens design can be kept simple, as it is not an imaging application. To a certain extent, lens aberrations would not deteriorate the signals. The ring detector assembly needs to be made out of a single 6" silicon wafer. The ring detector should be able to effectively collect light reaching the front face for a range of angles determined by the range of fiber locations. If there is a loss of collection efficiency at large angles, a correction factor must be worked out. Further, large area detectors will have slower response and larger noise that need to be estimated, in order to establish the feasibility of this design.

CONCLUSION

Several significant improvements to the ELD system have been implemented, resulting in improved measurement of smaller fibers (down to 0.6um), as well as improved spatial measurement which improves results for certain online applications. Future enhancements will again be focused on improving sizing accuracy, increasing the range of fiber sizes covered, and also on broadening the range of processes that the technology can be used on.

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