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GROWTH AND CHARACTERIZATION OF OPTICAL WAVEGUIDES FOR
10.6 MICROMETER LIGHT

ARTHUR D. LITTLE, INCORPORATED

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13. ABSTRACT

The growth and subsequent optical characterization of single crystal Ge fibers for transmission at 10.6 μ is described. Single crystal Ge fibers 0.007 to 0.020 inch in diameter were grown by a laser-heated floating zone technique. The technique was shown to be effective for producing optical quality fibers with absorption coefficients as low as 0.06 cm⁻¹, or 0.26 db/cm at 10.6 μ .

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TABLE OF CONTENTS

	<u>Page</u>
List of Figures and Tables	vi
I. INTRODUCTION	1
II. MATERIALS SELECTION	2
A. Wave Propagation	2
B. Candidate Materials	3
III. FIBER GROWTH	6
A. The Laser Heated, Floating Zone Fiber Growth Process	6
B. Ge Fiber Growth	8
IV. OPTICAL EVALUATION	12
A. Fiber End Polishing	12
B. Apparatus for Fiber Optical Evaluation	12
C. Attenuation Coefficient	14
D. Numerical Aperture	15
E. Discussion	18
F. Conclusions	22
V. REFERENCES	23

LIST OF FIGURES AND TABLES

<u>Figure No.</u>		<u>Page</u>
1	Single Mode Core Radius as a Function of Core and Cladding Indices	4
2	Optical Bench Used in CO ₂ Laser-Heated Fiber Growth Process	7
3	0.012" Diameter C Axis Sapphire	9
4	Schematic Representation of Apparatus Utilized for Optical Attenuation and Numerical Aperture Measurements	13
5	Relative Transmission Data for Fibers Evaluated	16
6	Numerical Aperture Representation	19
7	Micrographs of Fiber Surfaces	21

<u>Table No.</u>		
1	Optically Tested Fibers	17

I. INTRODUCTION

There is a need for several specific types of fibrous optical waveguides. At one end of the spectrum are extremely low loss, single mode fibers intended for use as optical communication channels operating in the near infrared. For other applications, fibers will be used with existing high power lasers, such as CO₂ lasers, for laser radar and other applications. For these applications the fiber waveguides will not transmit information over long distances; thus, they need not have such low losses, but ideally should transmit only a single mode and be capable of withstanding extreme atmospheric conditions.

After surveying materials which would be satisfactory for transmitting light at 10.6μ, germanium was selected to demonstrate growth of fiber waveguides by the CO₂ laser heated fiber growth process. The growth procedures used for these fibers and the subsequent optical characterization of the Ge fibers is the subject of this report.

II. MATERIALS SELECTION

A. Wave Propagation

A transverse electric wave of successively higher order may be propagated in a fiber without loss of energy^{1,2} if

$$\lambda < \frac{\pi r}{N} \sqrt{\frac{\epsilon_c}{\epsilon_0}} \sqrt{\frac{\epsilon_f}{\epsilon_c} - 1} \quad (1)$$

where r is the core radius, ϵ_f , ϵ_c , and ϵ_0 are respectively the dielectric constants of the fiber core, the coating, and free space; λ is the wave length of light in free space and N is successively 2.40, 5.52, 8.05, 11.79, and 14.93 being the zeros of $J_0(y)$ (the zero order Bessel function of the first type). Thus, the first order transverse electric mode will be propagated and the second order mode will be radiated radially if,

$$\frac{2\pi r}{5.52} \sqrt{\frac{\epsilon_c}{\epsilon_0}} \sqrt{\frac{\epsilon_f}{\epsilon_c} - 1} < \lambda < \frac{2\pi r}{2.40} \sqrt{\frac{\epsilon_c}{\epsilon_0}} \sqrt{\frac{\epsilon_f}{\epsilon_c} - 1} \quad (2)$$

substituting

$$\sqrt{\frac{\epsilon_c}{\epsilon_0}} = n_c$$

$$\sqrt{\frac{\epsilon_f}{\epsilon_0} - 1} = \sqrt{\left(\frac{n_f^2}{n_c^2}\right) - 1}$$

and $n_f^2 - n_c^2 = (n_f + n_c)(n_f - n_c) \approx 2n\Delta n$ into Equation (2)

yields

$$\frac{2\pi r}{5.52} \sqrt{2n\Delta n} < \lambda < \frac{2\pi r}{2.40} \sqrt{2n\Delta n} \quad (3)$$

