TECHNICAL NOTE

R. R. Bresee,[1] *Ph.D. and E. A. McCullough,*[1] *Ph.D.*

Discrimination Among Acrylic Fiber Types by Small-Angle Light Scattering of Single Fibers

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ABSTRACT: Small-angle light scattering (SALS) patterns and scanning electron photomicrographs of single fibers were recorded for 15 different types of acrylic fibers. SALS was seen to be a sensitive tool for discriminating among the fiber types and for characterizing fiber cross-sectional shapes, surface topography, opacity, and various forms of random bicomponent fibers.

KEYWORDS: criminalistics, synthetic fibers, lasers

Measurements of electromagnetic scattering from polymer materials have repeatedly provided useful means of investigating polymeric morphology. One form of electromagnetic radiation that is useful in characterizing polymeric films is visible light, and the scattering method is thus termed light scattering. Because a rather large electromagnetic wavelength is used in light scattering compared with that of X-ray or other radiation scattering, light scattering from polymeric solids is recorded only at relatively small scattering angles. Hence, the method is termed small-angle light scattering (SALS).

In view of the nondestructive, inexpensive, and simple nature of SALS and the fact that the method may be used on samples smaller than a microgram, an investigation was recently performed in our laboratory [*1*] into its suitability for the analysis of single fibers as in forensic science applications. That work demonstrated that SALS may easily discriminate among fibers of different generic groups, such as nylon and polyester. These results were so encouraging that we extended our investigation to the analysis of various fiber types within a single generic group. Our goal was to determine if the method is able to discriminate among fibers in the same generic group produced by different manufacturers or among fibers in the same generic group but different type produced by the same manufacturer. This paper reports the results of our efforts. We chose the acrylic group, and single fibers from 15 different acrylic types were examined.

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[1] Assistant professors, Department of Clothing, Textiles and Interior Design, Kansas Agricultural Experiment Station, Kansas State University, Manhattan, Kans. 66506.

This investigation of the discriminating power of SALS for single fibers progressed from generic group to fiber type within a generic group. In a subsequent paper we will progress to another level of discrimination by examining fibers of identical commercial origin (from the same yarn bobbin) that have been subjected to different kinds of consumer-like uses. While discrimination at these levels is quite an accomplishment for any method of analysis, it would be quite useless to the practicing forensic scientist if one additional step was not made. Hence, another study will be conducted to determine the variability of SALS patterns of numerous single fibers within several individual garments subjected to different kinds of actual consumer wear and care.

Apparatus and Experimental Procedure

The apparatus necessary for SALS measurements of fibers is simple to construct, and its use is straightforward as well as inexpensive. Only a brief description of the apparatus and methods will be given because details were discussed elsewhere [*1*].

A monochromatic, plane-polarized light source conveniently supplied by a polarized, low-power helium-neon laser is directed onto a fiber. Light scattered by the fiber is passed through a polarizer, which functions as an "analyzer," and is recorded photographically on 102- by 127-mm (4- by 5-in.) Polaroid film. Two spatial arrangements of the incident polarized beam and analyzer are typically specified. The two modes are termed Hv and Vv, where the large letters refer to the polarization direction of the analyzer, the small letters refer to the polarization direction of the incident beam, H denotes horizontal, and v denotes vertical.

