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Recent Developments and Applications of Ensemble Laser Diffraction Technology for Diagnostics of Nonwovens

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Abstract

Ensemble laser diffraction (ELD) technique has been recently 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. Optical system of ELD is designed to measure the light scattering patterns of fibers regardless of their locations and orientations. This feature has enabled for the first time to collectively measure the streams of fibers emerging from a spinner head like a meltblowing die.

The present paper reports the recent enhancements to the ELD technique and shows how they are helping with the advanced diagnostics of nonwovens.

The smallest fiber size measured by ELD is limited by the wavelength of the laser used for the measurement. Previous technology employed a red laser diode (wavelength: 635nm), which resulted in a minimum measurable fiber size of 2.5 μm . A violet (405nm) laser diode has been recently incorporated into the system. The shorter wavelength of this laser source allows for sizing of fibers down to about 1 μm in mean size. It also improves the measurement accuracy for fibers between 2.5 and 5 μm . This size range is common in both meltblown polymer fibers and glass fibers. This new system will allow for wider use of the ELD technology by industry.

A second improvement to the system has been the innovative redesign of the opto-mechanics to collect scattered light from fibers across a larger spatial region. In the previous design, fibers had to be located within ~75 mm of the receiving head in order to accurately size them. The new optics allow for accurate sizing out to 150 mm. Further, new algorithms are developed to measure fibers as far away as 500 mm from the receiver, provided that the mean size is larger than 4.5 μm . This is a significant improvement for many on-line applications of nonwoven fibers.

Finally, this paper reports measurements of fiber structure and morphology in addition to the size distribution. The light scattering pattern measured by the ensemble laser diffraction technique contains information about the radial refractive index profile in the fiber which evolves with the solidification of the fibers. Hence, information about solidification of the fibers in online applications is obtained. Similarly, certain characteristics of the scattering pattern reveal information about formation of crystals within the fibers. Online data are used to show the capability of ensemble diffraction in measuring the fiber structure and morphology.

Introduction

Recent studies have shown that Ensemble Laser Diffraction (ELD) technology can be applied to accurate sizing of both offline fiber samples and 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 located farther from the optics. Both of these changes allow the ELD technology to be applied to a broader set of production methods and fibers. Further, additional experimental results are obtained that show the ability of ELD to monitor not only the fiber size distribution but also the status of polymer solidification and crystallization.

Principle of the Technique

Figure 1 shows a schematic of the ELD system. A laser source produces a collimated beam of light, which is scattered by fibers. The diffracted light is collected by 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.

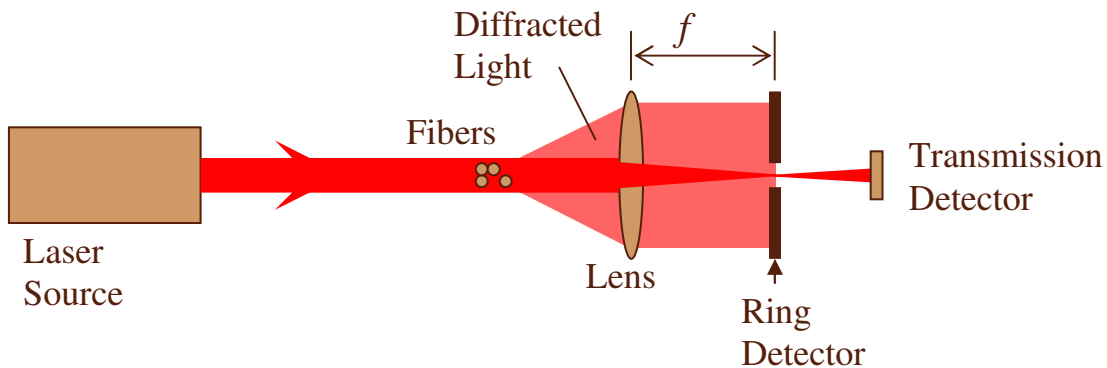


Figure 1: Schematic of ELD System

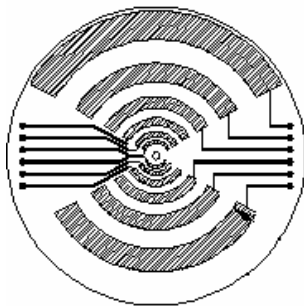


Figure 2: Schematic of the Ring Detector

Measurement of Small Fibers using Violet Laser Diode

The smallest fiber size measured by ELD is limited by the wavelength of the laser used for the measurement. Previous technology employed a red laser diode (wavelength: 635nm), which resulted in a minimum measurable fiber size of 2.5 μm . By incorporating a violet (405nm) diode as the laser source, sizing down to $\sim 1 \mu\text{m}$ is made possible.