The dimensional limits of a single mode fiber as a function of the index level and the difference between the indices of the fiber core and the cladding are plotted in Figure 1. It is apparent that the core and cladding indices must be closely matched to be compatible with the dimensional limitations of the laser heated, fiber growing apparatus which was available for this program. This is more restrictive to crystalline fibers than glass fibers, since the minimum diameter crystalline fiber that can be grown controllably is approximately one-third to one-quarter the diffraction limited spot size of the existing heating optics (spot size ~ 0.006 inch). Glass fibers are drawn to diameters which are very much smaller than the laser-heated region since different stability criteria prevail in a glass drawing process. With crystalline compound or elemental semiconductors, it is unlikely that values of $n\Delta n$ smaller than 10^{-2} will be found. Thus, single mode fibers will probably require core radii smaller than 70μ (diameter <0.0027 inch). The best match found for Ge ($n = 4.0021$)³ is InSb ($n = 3.953$)⁴ giving a maximum permissible core radius of 17μ (diameter <0.00067 inch). An uncoated Ge fiber can have a radius no larger than 1.9μ to limit the transmitted signal to a single mode.

B. Candidate Materials

At the onset of the program, it was decided to limit candidate materials to single crystals because it was felt that optical quality single crystal fibers could be produced sooner than glass fibers. At that time, there was more processing experience with single crystals than glasses. It was also decided to weigh heavily factors relating to processability with the CO_2 laser heat source in making a materials selection. Based on these decisions, crystalline semiconductors were emphasized and two other classes of potential materials -- crystalline alkali halides and calcogenide glasses -- were eliminated from consideration.

The properties of candidate semiconductor materials were reviewed. From these, Ge was selected for the fiber growth experiments. The criteria used for materials selection included optical absorption coefficient at 10.6μ ,

