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Improved accuracy of the laser diffraction technique for diameter measurement of small fibres

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The accuracy of a laser diffraction technique has been improved for small fibre diameter measurement by a calibration with SEM-measured internal standards. The calibration showed that the laser diameter is about 5% larger than the corresponding SEM diameter. After correction for systematic error via accurate calibration, the laser technique was found to be fast and capable of attaining a diameter measurement accuracy of 0.1 μm.

1. Introduction

Accurate and rapid fibre diameter measurement of high performance ceramic fibres such as those being developed at Dow Corning is essential for successfully determining tensile properties which are often the critical criteria for acceptance of these fibres. Due to the dependency of tensile properties on the cross-sectional area of the fibre, any error in diameter measurement is magnified by a factor of 2 in the area and hence in tensile strength or modulus. For a given sample length, the surface flaw population is proportional to the fibre circumference or the fibre diameter, while the interior flaw population is proportional to the cross-section or the square of diameter. Thus, there is a strong desire to produce smaller fibres in order to achieve stronger fibres. Assuming a fibre diameter of 5 μm, the diameter measurement needs to have an accuracy of 0.1 μm in order to reduce the effect of diameter error on tensile strength and modulus error to less than 5%. The optical microscopy technique commonly used for diameter measurement cannot, however, accurately measure smaller diameters, due to the limitation of the wavelength of light (e.g., 0.6 μm for green light). Although photomicrographic measurement can give accurate results, it is very time consuming. On the other hand, after correction for the systematic error through the use of SEM-calibrated internal standards, the laser diffraction technique was found not only to be fast (5 minutes per measurement) but also capable of meeting the goal of 0.1 μm accuracy.

2. Laser diffraction technique for diameter measurement

2.1. Derivation of laser diffraction equation

The theory for the diffraction of light from an infinite right cylinder (Fresnel diffraction) is quite complex and requires the use of vector quantities for the electric fields [1]. Fortunately, the cylinder may be approximated by its two-dimensional profile (Fraunhofer diffraction) when light approaches and leaves an object in plane waves. The light waves can be made parallel to simulate the Fraunhofer region if the light source is a laser beam, equipped with a collimating lens, and limited to a small angle of scattering ($\leqslant 30°$). During the calculation of the intensity pattern in the Fraunhofer diffraction, the resultant amplitude of the diffracted wave is a function of the diffraction angle. In addition, the path difference between diffracted rays is a whole multiple of wavelength, thus the fibre diameter, d, can be determined as a function of wavelength, λ, node length or half the distance between the two nth nodes ($\Delta Zn/2$), and screen distance, L, as shown below and as derived in Fig. 1.

$$d = n\lambda\left[1 + \left(\frac{2L}{\Delta Zn}\right)^2\right]^{1/2}$$

2.2. Review of diameter measurement by laser technique

The fibre diameter measurement by laser diffraction technique was reported more than 20 years ago [2, 3]. It was also recognized that since the laser diameter equation is derived from an approximation of the Fraunhofer diffraction, a correction of the systematic error is needed to improve the diameter accuracy. Such a correction was tried by Perry *et al.* [4], Gagnaire *et al.* [5], and Fischbach [6], but all those correction formulae are based solely on computation and on different assumptions about fibre optical properties. None has been confirmed quantitatively via comparison with measurements on calibrated fibres. Some calibrations were attempted by Perry *et al.* [4] and Fischbach [6] with the use of tungsten wires, but the calibrated data scatter broadly.

2.3. A set-up of laser diffraction

The basic layout of the laser diffraction set-up used for this study at Dow Corning is illustrated in Fig. 2. A low power helium–neon laser beam (0.8 mW) was focused using a 15 cm focal length lens to a diameter of about 100 μm. The fibre sample is mounted on top

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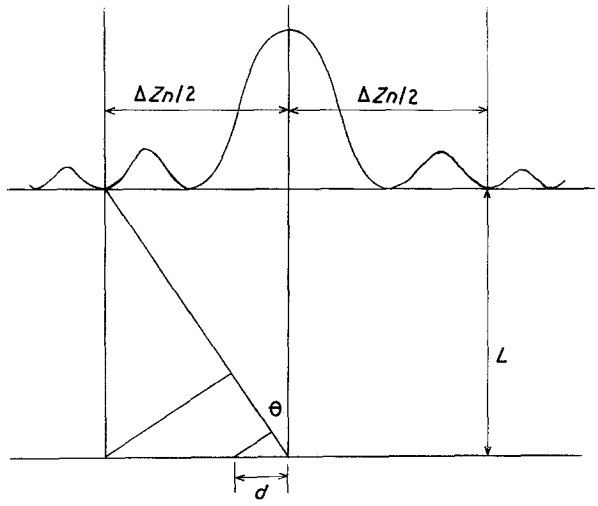