All measurements in the present study were Hv types made by using a Hughes 5-mW polarized laser with each fiber sample hung vertically in air. That is, vertically polarized light was directed on a fiber sample whose long axis was vertical, and the analyzer was turned to horizontal polarization. Sample-to-film distance was held constant for each fiber at 483 mm. Exposure times ranged from 3 to 15 s. Since all fibers examined were heavily crimped, a special procedure for sample mounting was adopted to insure reproducibility in scattering. This procedure consisted of attaching a 0.1-g weight to the lower end of the fiber in such a way that it was free to rotate, thereby allowing the release of torque from the fiber, and then scanning the length of the fiber to locate a segment of the fiber that was relatively straight through the entire width of the laser beam (about 0.8 mm). The most heavily crimped fibers typically required a fiber length of about 5 mm to obtain a good, reproducible pattern. An unsuitable fiber portion was easily recognized because its scattering pattern was characterized by criss-crossed streaks instead of predominantly horizontal scatter. Good fiber-to-fiber reproducibility was obtained when this procedure was followed. If less than 5 mm of a heavily crimped fiber is available, an alternate procedure may be used. A pinhole collimator may be placed in the path of the incident laser beam so that the portion of the fiber illuminated by the laser is reduced from the diameter of the uncollimated light beam to the diameter of the pinhole. In this way higher crimp frequencies and shorter fibers may be accommodated. Pinhole collimators may be made by piercing aluminum foil with a needle. Alternatively, pinholes of accurately determined diameters may be purchased commercially[2] in sizes as small as 0.005 mm. However, the use of a collimated incident beam will dictate longer exposure times.

Scanning electron microscopy (SEM) was performed with standard procedures.

Fifteen different types of acrylic fibers were examined. These included four types of Acrilan®, three types of Creslan®, five types of Orlon®, and three types of Zefran®. The specific types and some descriptive information are listed in Table 1. All fibers were obtained as raw fiber, courtesy of the producers.

[2]For example, The Ealing Corp., South Natick, Mass.

TABLE 1—*Acrylic fibers examined by SALS.*

Producer	Type	Luster	Cross-Sectional Shape	Chemical Composition
Acrilan (Monsanto)	B16	bright	bean	monocomponent
	B39	bright	bean	monocomponent
	S82	semidull	bean	monocomponent
	B94	bright	variable	random bicomponent
Creslan (American Cyanamid)	61K	bright	round	monocomponent
	61K	semidull	round	monocomponent
	68	semidull	round to bean	bicomponent
Orlon (du Pont)	21	semidull	acorn	bicomponent
	24	semidull	mushroom	bicomponent
	28	bright	dog-bone	monocomponent
	74	semidull	mushroom	monocomponent
	632	bright	dog-bone	monocomponent
Zefran (Badische Corp.)	A101	bright	round	bicomponent
	A201	bright	round	monocomponent
	A253	bright	round	monocomponent

Results and Discussion

Figures 1 to 7 are Hv photographs of some of the fibers listed in Table 1. To save space, the SALS patterns of some fibers are not included in this paper. However, each fiber had its own characteristic scattering pattern. Replications obtained by examining different fibers from the same yarn bobbin are also excluded to save space. Each fiber type, however, clearly possessed a consistent and reproducible SALS pattern.

SALS could discriminate among different fiber types within a single generic group as well as among different fiber types from the same producer. In addition, different fiber types from a given producer, such as Acrilan types, did not have identifiable similarities in their SALS patterns. The figures included herein illustrate these points.

A comparison of fiber-scattering patterns in Figs. 1 to 7 and the cross-sectional information included in Table 1 shows that SALS may be used to characterize fiber cross-sectional shapes. Figures 1 and 2 are scattering patterns of Zefran A201 and Orlon 632, respectively. While both acrylic fibers are bright and monocomponent, their cross-sectional shapes are different—Zefran A201 is round and Orlon 632 is shaped like dog's bone. The round cross section of Zefran A201 produces a scattering pattern that is symmetrical on either side of the meridian regardless of angular rotation of the fiber about its own axis. On the other hand, the dog-bone shape of Orlon 632 produces a scattering pattern that is sometimes asymmetrical with respect to the meridian when the fiber is rotated about its own axis so that the left half of the scattering pattern is different from the right half. This phenomenon arises because a round cross section necessarily exposes an equal amount of material to the incident beam on either side of dead center whereas a nonround cross section may or may not, depending on its orientation. When a nonround cross section is oriented symmetrically with respect to the incident beam, scatter will be symmetric on either side of the meridian, but when it is oriented asymmetrically with respect to the incident beam, scatter will be asymmetric on either side of the meridian. The fiber shown in Fig. 2 was not oriented symmetrically to the incident beam and hence produced asymmetric scatter. Figures 3 to 5 show a gradual trend in scattering from symmetric, through slightly asymmetric, to highly asymmetric and correspond to fiber cross-sectional views of round, to slightly off-round, to bean-shaped.