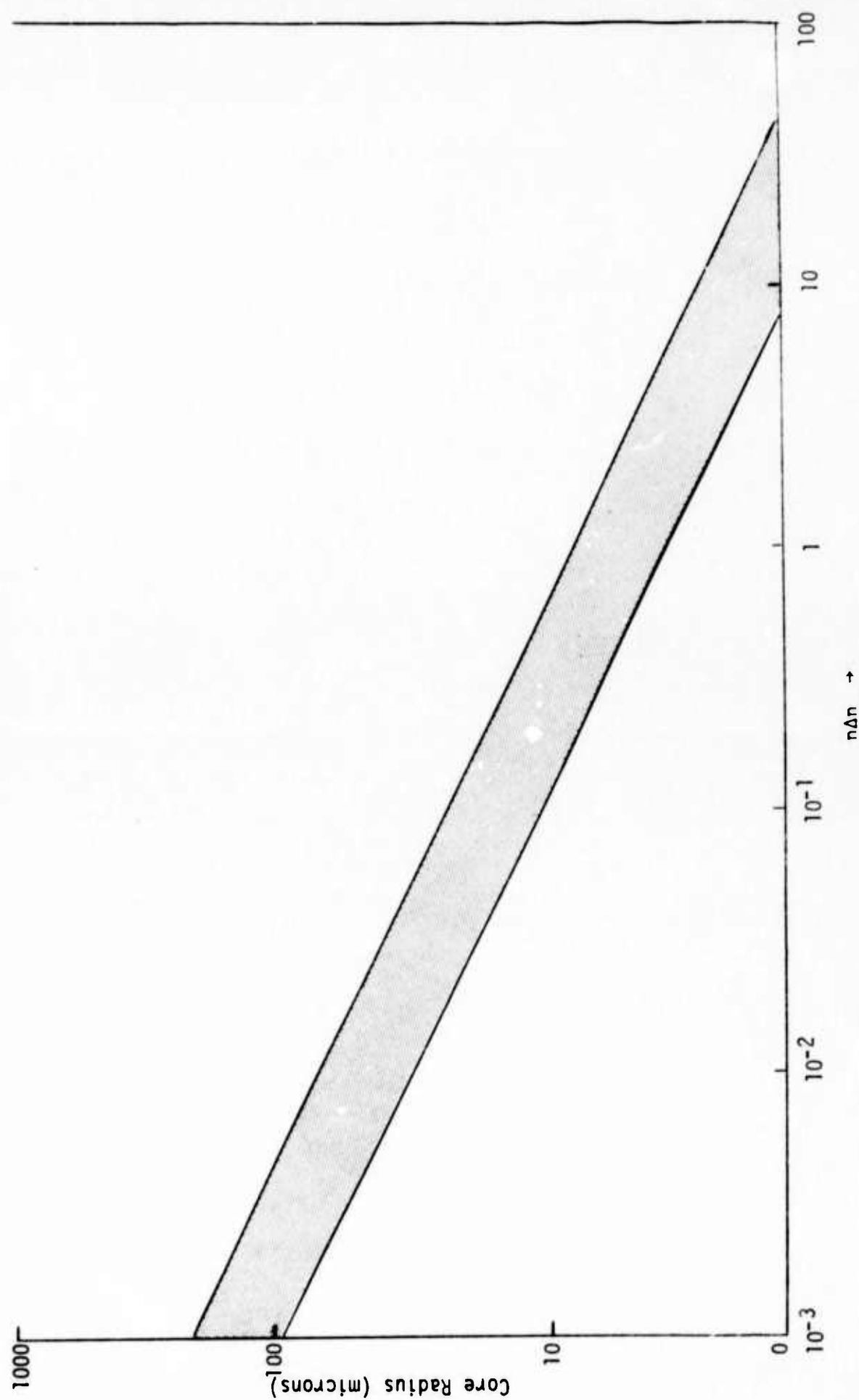


FIGURE 1 SINGLE MODE CORE RADIUS AS A FUNCTION OF CORE AND CLADDING INDICES

vapor pressure of the melt, congruent melting, absence of polymorphic phase transitions, chemical stability, strength, toxicity and availability. Vapor pressure of the melts eliminated many materials from consideration. Materials such as GaAs can probably be grown by the process, but major equipment changes would be required to operate in an As atmosphere.

Germanium appeared the singular choice of the elemental or compound semiconductors for this program. It can be heated easily with 10.6 μ m radiation once thermally induced free carrier absorption (thermal runaway)⁵ becomes significant. It has a low vapor pressure ($P \leq 10^{-6}$ atm) at its melting point and has no solid state phase transformations. Room temperature 10.6 μ m optical absorptivities on the order of $1.7 \times 10^{-2} \text{ cm}^{-1}$ can be achieved⁶ so it should make an acceptable optical waveguide provided good surface finishes can be maintained.

Optical attenuation coefficients of semiconductor or single crystal fibers cannot reasonably be expected to reach the levels exhibited by good silicate glass fibers with nominally 1 μ m wavelength light. The bulk absorptivities of semiconductors are generally in the range of 10^{-3} to 10^{-2} cm^{-1} for 10.6 μ m light.⁶ Thus attenuation coefficients of the order of 5×10^3 db/km would be considered optimistic for a semiconductor optical waveguide for 10.6 μ m light.

III. FIBER GROWTH

A. The Laser Heated, Floating Zone Fiber Growth Process

The process used for growing the 10.6 μ m optical waveguides is based on techniques and apparatus developed at ADL. The analytical and physical descriptions of the process are given in previously published NASA reports,^{7,8} thus will only be described briefly here.

Fibers are grown from a CO₂ laser heated, floating zone melt which is supported between the feed rod and fiber by the surface tension of the liquid. High purities are possible, since the melt does not come into contact with foreign materials. The CO₂ laser heat source permits virtually complete freedom in selection of ambient atmospheres. This is used to advantage in this program for the elimination of impurities.

The CO₂ laser beam is expanded to two diameters by a lens and mirror combination and divided into two beams with a dielectric coated GaAs beam splitter. The two beams are then brought into the optical bench shown in Figure 2. This optical bench physically divides each of the two circular beams into two semicircular beams. The four semicircular beams are deflected to 5-inch focal length spherical mirrors which focus the beams onto the region of the floating zone melt. Energy density can be adjusted by varying the zone to mirror distance and the laser power output.

With small diameter fibers, the surface tension forces exceed gravity forces to an extent that the zone shape is not affected by the direction of pull. Up, down or horizontal growth processes have the same zone shape. Downpull processes generally produce better quality fibers, since gas bubbles in the molten zone tend to migrate away from the solidification interface. Also, there is less condensation on surfaces of the fibers.

Feed rods are inserted and fibers withdrawn by means of crystal pulling heads. The maximum fiber length is approximately 8 inches with the existing fiber withdrawal mechanism. A completely continuous process is feasible, but it has not been developed.