Three fiber samples made up of small meltblown fibers were tested using the red diode and violet diode. The samples were then analyzed using scanning electron microscopy (SEM). The results obtained are shown in figure 3.

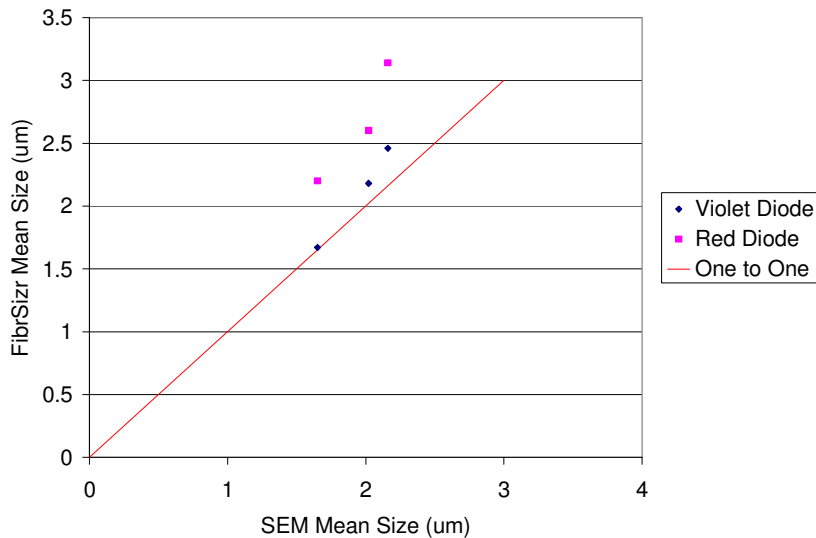


Figure 3: Diffraction Size Comparisons with SEM for Violet and Red Diode

The violet laser produces more accurate measurements of fiber size. The red diode still shows a decreasing size trend, but measures sizes significantly larger than the SEM size. The fiber size based on violet diode laser is slightly larger than the SEM size. This may be due to fiber bundling or other effects.

Enhancement of Spatial Measurement Range

As shown in Figure 1, the ELD system uses a lens to collect the scattered light and the ring detector is located in the back focal plane of the lens. This arrangement makes the diffraction measurement insensitive to the fiber location, as illustrated in Figure 4. Light rays scattered at a certain angle reach a fixed point on the ring detector. In other words, the ring detector measures the angular scattering pattern of the fibers regardless of their locations. A ring at a distance r from the center of the ring detector receives light scattered at an angle r/f , where f is the focal length of the collecting lens.

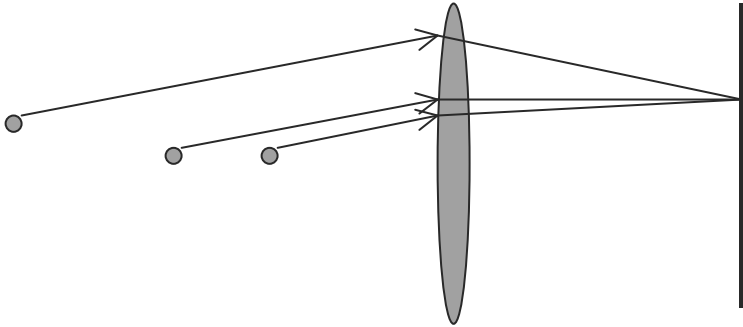


Figure 4: Position insensitivity of diffraction pattern measurement

Scattered light reaching the ring detector is not biased by the fiber location. However, if the fiber is too far from the lens, scattered rays beyond a certain scattering angle miss the lens and do not reach the ring detector. With increasing distance from the lens, the largest measurable scattering angle is reduced. This limits the ability of the system to measure small fibers that have significant features in their scattering patterns at large angles.