Figure 1 Derivation of laser diffraction equation

$$\sin\theta = \frac{n\lambda}{d} = \frac{\Delta Zn/2}{[L^2 + (\Delta Zn/2)^2]^{1/2}}$$

$$d = n\lambda\left[1 + \left(\frac{2L}{\Delta Zn}\right)^2\right]^{1/2}$$

of four movable stages including 360° rotation and three micrometer-controlled precision translations of up-and-down, parallel, and perpendicular to the laser beam. With the help of these movable stages, the sample can be readily placed in the beam at the focal point.

A graduated rail was situated perpendicular to the beam direction at a distance of 147.32 cm from the sample. A movable screen was used to locate the nodes or the minima in the diffraction pattern, and the node positions were noted by the rail graduations with vernier reading down to 0.1 mm. The fibre diameter can be readily calculated by the laser diffraction equation based upon the laser wavelength of 0.632 816 μm, the fibre-to-screen of 147.32 cm, and node-to-node distance which can be measured quite accurately.

In order to facilitate the taking of accurate data, many modifications were made on the current laser set-up. These modifications include blanking out the bright spot of the central maximum, using red light illumination for reading the node location scale, using a white frame on the card used to detect nodes, and the fabrication of adjustable sample holders where the sample is capable of a controlled adjustment in a plane perpendicular to the laser beam. This adjustment is a great help in the alignment of the sample with respect to the laser beam.

3. Calibration of laser method for accurate diameter measurement

3.1. Confirmation of the need for calibration

The laser equation is derived from the Fraunhofer slit approximation, thus a correction for the systematic error is needed to improve the accuracy of the laser diameter measurement. The fibre diameter is a function of laser wavelength, e.g. 0.632 816 μm for He–Ne laser, or six significant digits, distance of fibre from screen, e.g. 147.32 cm or five significant digits, and node length which is mostly four significant digits (cm). Thus, the fibre diameter should have four or at least three reliable significant digits, e.g. *xx.x* or *x.xx* μm. This means that an accuracy of 0.1 μm or better should be within the easy reach of the laser diameter measurement. This, however, is often not the case, showing the necessity of a systematic error correction.

3.2. General problems affecting diameter measurement

The accuracy of diameter measurement not only depends upon the type of measuring technique used, but also is greatly affected by the following factors.

3.2.1. Non-uniform fibre diameter within the gauge length

It is common to find non-uniform diameter along the one inch gauge length for almost all fibres including the commercial tungsten fibre. One way to compensate for such an affect on tensile properties is to

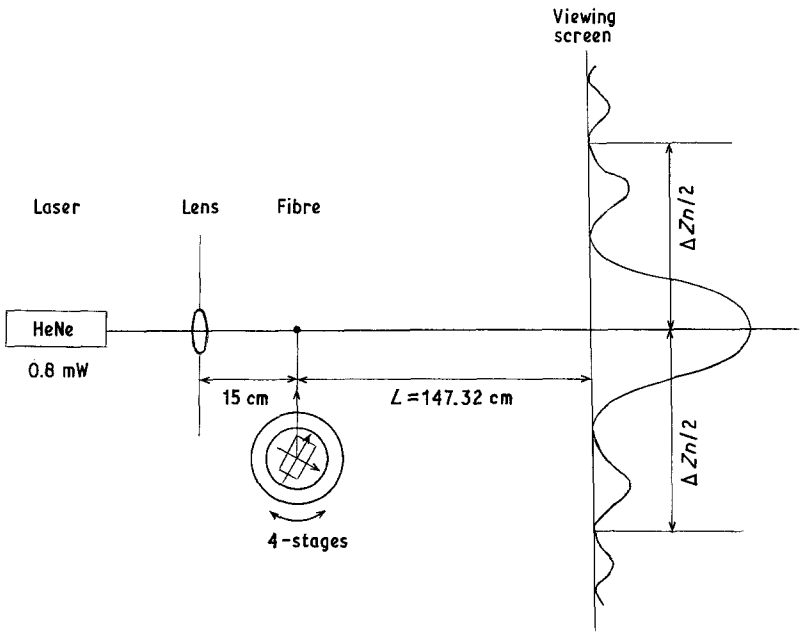

Figure 2 Laser diffraction set up at Dow Corning.

measure diameter at three or more different places along the one inch gauge length, e.g., middle and two ends and uses the average diameter for the calculation of tensile properties. In terms of preparing the SEM fibre standards to calibrate the laser method, a sharp marking on the mounting tab is needed so that both SEM and the laser are measuring the same diameter.