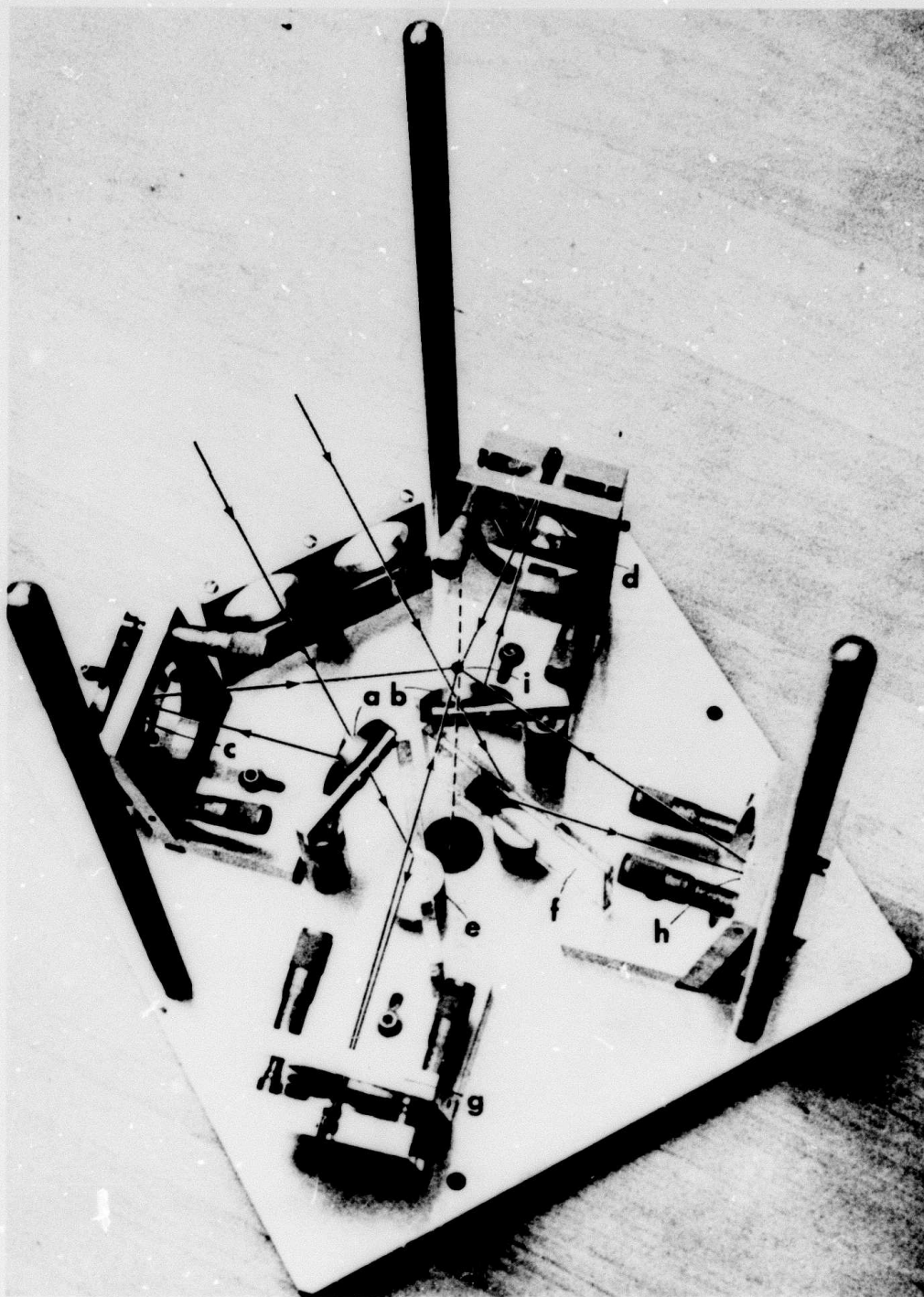


FIGURE 2 OPTICAL BENCH USED IN CO₂ LASER-HEATED FIBER GROWTH PROCESS
Input Rays are Divided by Mirrors a and b and Ultimately
Focused on i by Mirrors c, d, g and h.

The crystallographic orientation of the fibers is readily defined. Seeding is done in a conventional manner by initiating growth from an oriented single crystal. The orientation may be established by sectioning an oriented seed from a crystal or initiating growth from a single crystal fiber mounted in a goniometer. The latter technique is generally used.

The diameter of the fiber is controlled by adjusting the relative insertion and withdrawal rates. Fiber diameter variations are caused primarily by variations of the mass flow rate of the feed into the molten zone due to density and dimension variations of the feed rod. It has been found that fiber diameters are more uniform than the feed because an averaging process occurs during growth. The axial diameter variation of a nominally 0.040 inch sapphire fiber will typically be of the order of ± 0.0001 inch. The single crystal fibers are not usually round, as shown in Figure 3. The hexagonal cross-section of this c axis sapphire results from the development of prismatic growth facets. This tendency to facet appears general; however, it becomes less apparent as fiber diameters are reduced.

B. Ge Fiber Growth

The growth of optical quality Ge fibers by the laser heated, floating zone technique presented unexpected difficulties due to the presence of inclusions and a persistent surface film which often appeared during growth. Hence, a large part of the effort was directed at bulk purification and removal or prevention of this surface film.