Hence, it is desirable to keep the fibers closest to the lens. In the case of online measurements, fibers cannot be too close to the lens, which is kept clean by purge airflow as illustrated in Figure 5.

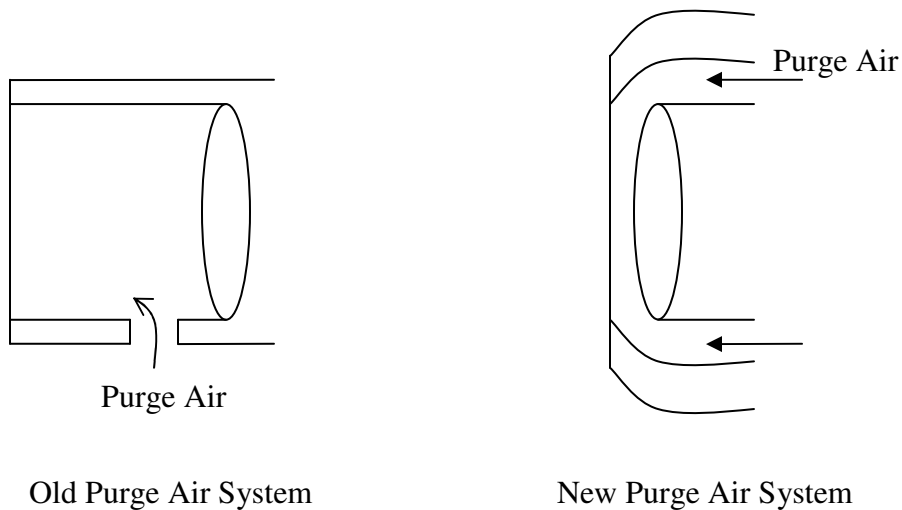


Figure 5: Purge Air Flow System

In the old system, purge air was supplied in front of the lens and required a certain tube length for the flow to be streamlined. As a result the fibers could not be brought close to the lens. The new purge air system brings air to the lens that is already streamlined and helps shorten the tube length in front of the lens.

Figure 6 shows the efficiency of scattered light collection for various detector rings if the fibers are located at a distance of 125 to 150 mm from the front of the receiver. Inner rings collect 100% of the light, whereas there is a loss of collection for the outer rings due to the finite diameter of the lens. The measured data of the scattered light may be divided by the efficiency factors in Figure 6 to estimate the true scattering pattern. However, the correction process is not reliable if the collection efficiency is too small.

In the case of old system the collection efficiency on the two outer rings is too small to obtain reliable measurement. Hence very small fibers cannot be measured. For the same setup, the new system has more than 50% collection efficiency for all the rings and hence, does not limit the measurable minimum fiber diameter, even if the fibers are located as far as 150 mm from the receiving optics.

Similar simulations have shown that accurate measurements can be obtained with the new system at a distance of 500 mm, provided that the mean fiber diameter is at least $4.5 \mu\text{m}$.