The dependence of scattering symmetry of fiber cross-sectional shape led us to further uses of this phenomenon in examining fibers of nonround cross section. Scattering peri-

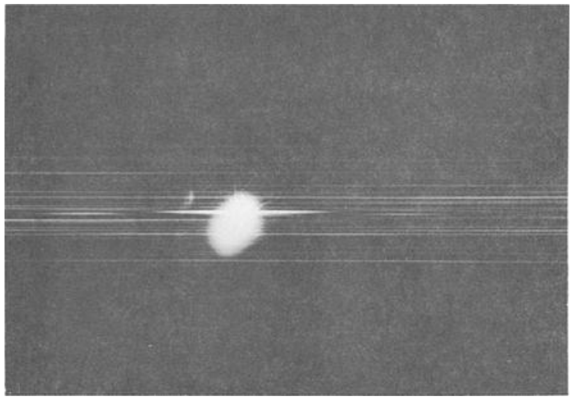

FIG. 1—*SALS Hv pattern of a Zefran A201 acrylic fiber suspended in air (fiber axis vertical).*

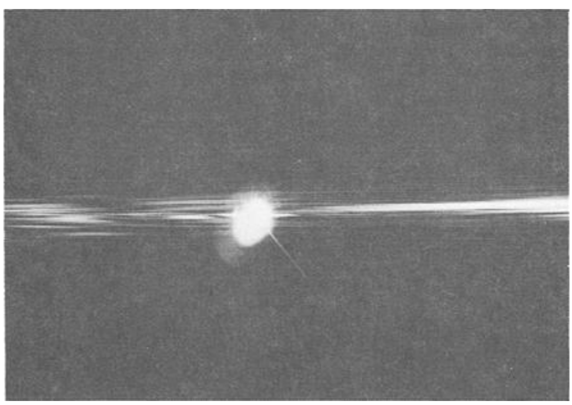

FIG. 2—*SALS Hv pattern of an Orlon 632 acrylic fiber suspended in air (fiber axis vertical).*

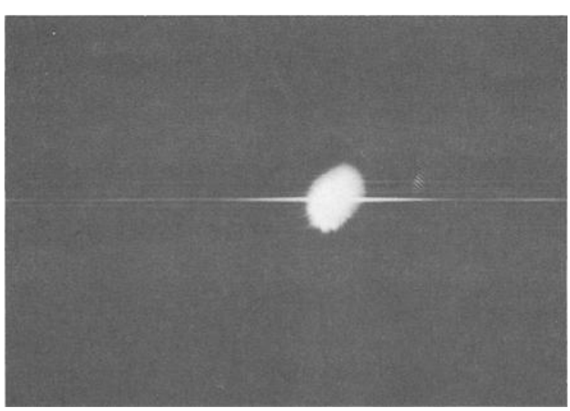

FIG. 3—*SALS Hv pattern of a Creslan 61K acrylic fiber suspended in air (fiber axis vertical).*

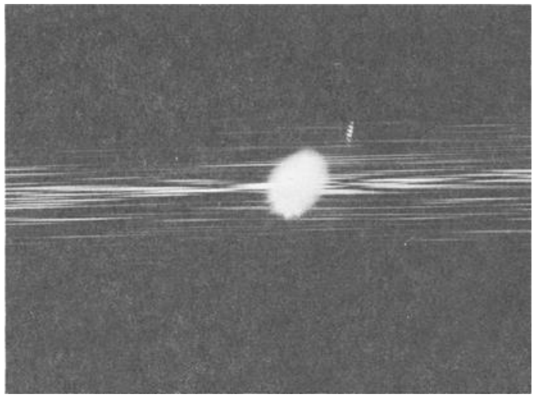

FIG. 4—*SALS Hv pattern of Creslan 68 acrylic fiber suspended in air (fiber axis vertical).*