Initially, a germanium boule purchased from United Mineral and Chemical Corp., New York, N.Y., was used as feed. It was cut, rounded by grinding, and then etched in CP4. Multiple zone refining pulling runs with up to six passes did not yield satisfactory feed material for growth of optical quality fibers. In an effort to purify the germanium, the UM and C material was remelted and directionally solidified in a vitreous carbon boat under an oxygen partial pressure of 10^{-19} ppm. The feed from this material again did not yield satisfactory fibers. In a further effort to purify the starting germanium, a liquid encapsulation Czochralski run was



FIGURE 3 0.012" DIAMETER C AXIS SAPPHIRE

made in which a germanium crystal boule was grown from the melt through a molten layer of B_2O_3 . It has been reported that the molten B_2O_3 acts as a getter for oxygen impurities.⁹ The B_2O_3 treated material had lower than intrinsic electrical resistivity (approximately 8 ohm-cm) and bulk transmission was reduced by a factor of 3 at $10.6\mu m$; therefore, this material was not converted to fibers.

Next, an optical grade germanium from Eagle Pitcher Industries, Inc., Quapaw, Oklahoma was used for fiber growth. The as-received crystal was cut axially into 0.125×0.125 inch cross-section bars which were rounded to cylindrical shapes by grinding. They were then etched in CP4, washed with distilled water, re-etched in an oxide etch,* washed in distilled water, and finally rinsed in methanol. Etching of feed rods cut from the as-received Eagle Pitcher crystals revealed regions of high impurity content. Chemical analysis of these regions indicated carbon content of approximately 100 times and oxygen content of approximately 50 times the bulk concentrations for the remaining sections of the rod. Zone refining runs with these materials resulted in a zone leveling mode of growth with these impurities being distributed throughout the grown crystal. To avoid this, feed rods were grown from selection sections of the as-received stock.

The initial zone refining run was carried out in a flowing atmosphere of Ar-10% H_2 at 2-3 psig. Previous to the run, the furnace chamber was evacuated to a pressure less than 25 microns Hg, backfilled with argon and then re-evacuated. The Ar-10% H_2 which was used for the run was passed through a "deoxo" unit built by Engelhard Industries, Newark, N.J., and then a liquid nitrogen cold trap before entering the furnace chamber. A gas flow rate ($6-7 \text{ ft}^3/\text{hr}$) was maintained during the run and the exiting gas was continually analyzed for oxygen content. Oxygen partial pressures were monitored with a "Thermox Meter" built by Thermo-Lab Instruments, Inc.,

*U.S. 3,107,188 (Cl.156-17). The oxide etch contained
8g HF, 50.3g H_2O , 186g H_3PO_4 , and 103g NH_5F_2 .

Glenshaw, Penna. In addition to the pretreatment of the gas, a tantalum oxygen getter was run in the fiber growth chamber during zone refining. Throughout the course of the run, the oxygen partial pressure was maintained at less than 10^{-19} ppm. The initial pass zone refining was made at approximately one inch per hour with the growing crystal being rotated at 100 rpm.

The zone refined rod had a significant amount of second phase particulates which accumulated at surface facets on the crystal. The larger of these were removed by grinding. Next, a double etching and washing procedure was carried out to remove any additional apparent second phase material. The initial etch was with CP4 and the second etch was with an oxide etch. Washing was with distilled water and after the final wash, a methanol rinse was employed.

Two additional zone refining runs with diameter reductions were carried out with the same post-growth etching and cleaning procedures being followed. This yielded feed rods from which satisfactory optical fibers could be grown.

The optical fibers were grown under the same atmospheric conditions as the feed rods or under a static atmosphere of the same gas. In either case, the Ar-10% H₂ underwent the same scrubbing procedures and the Ta oxygen getter was used during the runs. The fibers were grown without rotation from a <100> oriented seed crystal at rates ranging from 3.4 to 6.8 inches per hour at attenuation ratios ranging from 10-25 to 1. The fiber diameters ranged from 0.007 to 0.020 inch.

IV. OPTICAL EVALUATION

A. Fiber End Polishing

The fibers were prepared for optical characterization by polishing the ends. This was accomplished with the aid of a polishing jig in which the fibers were supported in wax. After initial grinding, the fibers were polished with 30 micron SiC on nylon with distilled water and a soap used to prevent particle agglomeration. The final polish with 0.06 micron Al_2O_3 was also on nylon with distilled water and soap. It yielded a surface which was not completely scratch-free, but represented a trade-off between rounding and achieving an adequate surface finish. An improved polishing technique should improve the apparent transmission of the fibers.