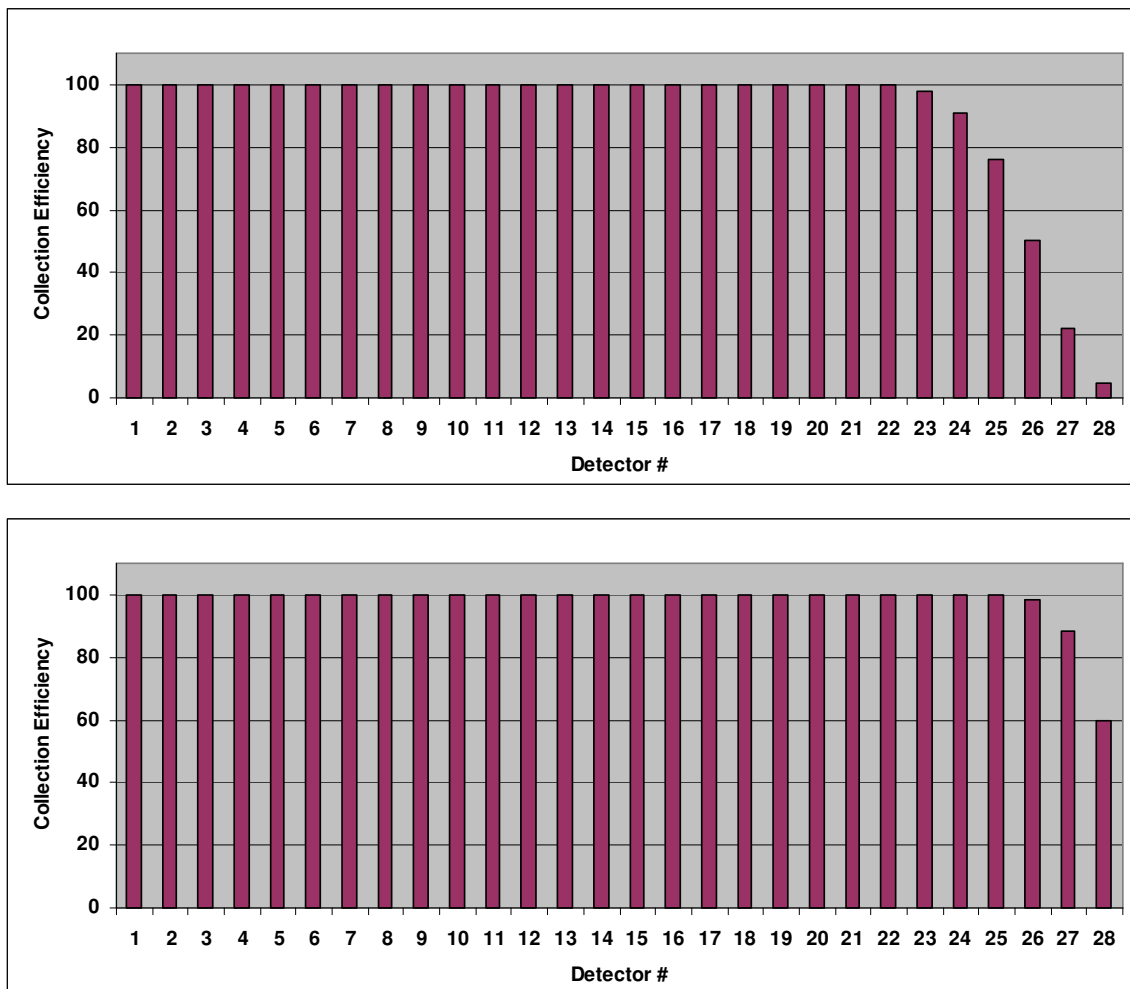


Figure 6: Collection efficiencies of the scattering detectors for the old (upper plot) and the new (lower plot) purge air system

As reported earlier, the ELD system is not only insensitive to the fiber location for a substantial spatial range, but also insensitive to the fiber orientation for all practical purposes (Fandrey & Naqwi, 2003). The present improvements have enabled unbiased measurements of randomly located and randomly oriented fibers from a large spatial region.

Measurement of Fiber Properties other than Size

Results of recent measurements at Biax FiberFilm are shown in Figure 7. Fiber diameter decreases with distance from the die. The drop in the mean size is rapid in the beginning and slows down later. Due to fiber bundling, the upper end of the size distribution increases beyond 6" from the die. This is consistent with the results reported by Fandrey & Naqwi (2003) and Moore et al. (2004).

Evolution of various optical parameters in the above process is considered in Figures 8 and 9. As the polymer solidifies, the refractive index increases at the fiber axis. This results in the 'central peak' emerging in the forward light scattering pattern. According to Figure 8, the peak begins to appear at about 5 inches from the die and continues to grow up to 20 inches. This information may be useful in the numerical simulation of the process. Further, this peak is a measure of the stresses in the fiber which may affect the mechanical properties of the final product, such as tear strength of the fabric.

