THE BELL SYSTEM TECHNICAL JOURNAL
Vol. 57, No. 6, July–August 1978
*Printed in U.S.A.*

***Atlanta Fiber System Experiment:***

# Reproducibility of Optical Fibers Prepared by a Modified Chemical Vapor Deposition Process*

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(Manuscript received January 21, 1977)

*The reproducibility of low-loss glass-fiber optical waveguides prepared from preforms produced by a "modified" chemical vapor deposition process was determined. The fibers have a multimode, graded-index core of germania borosilicate glasses and a cladding of fused silica. The quality of the fibers is expressed in terms of core/cladding dimensions, circularity, and concentricity, as well as loss spectrum, normalized index-of-refraction difference* $\Delta$*, and graded-index profile characteristic* $\alpha$*.*

## I. INTRODUCTION

A variety of multimode, low-loss glass fibers consisting of a core of germania borosilicate glasses and a cladding of fused quartz have been prepared over the past several years using the modified chemical vapor deposition (MCVD) process. The primary purpose has been to provide experimental glass fibers in response to in-house requests. Most of the early fibers used for prototype fiber optic cable studies leading to the Atlanta Fiber System Experiment[1] were prepared in this laboratory. The requests for fibers were seldom alike in terms of core/cladding dimensions, index profiles, index differences, and other properties. Although the conditions of preparation and properties of these fibers were monitored, it became desirable to establish the reproducibility of the process. Equally important, this would enable us to determine where and possibly how we should make improvements if needed.

* Part of this work was presented as a paper at the First European Conference on Optical Fiber Communication at London, The Institute of Electrical Engineers, September 16–18, 1975.

A series of fibers was to be fabricated on the basis of an arbitrarily chosen set of objectives listed in Table I. All process parameters selected for meeting these requirements were attempted to be held constant. The reproducibility achieved in these fibers is to be expressed in terms of properties such as core/cladding dimensions, loss spectrum, normalized index difference Δ, graded index profile characteristic $\alpha$, circularity, and core/cladding concentricity as well as their comparison to the objectives.

## II. PROCEDURE

The MCVD process,[2] as practiced for this study, is summarized here in conjunction with the schematic shown in Fig. 1.

A chemically cleaned tube (12 mm × 14 mm × 92 cm) of fused quartz is rotated in a glass working lathe. An oxy-hydrogen torch, while moving to the right at slow speed (0.35 cm/s), heats the tubing to ~1650°C. The torch returns to the left, at a faster speed (1.5 cm/s) and lower flame temperature, to complete one cycle. Throughout the torch cycle, controlled proportions of semiconducting grades of B, Ge, and Si chlorides in an oxygen carrier gas flow through the fused quartz tube. The deposition and simultaneous fusion of a thin layer of core glass occurs along the internal surface of the tube in the region of the torch as it moves from left to right only.

The flow conditions of the reactants at an ambient temperature of 23° to 25°C are listed in Table II. An initial layer of borosilicate glass is deposited during the first two torch cycles. The numbers in parentheses are the flow rates of oxygen through the bubblers containing the Ge and

Table I — Fiber property objectives

| | |
|---|---|
| Loss (0.82 μm) | ≤4 dB/km |
| Index profile, $\alpha$ | ~2.0 |
| Index difference, Δ | 0.013 |
| Core diameter | 55 μm |
| Fiber diameter | 110μm |

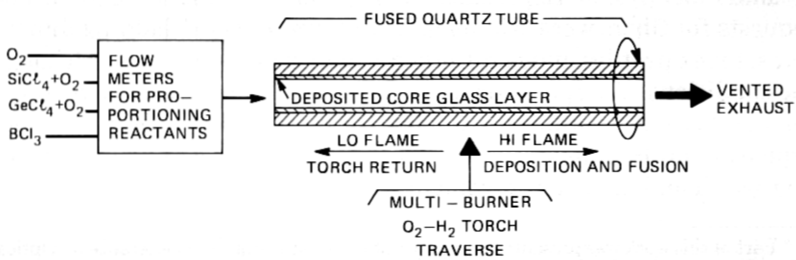



Fig. 1 — Schematic diagram of the modified chemical vapor deposition (MCVD) process.