B. Apparatus for Fiber Optical Evaluation

Numerical aperture and attenuation coefficients were determined with the apparatus shown schematically in Figure 4. Chopped radiation is focused onto the fibers. All exiting radiation is collected with a spherical reflector which in turn focuses it onto a detector which measures its intensity utilizing a phase lock amplifier. During optical evaluation, the apparatus is covered with a black cloth to prevent visible radiation from striking the fiber. Germanium fibers become virtually opaque in a lighted environment.

The following points describe the specific components in this apparatus.

1. The IR source is a Perkin-Elmer No. 4570244 Mounted Source Assembly which employs a ceramic-coated resistance wire heated to approximately 1100°C.
2. An aperture is used at the exit of the source to minimize the light scattered into the system.
3. The light is modulated at 36.5 Hertz with a mechanical chopping wheel.

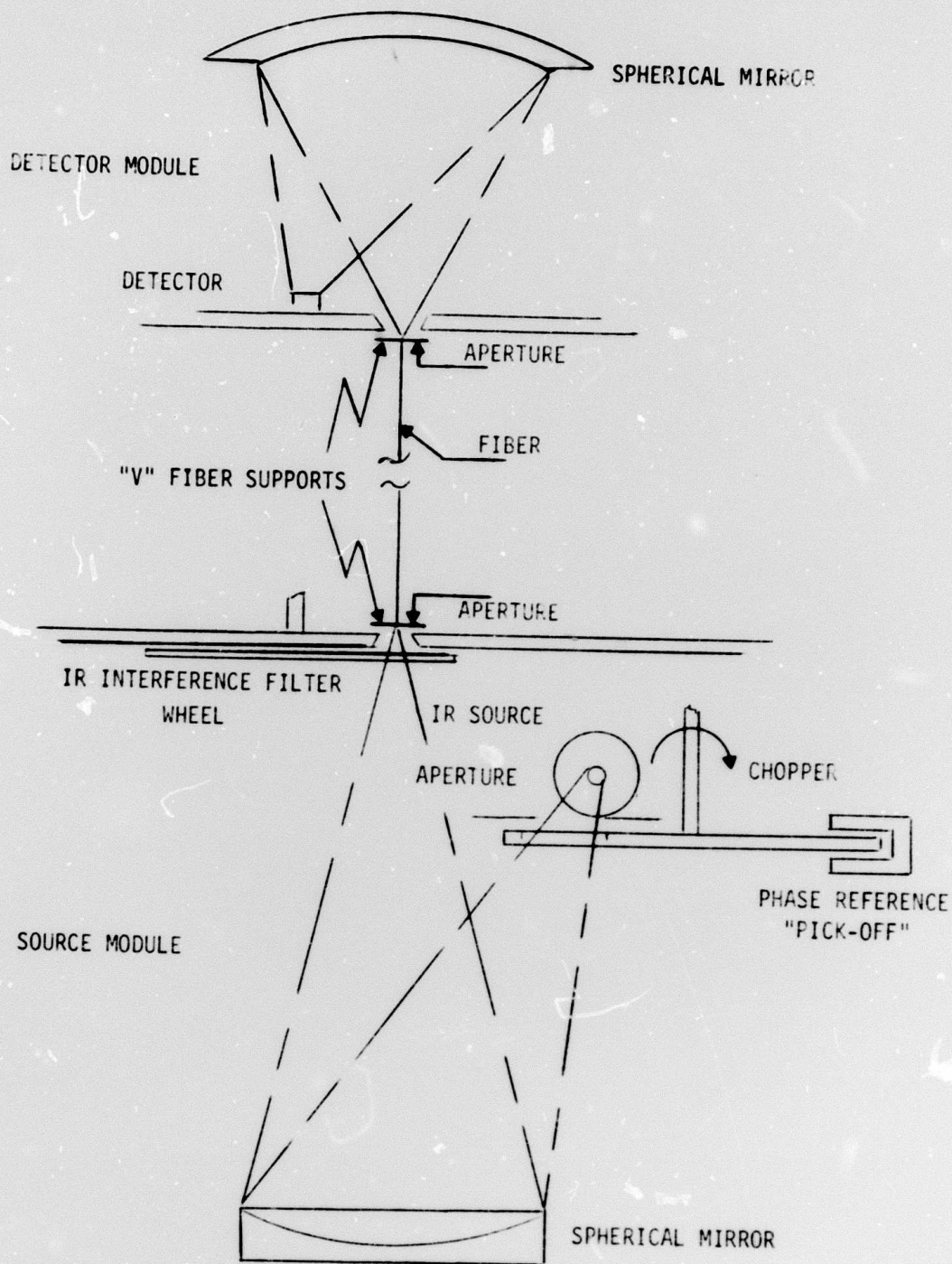


FIGURE 4 SCHEMATIC REPRESENTATION OF APPARATUS UTILIZED FOR OPTICAL ATTENUATION AND NUMERICAL APERTURE MEASUREMENTS

4. A 6-inch diameter, 6-inch focal length mirror is used to focus the emitted radiation onto the entrance end of the fibers. The entrance cone angle is approximately 30° .
5. The wavelength of the incident radiation is defined by filters. Two-tenths micron bandwidth filters centered at $3.5\mu\text{m}$ and $10.6\mu\text{m}$ have been selected. An open position is used for alignment.
6. The radiation exiting the fibers is captured by a 6-1/2 inch diameter, 3 inch focal length mirror and focused onto a detector. The acceptance angle of the detector module is approximately 55° .
7. A large area pyroelectric detector (~ 3 mm square) is used to compensate for aberrations caused by positioning the detector off axis as well as misalignment.