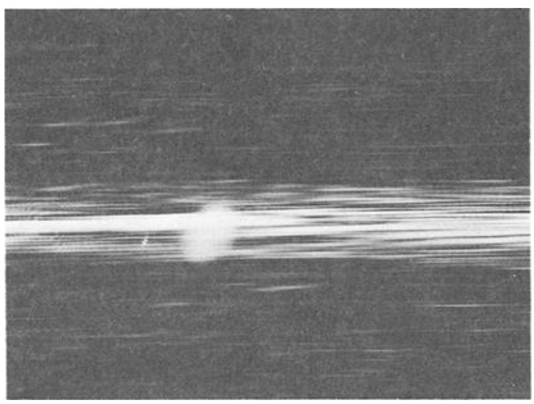

FIG. 5—*SALS Hv pattern of an Orlon 28 acrylic fiber suspended in air (fiber axis vertical).*

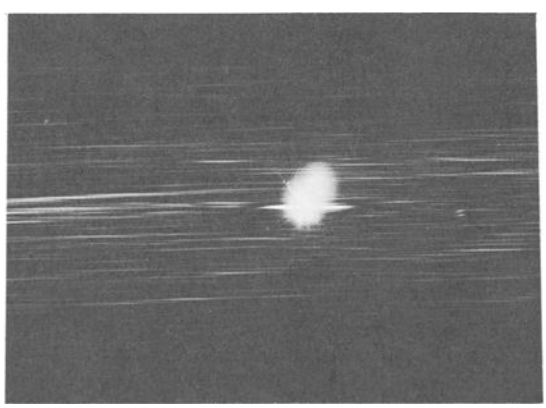

FIG. 6—*SALS Hv pattern of an Acrilan B94 acrylic fiber suspended in air (fiber axis vertical).*

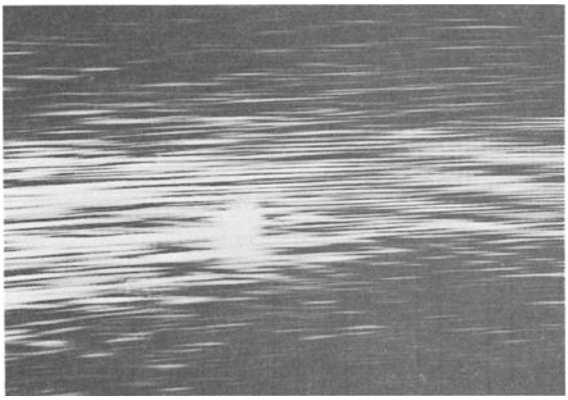

FIG. 7—*SALS Hv pattern of an Acrilan B94 acrylic fiber suspended in air (fiber axis vertical).*

odicity may be used to calculate the size of the structure producing the periodicity [*1*]. In particular, the equatorial periodicity, if identifiable, may be used to calculate fiber diameter. It thus seemed possible to characterize fiber cross sections quantitatively without cutting and mounting a lateral slice of the fiber (as required in microscopy) by examining changes in the length of the equatorial scatter as a function of the rotation of the fiber about its longitudinal axis. For example, a fiber of dog-bone-shaped cross section would be expected to produce a variable-length equatorial scatter ranging from a long period corresponding to the narrowest cross-sectional area viewed to a short period corresponding to the widest cross-sectional area viewed.