Table II — Flow conditions of reactants (cc/min)

| Torch Cycles | $BCl_3$ | $SiCl_4$ | $GeCl_4$ | Additional $O_2$ |
|---|---|---|---|---|
| 2 | 24 | 81 (255) | — | 500 |
| 47 | 8 | 81 (255) | 4.5–47.4 (70–500) | 500 |

( ) = oxygen to bubblers.

Si tetrachlorides. To obtain the 2:1 diameter ratio of the cladding to core in the final fiber, an additional 47 torch cycles were required for depositing the germania borosilicate core glass. The flow rate of the $GeCl_4$ was increased automatically in 47 increments, or one per torch cycle, over the range from 4.5 to 47.4 cc/min. This provides the compositional gradient in the core glass required for the graded-index profile.

The fused quartz tubing is then collapsed into a solid rod within three additional torch cycles, at which time the speed of the torch is reduced (0.2, 0.1, and 0.03 cm/s) to raise the transient temperature of the tubing to the level of 1850° to 1950°C. During collapse, the flow of reactants was stopped except for oxygen until the last cycle of the torch. This produces a preform as seen in Fig. 2, measuring ~8 mm in diameter and 60 cm in length, that yields 3 km of 110-$\mu$m diameter fiber.

The preforms were drawn into fibers using the apparatus shown in Fig. 3. The basic components consist of a preform support and feed mechanism at the top, a high temperature heat source, a fiber diameter monitoring unit, and a winding mechanism at the bottom. An experimental coating system, shown just above the winding drum, was not used in this study. The overall assembly is mounted on a heavy aluminum frame measuring ~1.2 × 1.8 × 2.4 m.

The feed mechanism consists of a variable speed motor and control system* capable of providing less than 1 percent variation and full torque at low speeds of 0.5 to 1.5 cm/min. The heat source is a graphite resistance furnace† provided with a zirconium oxide‡ muffle tube and oper-

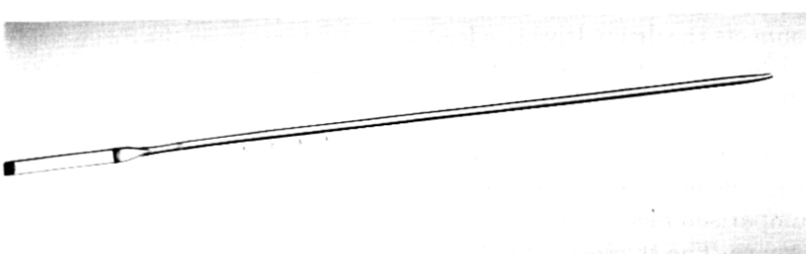


Fig. 2 — Collapsed glass preform obtained from the MCVD process (8 mm dia. × 60 cm).

* Motomatic E550M, B & B Motor and Control, N.Y., N.Y.
† Model 6000-2020, Astro Industries, Santa Barbara, California.
‡ No. 1706 Zircoa Div., Cohart Refractories, Solon, Ohio.

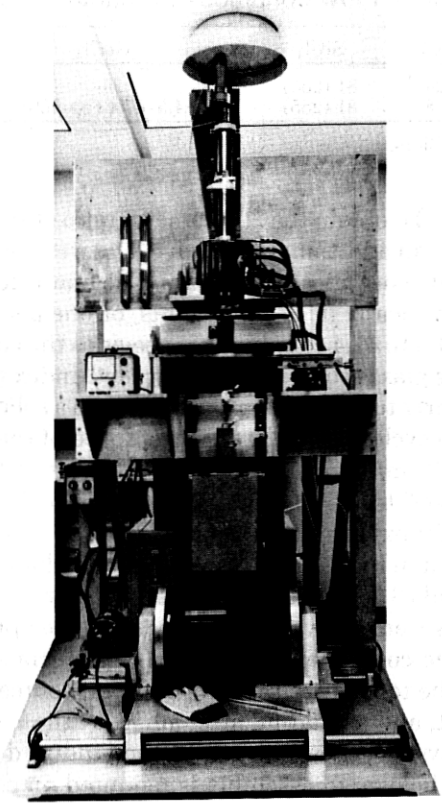


Fig. 3 — Overview of the laboratory fiber drawing facility.

ated with a 50/50 mixture by volume of He and Ar (8–10 L/min.) gases to prevent oxidation of the graphite heating element.