3.2.2. Debris on fibre surface

Debris on the fibre surface may contain dust particles, fibre fragments, glue balls, etc. Such debris will tend to make the diameter measurement somewhat larger and cause overlapping multiple laser diffraction patterns and fuzzy nodes. Such an unclean fibre location should be avoided by translating the fibre to another clean spot.

3.2.3. Non-circular cross-section

No diameter measurement method can produce an accurate diameter for a fibre having a non-circular cross-section. For example, a measurement of short axis or small diameter on an oblong fibre will make tensile strength arbitrarily higher, while a lower tensile strength will be obtained if the long axis or large diameter is measured. The tensile strength of an oblong fibre can, however, be correctly calculated by determination of the cross-section area or of the "equivalent" diameter obtained from a number of diameter measurements taken as the fibre is rotated about its longitudinal axis.

3.3. Preparation of calibration standards

In order to calibrate the laser technique, i.e., to determine the systematic error, six fibre standards were prepared as follows.

3.3.1. Range of fibre size

The six fibre standards were selected to have a diameter range from 5 to 28 μm, a range which should cover most of the small diameter fibres likely to be encountered. For larger fibres, laser diffraction would not offer a significant improvement in accuracy over optical microscopy.

3.3.2. Effect of non-circular and non-uniform size

The key to establishing an accurate calibration of the laser diffraction method is the clear identification of the locations at which diameter measurements by both laser and SEM will be made, in order to eliminate the effect of non-uniform diameter along the gauge length. To eliminate the possible effect of non-circular cross-section, the standard fibres were held in the same orientation to the SEM and the laser. Each fibre standard was clearly marked on the mounting tab at its centre and 2 mm each above and below the centre. These three positions were first measured by SEM and later repeated by laser technique.

3.3.3. SEM measurement

SEM is so far the method most capable of providing accurate diameter data, although it may take the longest time to do so. To obtain an accurate diameter measurement, the SEM micrograph of a fibre was calibrated with NBS-standard SRM484B. In addition, the fibre micrograph was taken at conditions as close to that of the NBS standard as possible, e.g., same magnification, same direction of measurement, same target to sample distance, same sample height, etc. The fibre standard is mounted on a one-piece paper tab, and is coated on both sides of the tab with gold. The standard is then mounted on the same sample holder with the NBS standard using two drops of carbon paint. It is noted that the 10 μm bar on a SEM micrograph cannot be used as a precise calibration standard because the distance will drift with time and may deviate as much as 6% from that of the NBS standard.

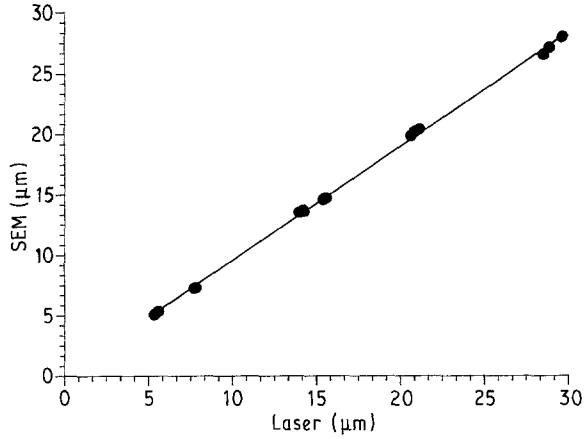

Figure 3 SEM plotted against laser diameter.

3.4. Calibration of laser technique

Each of the six fibre standards was measured at its midpoint and at 2 mm each above and below the centre where these three locations of measurement were clearly marked on the mounting tab. These six fibres were measured by the laser method at exactly the same locations as measured by SEM. The SEM data were found to have excellent correlation ($R \geqslant 0.9996$) with the laser data as shown in Table I, Fig. 3 and below.

$$\text{SEM} = 0.1084 + 0.9496\ (\text{Laser})\quad R = 0.9996$$

$$\text{SEM/Laser} = 0.95 \pm 0.01$$

The correlation equation has a slope of about 0.95 and an intercept close to zero, thus, the laser results are about 5% larger than the corresponding SEM values.