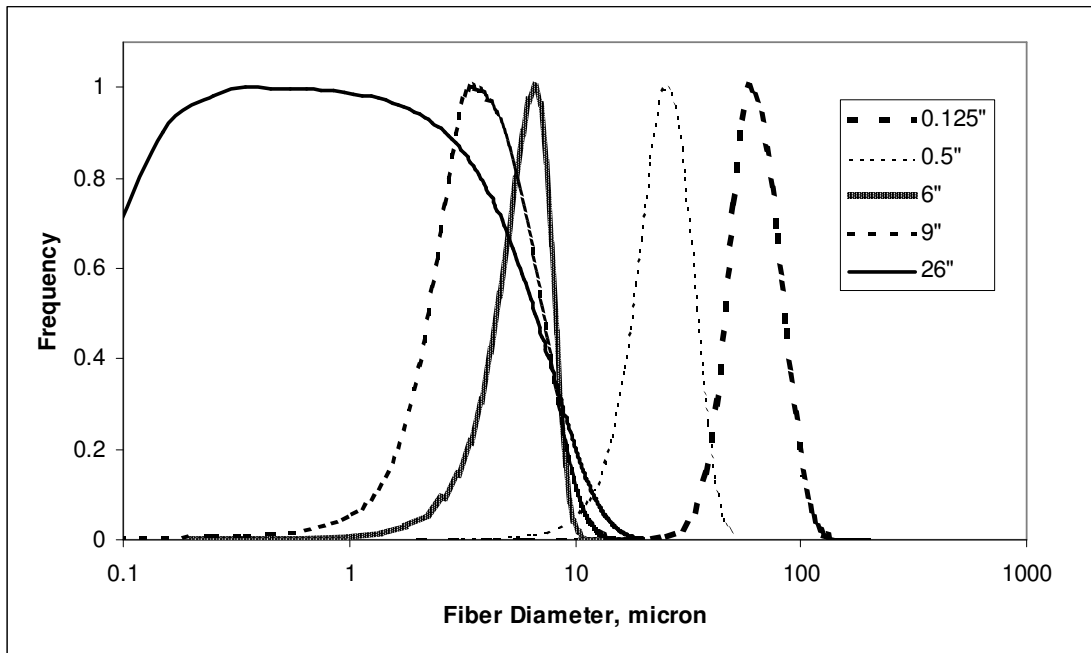


Figure 7: Fiber size distributions at various distances from a meltblown die

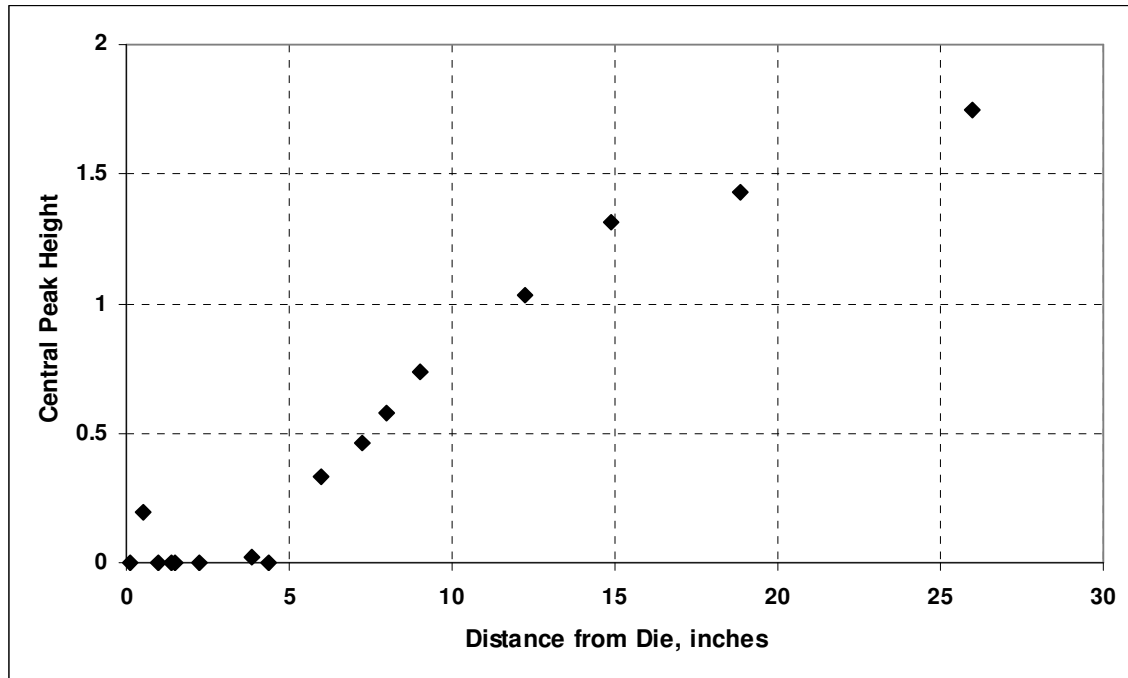


Figure 8: Evolution of radial refractive index profile

Wide-angle scattering is a measure of the inhomogeneity within the fiber, caused by crystallization. This effect is negligible very close to the die but appears within 1” from the die, as shown in Figure 9. Also shown in Figure 9, is the modulation depth of the diffraction pattern, which is degraded due to inhomogeneity near the fiber surface. As the surface inhomogeneity becomes strong light does not penetrate deep into the fiber and wide angle scattering is reduced. However, when fiber size becomes small (i.e. a few microns) and comparable to laser wavelength, surface of the fiber begins to appear smooth and modulation depth increases. At the same time wide-angle scattering increases as the incident light does penetrate fully into the fiber due to its small diameter.