The winding mechanism consists of a black anodized aluminum drum (28 cm diam. × 45 cm) driven by a variable speed motor.* A gear belt connects the drum drive to a lead screw that traverses the drum to give a 0.30-mm (0.012-in.) pitch to the fiber winding. Typically, a 1100-m length of fiber is wound on a drum in a single layer. A drawing speed of 1 m/s was used in this work.

The diameter of the fiber was monitored during drawing by means of the device† mounted just below the furnace. It employs an optical comparison technique with the window located about 12 cm below the furnace. The shadow of the fiber is compared to a pre-set illuminated slit opening that has been calibrated previously. The size of the fiber

* Bodine NSH34RH, B and B Motor and Control, New York, N.Y.

† Model SSE-SR Milmaster, Electron-Machine Corp., Umatilla, Fla.

relative to the slit opening is indicated on a nullmeter having a precision of ±0.13 μm (±0.000005 inch). Normally, the preform feed speed and furnace temperature are held constant while the speed of the winding drum is adjusted manually to maintain a constant fiber diameter.

Initially, 18 lengths of fused quartz tubing* were selected at random. Prior to cleaning, each tube was numbered for identification by means of a diamond scribe at the designated left-hand end. An axial mark at the same end served to indicate the "north" orientation of the tubing when viewed from either end. The minimum and maximum outside diameter (O.D.) and wall thickness corresponding to the four main compass directions at either end were determined by means of a vernier caliper. Variations ranging in outside diameter from 0.02 to 0.13 mm and in wall thickness from 0 to 0.16 mm were found in the 18 lengths of tubing. In addition, the location and amount of the maximum warp along the length of the tubing was determined using a feeler gauge to measure the maximum clearance between the tubing and a ground stone reference plane as the tube was rotated about its axis. The warp was typically in the form of a bow that ranged from a minimum of 0.13 to a maximum of 0.61 mm. A further characterization of the quality of the fused quartz tubing was attempted by a cursory inspection for visual blemishes as revealed by illuminating the tubing from either end by means of a high-intensity fiber-optic lamp. The predominate type of blemish manifested itself as a bright speck of scattered light that resulted, in most cases, from small bubbles within the glass tubing. Several tubes contained <10 specks/in., while others ranged up to ~50 specks/in. In addition, a number of scratches, smears, and hazy areas were recorded for many of the tubes. No correlation has been established between the visual quality of the tubing and the quality of the resulting fiber.

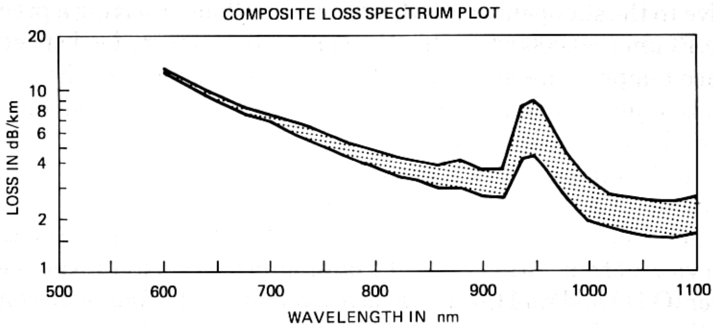
Fibers were prepared from 12 of the 18 lengths of tubing selected. Tubes numbered 2, 6, 13, 14, 15, and 17 were excluded from this study because of accidental breakage before preform preparation (nos. 2 and 6), excessive variation in wall thickness causing oval cores in the collapsed preforms (nos. 13, 14, and 15), and being classified as a spare preform (no. 17).

## III. RESULTS AND DISCUSSION

In this study, two 1-km lengths of fiber were drawn from each preform using the above drawing facility. The following data were obtained from an examination of these fibers and provide an indication of their quality and reproducibility.

Figure 4 is a composite semilog plot of the loss spectrum of a kilometer length of fiber from each of 11 preforms and two 1-km lengths from one other preform. The Arabic number identifies the preform, while the

* Commercial Grade T08, Amersil Corp., Sayreville, New Jersey.