8. The signal from the detector is measured with a phase-lock amplifier whose reference is set by an optical "pick-off" on the chopper wheel.
9. The fibers are supported at each end by "V" shaped jigs.

C. Attenuation Coefficient

The attenuation coefficient is determined by measuring the light transmitted through the fibers as a function of length. In this work, transmission for a series of fibers of varying lengths and cross sectional areas was measured. The transmission data was normalized and a relative transmission was calculated as follows:

$$T_R = \frac{T_f/A_f}{T_{w/o}/A_d}$$

T_R = Relative transmission

T_f = Transmission of fiber (mV)

A_f = Area of Fiber (cm^2)

$T_{w/o}$ = Transmission without fiber (mV)

A_d = Effective area of the detector (cm^2)

The attenuation coefficient is determined from the slope of a line representing the relative transmission versus fiber length on a semi-log plot. Data for the fibers evaluated are presented in Figure 5 and the tested fiber dimensions are given in Table 1.

An absorption coefficient calculated from the plot of all the data yielded a value of approximately 0.15 cm^{-1} or 0.67 db/cm calculated as $10 \log I/I_0$. If the two sections of sample 31-1 which gave the best transmission data were used, an absorption coefficient of 0.065 cm^{-1} or 0.26 db/cm is obtained. These values compare favorably with bulk absorptivity values obtained from the literature which ranged from $0.02\text{-}0.03 \text{ cm}^{-1}$ at $10.6 \mu\text{m}$.⁶

D. Numerical Aperture

An apparent numerical aperture for these fibers was determined by selectively measuring the light transmitted through a fiber as a function of the angle of incidence measured relative to the fiber axis. The center section of the source mirror was blocked with successive discs increasing incrementally by 1 inch diameters and transmission values were measured. Transmission values for a concentric annulus around the fiber axis were