Part of the data in Figure 9 is wavelength dependent. However, the optical parameters in this figure do provide information about when the crystallization begins and when does it begin to affect the fiber surface. These pieces of information may be useful in simulation of the process and in predicting the properties of the final product.

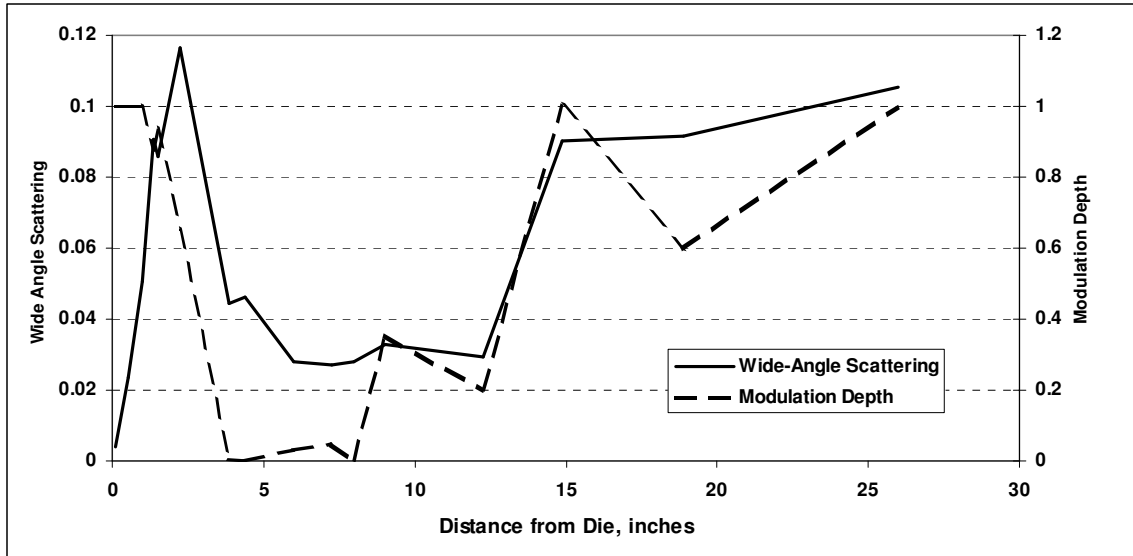


Figure 9: Evolution of crystallization related optical parameters

Conclusion

Several significant improvements to the ELD system have been implemented, resulting in improved measurement of smaller fibers (down to $\sim 1 \mu\text{m}$), as well as improved spatial measurement which provides better results for certain online applications. Further, it is shown that ELD measures not only the fiber size distribution but also determines the status of crystallization and solidification of the fibers in a fiberizing process.

Acknowledgements

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