OPTICAL LOSS IN dB/km

| PREFORM –FIBER | WAVELENGTH IN nm | | |
|---|---|---|---|
| | 820 | 900 | 1060 |
| 1-I | 3.9 | 3.0 | 1.9 |
| 3-I | 3.8 | 3.1 | 1.8 |
| 4-II | 3.5 | 2.6 | 1.5 |
| 5-II | 4.4 | 3.7 | 2.5 |
| 7-I | 4.1 | 3.5 | 2.0 |
| 8-I | 3.6 | 2.7 | 1.6 |
| 9-I | 3.9 | 3.0 | 1.8 |
| 10-I | 3.5 | 2.9 | 1.7 |
| 11-I | 3.7 | 2.9 | 1.6 |
| 11-II | 3.9 | 3.1 | 1.8 |
| 12-I | 3.8 | 2.9 | 1.6 |
| 16-II | 3.5 | 2.6 | 1.6 |
| 18-I | 3.7 | 3.0 | 1.8 |
| | (RMS = 3.8) | | |

Fig. 4 — Composite loss spectrum plot and tabulated loss values (13 1-km fibers among 12 preforms, $GeO_2$-$B_2O_3$-$SiO_2$ core and fused silica cladding).

Roman numeral refers to the first or second kilometer drawn from that preform. The curves fall within narrow limits. The corresponding tabulated values at wavelengths of 820, 900, and 1060 nm include minimum values of 3.5, 2.6, and 1.5 dB/km, respectively, with most of the fibers being within ½ dB or less of these minima. The RMS value at 820 nm is 3.8 dB/km.

The graded-index profile characteristic $\alpha$ and the normalized maximum index of refraction difference $\Delta$ were obtained by a technique developed by Wonsiewicz et al.[3] The method provides a computer-generated plot of the index profile in $\Delta n$ versus fiber radius as illustrated in Fig. 5. The data are obtained from the pattern of a thin, polished cross-section of a fiber as viewed in an interference microscope. A computer program also determines the value of $\alpha$ corresponding to the curve that fits the data best in accord with the basic equation shown in the diagram. The maximum normalized $\Delta$ is determined from the maximum $\Delta n$ of the plot. The dip at the center among the data points results from a depletion of Ge along the center of the fiber by volatilization during the collapsing of the preform. The existence of this condition has not been shown to be detrimental to the transmission characteristics of the fiber.

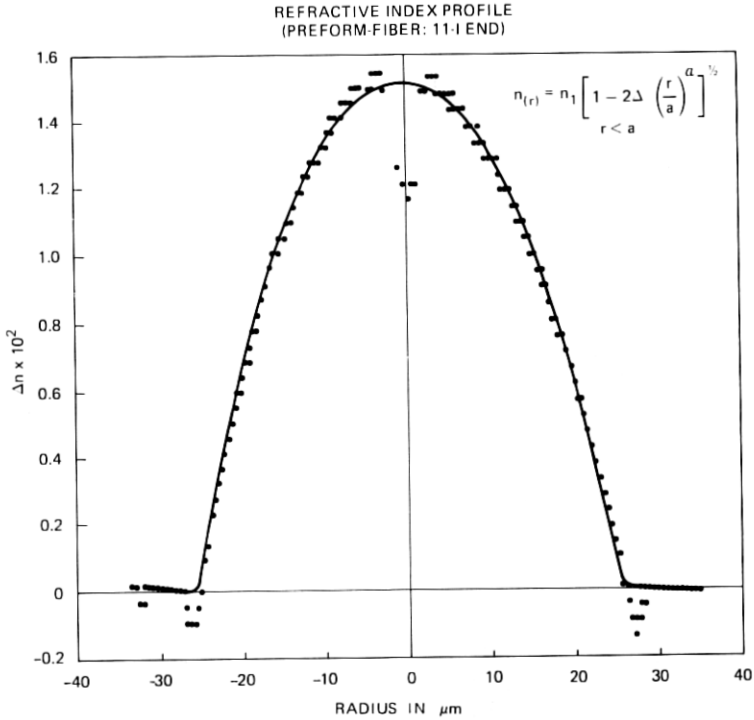



Fig. 5 — Index of refraction profile for preform—fiber: 11-I.

The dip at either edge of the profile corresponds to the borosilicate layer at the core cladding surface.