4. Verification of laser technique

A verification of the laser diffraction technique was obtained when the results of tensile testing of two HPZ fibres having very small diameters of 5 and 8 μm were shown to have very good agreement with those tests wherein diameters were accurately measured by non-laser methods. Fibre A was previously tested at SoRI (Southern Research Institute) using photomicrography to measure the diameter and was also tested at Dow Corning twice using the optical microscope and once using SEM (fractography) to measure diameter. Fibre B was tested at SoRI using photomicrography and also twice at Dow Corning using

TABLE I Calibration of laser apparatus

Fibre		n	Node Location (cm)			Diameter (μm)		
No	ID		High	Low	Net	Laser	SEM	SEM/Laser
W-5/C	W-Wire	2	100.09	29.35	70.74	5.42	5.17	0.95
W-5/−2		2	99.90	30.62	69.28	5.53	5.32	0.96
W-5/+2		1	82.77	47.50	35.27	5.32	5.05	0.95
01/C	HPZ-249							
01/−2	-1012F	2	89.37	40.28	49.09	7.70	7.24	0.94
01/+2		1	77.21	53.30	23.91	7.82	7.28	0.93
W-11/C	W-Wire	3	84.80	45.21	39.59	14.26	13.60	0.95
W-11/−2		3	85.32	45.00	40.32	14.00	13.51	0.97
W-11/+2		3	85.10	45.41	39.69	14.22	13.70	0.96
15/C	Nicalon	4	89.96	40.77	49.09	15.40	14.60	0.95
15/−2	-4768	4	89.75	40.89	48.86	15.47	14.70	0.95
15/+2		4	89.39	40.89	48.50	15.58	14.70	0.94
03/C	Fibre	4	83.00	47.35	35.65	21.08	20.4	0.97
03/−2	FP	4	83.39	46.97	36.42	20.64	19.9	0.96
03/+2		4	83.34	47.29	36.05	20.85	20.2	0.97
06/C	MPS-	6	84.77	45.66	39.11	28.86	27.15	0.94
06/−2	VI-119	6	84.81	45.21	39.60	28.50	26.6	0.93
06/+2		6	84.06	45.97	38.09	29.61	28.0	0.95
								0.95+/−0.01

optical microscopy. The tensile and modulus results obtained using diameters measured by SEM, photomicrograph, and laser all agreed closely (see Table II). The less accurate, larger diameter measured by the optical microscope translated into lower tensile and modulus values.

5. Conclusions

The conclusions are as follows.

(1) The laser diffraction equation is derived from the Fraunhofer slit approximation, thus a correction for the systematic error is needed in order to improve the accuracy of the laser diameter measurement.

(2) In order to correct the systematic laser error, internal standards should be used which are accurately calibrated by SEM and also clearly marked so that the same diameter locations and the same orientation to the source of incident radiation are measured by both laser and SEM to completely eliminate the effect of non-uniform and non-circular diameter along the length of fibre standards.

(3) The SEM data were found to correlate very well with the laser data. The correlation equation has a slope of about 0.95 and an intercept of close to zero, thus, the laser diameter is about 5% larger than the corresponding SEM diameter.

(4) After correction for the systematic error, the laser diffraction was found to be capable of meeting a diameter measurement goal of 0.1 μm accuracy with a short (5 min) measurement time. In addition to fibre diameter, the fibre cross-section can be also accurately determined by the laser diffraction technique.

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TABLE II A comparison of tensile test results between Dow Corning and SoRI for two small fibre samples

Test Location	No. Data Points	Diameter (μm)			Tensile Strength (MPa)	Elastic Modulus (GPa)
		Average	Range	Method		
(1) Fibre A						
SoRI	10	8.1 ± 1.2	7.0–10.8	PM	2082 ± 503	206 ± 23
DC	12	9.1 ± 1.3	8.1–12.7	OM	1586 ± 359	165 ± 32
DC	12	8.4 ± 1.3	6.8–12.2	OM	1786 ± 462	175 ± 43
DC	12	7.6 ± 1.4	6.0–11.4	SEM	2173 ± 510	212 ± 45
DC	10	7.4 ± 0.5	6.6–8.4	Laser	2090 ± 421	199 ± 26
(2) Fibre B						
SoRI	20	4.7 ± 0.3	4.3–5.2	PM	3331 ± 979	217 ± 29
DC	39	5.3 ± 0.3	4.8–5.9	OM	2876 ± 559	190 ± 23
DC	28	5.2 ± 0.3	4.8–5.6	OM	3028 ± 552	189 ± 23
DC	12	4.9 ± 0.2	4.4–5.2	Laser	3324 ± 1014	219 ± 35

SoRI Southern Research Institute
DC Dow Corning
PM Photomicrograph
OM Optical Microscope
SEM Scanning Electron Microscope

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