Dependence of the length of the equatorial period on the rotation angle of the fiber was found as expected for acrylic types having nonround cross-sectional shapes, whereas fibers of circular cross sections produced no change in the length of the equatorial scatter when rotated. Although not every fiber scattering pattern contains a well-defined equatorial periodicity to allow examination by this procedure, most fibers are amenable to this analysis. Figures 1 to 4 and 6 demonstrate usable scatter in this respect, while Figs. 5 and 7 exhibit no discrete equatorial scatter. This same principle can be used to obtain greater reproducibility in SALS patterns from fibers of nonround cross-sectional shapes. That is, the fiber can be rotated until either the maximum or minimum in equatorial periodicity length is obtained, since either of these two observations may be easily reproduced with another fiber. Thus, a procedure exists for both quantitatively characterizing fiber cross-sectional shapes without preparing a cross-sectional mount as in microscopy and for obtaining greater reproducibility in SALS patterns of fibers with nonround cross-sectional shapes.

Refractive indices of acrylic fibers and air are about 1.5 and 1.0, respectively. Consequently, SALS patterns of these fibers suspended in air would be expected to exhibit much surface scatter. This hypothesis was tested by mounting some fibers between microscope slides in oil of refractive index equal to that of the fiber so that surface scatter would be eliminated. We found that nearly all scattering previously observed from fibers mounted in air was eliminated and concluded that nearly all scatter observed from these fibers was due to surface interactions. With this observation in mind, we investigated the relation between fiber surface topography and total amount of light scattering exhibited by qualitatively comparing the fiber's surface roughness as revealed by SEM with the total scatter determined by SALS. We found that, in general, the rougher the fiber surface, the more scattering observed. A comparison of Figs. 3 and 4 illustrates the dependence of total scattering on fiber surface topography—the fiber of Fig. 4 has a rougher surface than that of Fig. 3.

Application of a finish that alters the fiber's surface topography produces a SALS pattern different from that normally expected from the fiber when all other factors of fiber structure are considered. This phenomenon is clearly illustrated in Figs. 2 and 5. Both fibers are bright, monocomponent Orlon fibers with dog-bone-shaped cross sections and might be expected to have similar SALS patterns. However, Orlon 28 (Fig. 5) contains a producer-applied surface modifier and the Orlon 632 (Fig. 2) is unaltered. The scattering from Orlon 28 is greater than that from Orlon 632. Examination of these two fibers by optical and scanning election microscopy showed only a slight difference in surface roughness between the two fiber types. Consequently, the sensitivity of microscopy to fiber surface modification was much less than that of SALS. This is to be expected since SALS patterns reflect sampling from the entire fiber surface whereas only one side of a fiber is seen at any given time by microscopy. The greater sensitivity of SALS in detecting differences in surface topography constitutes a major advantage of SALS over microscopy when fibers with subtle surface differences are compared, for example, when similar fibers subjected to various finishing treatments such as fabric softening are examined. Thus, SALS offers a sensitive means to characterize fiber surface topography as well as to detect surface modification.

Examination of SALS from Acrilan B94 was very interesting. Acrilan B94 is a random bicomponent fiber, meaning that two kinds of polymer were mixed in random proportions prior to extrusion. This practice results in fibers that vary continuously in chemical composition from the extreme of 100% of one polymer to 100% of the other polymer. Physical composition of the resulting fibers varies continuously from a homogeneous smooth fiber corresponding to a pure monocomponent fiber to a heterogeneous mix of two phases in the case of the 50:50 blend of polymers. A textile composed of random bicomponent fibers may be very difficult to analyze since chemical and physical properties vary over a wide range.

Numerous scattering patterns were recorded of Acrilan B94. As expected, a wide range of scattering was exhibited. The extremes are presented in Figs. 6 and 7. Figure 6 indicates that the corresponding fiber is characterized by a relatively smooth surface texture and nearly round cross section. Thus, the scattering pattern probably represents a nearly homogeneous monocomponent fiber. On the other hand, Fig. 7 indicates that the corresponding fiber has a very rough surface texture and highly nonround cross section. Thus, the scattering pattern probably represents a blend of the polymers near 50:50. Evidence of this interpretation is presented in Figs. 8 and 9, SEM photomicrographs of two different Acrilan B94 fibers. Figure 8 corresponds to a fiber expected to produce scattering like that of Fig. 6, and Fig. 9 corresponds to that of Fig. 7. Thus, SALS offers a convenient and nondestructive way to characterize the full range of random bicomponent polymer mixtures.