Figure 6 compares the range of values for $\alpha$ and $\Delta$ determined from "four" fiber samples within each preform, as indicated by the height of the bar, and among preforms. The RMS values for $\alpha$ were calculated to be 2.14 and for $\Delta$ to be 0.012. The precision of measurement for $\alpha$ is $\pm 0.05$ and for $\Delta$ is $\pm 0.001$. Although the observed $\alpha$ values are higher than intended, they could be corrected by adjusting the flow rates of the reactants and other parameters. The values for $\Delta$ in this study tend toward the low side for two reasons: (*i*) a decrease in the germania concentration at the center of the core and (*ii*) the thickness of the central region of the core of the thin section sample being less than the apparent value because of preferential abrasion during polishing.[4]

While the diameter of the fiber was monitored and controlled manually during drawing, it was measured subsequently at a precision of $\pm 1/4$ μm by an off-line forward light scattering technique described by Watkins.[5] The diameters along 556 m from each of several randomly selected fibers were determined at the rate of one measurement per meter. Figure 7 illustrates a typical linear plot of the diameter variation for such a length. The minimum, maximum, and average diameters and

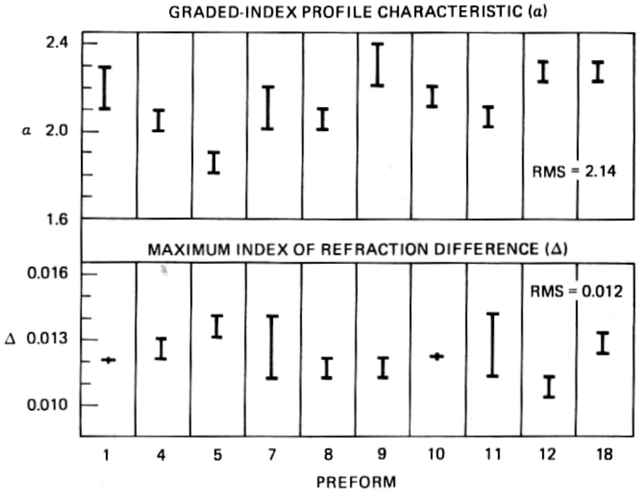
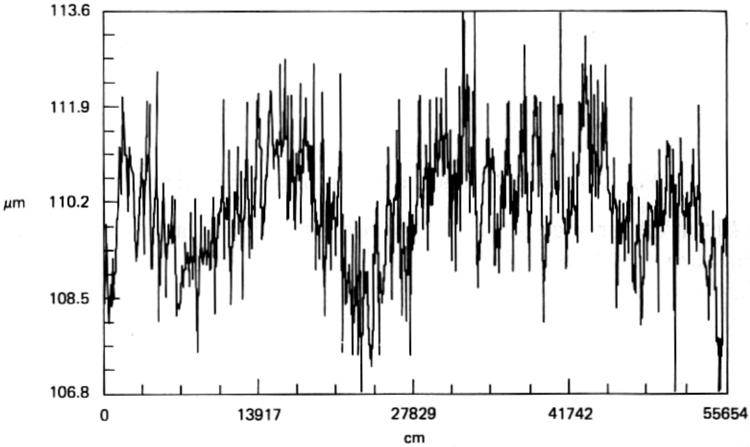



Fig. 6 — Graded-index profile characteristic ($\alpha$) and maximum index of refraction difference ($\Delta$) vs preform (interference microscopy; sample thickness ~50 $\mu$m).




| PREFORM —FIBER | MIN (μm) | MAX | AVG | % STND DEV |
|---|---|---|---|---|
| 5-1 | 107.0 | 113.1 | 110.2 | 1.02 |
| 8-1 | 105.7 | 113.2 | 109.7 | 1.30 |
| 11-1 | 104.8 | 114.5 | 109.6 | 1.72 |
| 12-1 | 105.7 | 112.5 | 109.0 | 1.09 |
| 12-2 | 104.9 | 113.5 | 109.7 | 1.45 |
| 16-1 | 106.7 | 115.0 | 109.7 | 1.13 |

Fig. 7 — Fiber diameter variation vs. 556-m length of preform—fiber: 5-I; and minimum, maximum, average, and percent standard deviation of fiber diameter within and among preforms.

the percent standard deviation within and among preforms are tabulated. Considering that diameter control was by manual feedback during drawing, the average values are relatively close to the objective of 110 $\mu$m.

The circularity and concentricity of the core and cladding are important properties related to fiber alignment when splicing. They were determined by microscopic examination of four fiber samples per preform corresponding to the positions shown in the diagram at the top of Fig. 8. The minimum and maximum diameters were determined by means of dividers and a calibrated micrometer scale from a photograph of a cross-section of the fiber, such as that shown on the left.

Circularity for either the core or cladding is expressed as the ratio of the minimum and maximum diameter of each. The concentricity of the core-cladding is defined as the separation ($\Delta cp$) expressed in micrometers between the center points formed by the intersection of the minimum and maximum diameters for each of the core and cladding.

In Fig. 9, the minimum-maximum diameter ratios of the core and cladding are shown for fiber samples among each of 12 preforms. Each dot represents a diameter ratio measurement. The numerical range of values observed for the diameters among the four samples within each preform is also shown. In general, the circularity of the cladding is better than that of the core both within a preform and among preforms. The circularity of the core becomes fixed, essentially during the collapsing stage of the preform, while that of the cladding may be upgraded during the drawing step. The circularity of the core and cladding vary independently of each other as seen in the case of preforms 4, 9, and 18. It was also observed that the variation in core circularity increases with the variation in wall thickness of the deposition tubing.

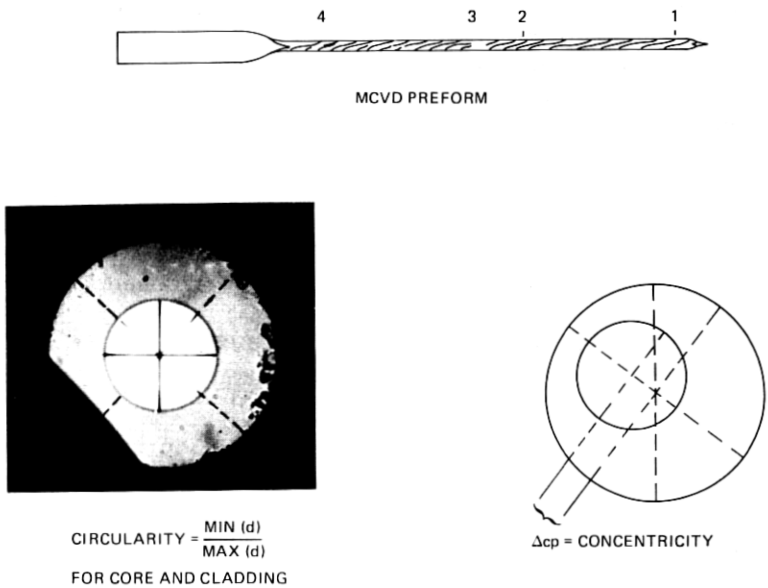



Fig. 8 — Core-cladding circularity and concentricity as defined and positions tested along preform.

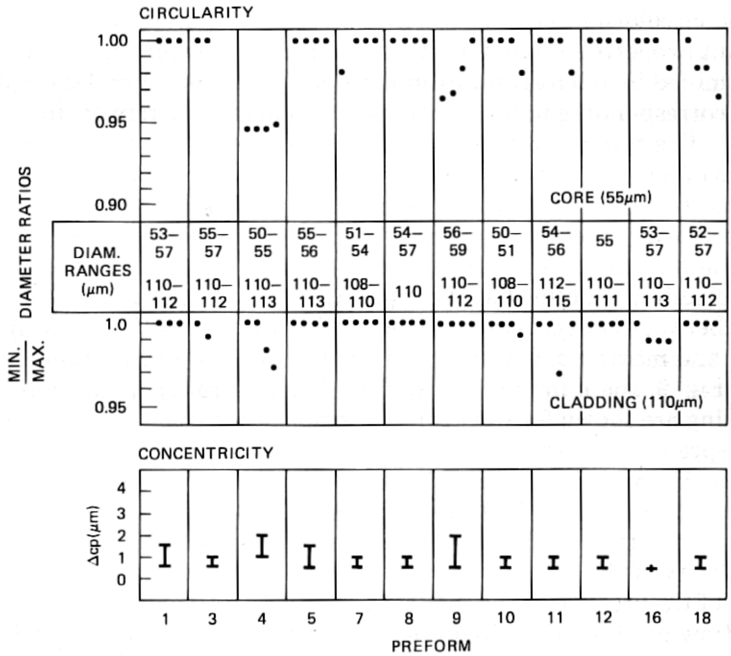



Fig. 9 — Circularity and concentricity of fiber core and cladding within and among preforms.