It is sometimes useful to compare the exposure times used to obtain photographs of SALS. Exposure times necessary to obtain identical negative density depend directly on fiber opacity and serve to further characterize the fiber in question. The most straightforward application of this principle is to characterize the opacity of different fibers of the same fiber type but different luster. Opacity may be commercially altered by the addition of an opaque pigment to the fiber dope prior to extrusion. This procedure was used to produce two samples of variable opacity used in this study: Creslan 61K, bright, and Creslan 61K, semidull. Whereas scattering patterns for both fibers are identical, exposure times required to obtain identical negative density were 5 s for the semidull fiber and 3 s for the bright fiber. Clearly the opacity agent added to the semidull fiber absorbed a significant portion of the incident laser light. Consequently, SALS provides a means of quantitatively

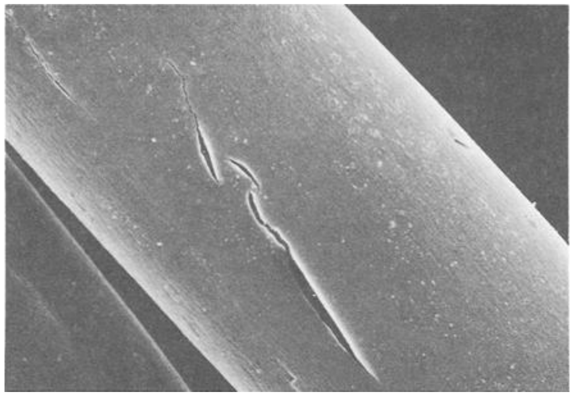

FIG. 8—*SEM photomicrograph of an Acrilan B94 acrylic fiber.*

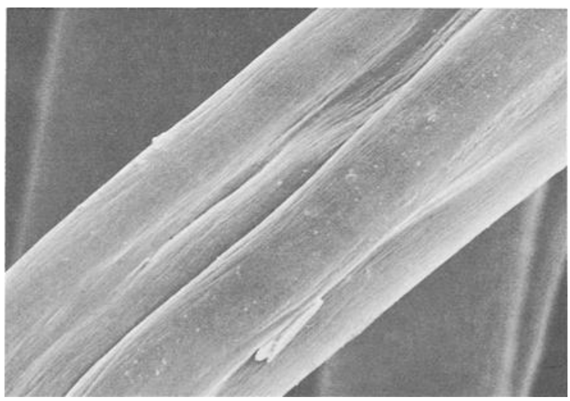

FIG. 9—*SEM photomicrograph of an Acrilan B94 acrylic fiber.*

characterizing fiber opacity. This phenomenon will be more thoroughly investigated in a subsequent publication.

Summary

SALS provides a useful method of discriminating among single fibers of different fiber types within the same generic group. It is also useful in characterizing fiber cross-sectional shapes, surface topography, modifications to surface topography, opacity, and various forms of random bicomponent fibers. SALS was more sensitive than microscopy when fibers with subtle differences in surface topography were compared. In addition, procedures for increasing the reproducibility of SALS measurements of fibers with nonround cross sections or heavy crimp were proposed.

Acknowledgments

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Reference

[*1*] Bresee, R. R. and Donelson, D. S., "Small-Angle Light Scattering for Analysis of a Single Fiber," *Journal of Forensic Sciences*, Vol. 25, No. 2, April 1980, pp. 413-422.

Address requests for reprints or additional information to
Randall R. Bresee, Ph.D.
Department of Clothing, Textiles and Interior Design
Kansas Agricultural Experiment Station
Manhattan, Kans. 66506