The concentricity values of the core-cladding (Fig. 9), represented by four determinations within each preform, are typically low in magnitude and range. The separation between center points is usually less than a micrometer, while the maxima of 2 micrometers occur with preforms exhibiting poorer circularity.

The objective and observed RMS values for the properties of interest in this study are compared in Table III. The table also summarizes the reproducibility of fibers prepared by a "modified" CVD process with our equipment. The percent standard deviations within a preform for loss, $\alpha$, and core diameter are about one-half that observed among preforms,

Table III — Reproducibility summary (MCVD:$GeO_2$-$B_2O_3$-$SiO_2$ core; fused quartz cladding)

| Property | Objective | Observed rms value | Percent standard deviation | |
|---|---|---|---|---|
| | | | Within preform | Among preforms |
| Optical loss (0.82 μm) | ≤4 dB/km | 3.8 | 2.5 | 6.5 |
| Index profile ($\alpha$) | ~2.0 | 2.14 | 3.0 | 6.0 |
| Index difference ($\Delta$) | 0.013 | 0.012 | 5.0 | 5.0 |
| Core diameter | 55 μm | 54.8 | 1.5 | 3.2 |
| Fiber diameter | 110 μm | 109.9 | 1.27 | 1.28 |

while the standard deviations for $\Delta$ and fiber diameter are about the same within and among preforms. The data indicate that the "modified" CVD process and equipment as discussed are capable of providing low-loss optical fibers of adequate quality and reproducibility for many applications. The following process and equipment modifications are suggested for enhancing the uniformity of dimensional and optical properties.

(*i*) There is a need for better dimensional uniformity and precision in the fused-quartz tubing. In particular, there is a marked dependency of core circularity upon the uniformity of the wall thickness of the preform tubing. A 3-percent variation appears to be the upper limit that can be tolerated for the change in wall thickness of the tubing used in this study and a 0.4-percent variation as the upper limit on the outside diameter of the tubing. The presence of blemishes of the type discussed earlier did not affect the optical properties of the fibers. However, they are recognized as possible factors affecting the strength of the fibers and are being included in other studies initiated recently.

(*ii*) Improved control of the MCVD reactant flows and a more uniform deposition temperature are expected to aid in achieving improvements to $\Delta$ and $\alpha$. These experiments have shown that maintaining a uniform deposition temperature is important for optimizing the reproducibility of the MCVD process. This applies not only to the temperature along the length of the silica tubing during each torch cycle as it traverses from left to right, but also throughout the deposition process of a preform and from preform to preform. Temperature variations of as much as 40° to 50°C were observed to have occurred at all of these stages in the course of conducting the above study.

In a subsequent series of experiments, a more uniform deposition temperature was attained by more careful monitoring and by frequent manual adjustments of the torch gases. This resulted in lowering the range of $\alpha$ values (for 14 samples from among three preforms) to within the precision of error of the measurement technique. These preliminary experiments indicate that electronically controlled flow valves for both the reactants and the torch gases as well as a feedback system to control the tube temperature would significantly improve the reproducibility of the process.

(*iii*) The incorporation of a capstan-drive, fiber-diameter monitoring by means of a laser and electronic feedback controls to the fiber drawing apparatus should improve control of the fiber diameter.

## IV. ACKNOWLEDGMENTS

The authors wish to express their appreciation for the assistance received in the following phases of the investigation from C. L. Collins and R. A. Becker for discussions about the statistical design of the experiments, M. Drohn for preform preparation, L. S. Watkins and P. H. Krawarik of Western Electric Engineering Research Center for diameter analysis, E. A. Sigety for polishing fiber cross sections, J. R. Simpson for spectrum loss, index, and profile data, and W. G. French for data processing.

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