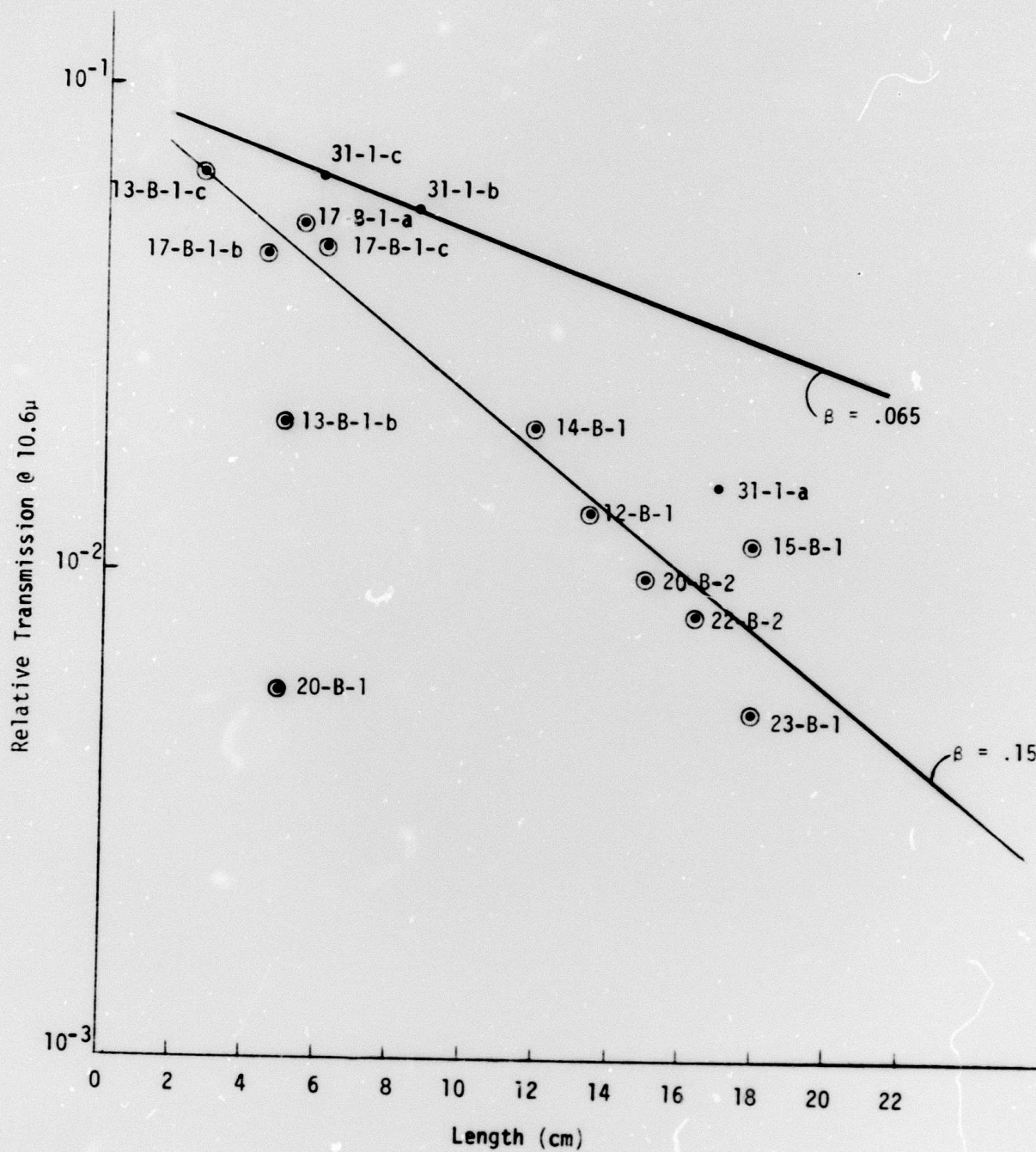


FIGURE 5 RELATIVE TRANSMISSION DATA FOR FIBERS EVALUATED

TABLE 1
OPTICALLY TESTED FIBERS

	<u>Diameter</u> (cm)	<u>Length</u> (cm)
12-B-1	.061	13.5
13-B-1	.043	12.2
13-B-1-b	.043	4.9
13-B-1-c	.043	2.7
14-B-1	.045	11.9
15-B-1	.033	17.8
17-B-1-a	.036	5.4
17-B-1-b	.036	4.4
17-B-1-c	.036	6.1
20-B-1	.017-.018	4.8
20-B-2	.028	14.9
22-B-2	.033	16.2
23-B-1	.033	17.9
31-1-a	.045	17.8
31-1-b	.045	8.5
31-1-c	.045	5.9

then determined by subtracting successive measurements. The angle included in each annulus was 2 to 3°. A relative transmission value was then determined for each annulus as follows:

$$T_{R,\theta} = \Delta T_{\theta,f} / \Delta T_{\theta,w/o}$$

$$T_{R,\theta} = \text{Relative transmission}$$

$$\Delta T_{\theta,f} = \text{Signal for cone } \Delta\theta_i \text{ with fiber (mv)}$$

$$\Delta T_{\theta,w/o} = \text{Signal for cone } \Delta\theta_i \text{ without fiber (mv)}$$

The relative transmission values for each fiber were then normalized to the largest relative transmission value.

The normalized $T_{R,\theta}$ data for several fibers is plotted as a function of the annular cone angle in Figure 6. The annular cone angles plotted correspond to the weighted centers of the 2-3° annuli. Half of the cone area lies on either side of the cited angle. Plotted data runs from zero to 12° which is the practical limit for measurements with the apparatus employed. For the best fibers transmission versus angle showed only a small drop out to 12° from the fiber axis. For others, the transmission dropped to approximately 15% at 8° from the fiber axis.

E. Discussion

Optical attenuation in the waveguide occurs due to bulk absorption in the core, absorption of the evanescent wave in the cladding, anomalous absorption or scattering losses at the cladding-core interface (or free interface if unclad), scattering sites in either core or cladding and leakage of the evanescent wave to adjacent dielectrics. The numerical aperture and attenuation coefficient measurements showed that major fractions of the transmitted light was lost at specific points in the fibers. Fiber 31-1 was evaluated at lengths of 17.78 cm, 8.51 cm and 5.91 cm. The transmitted

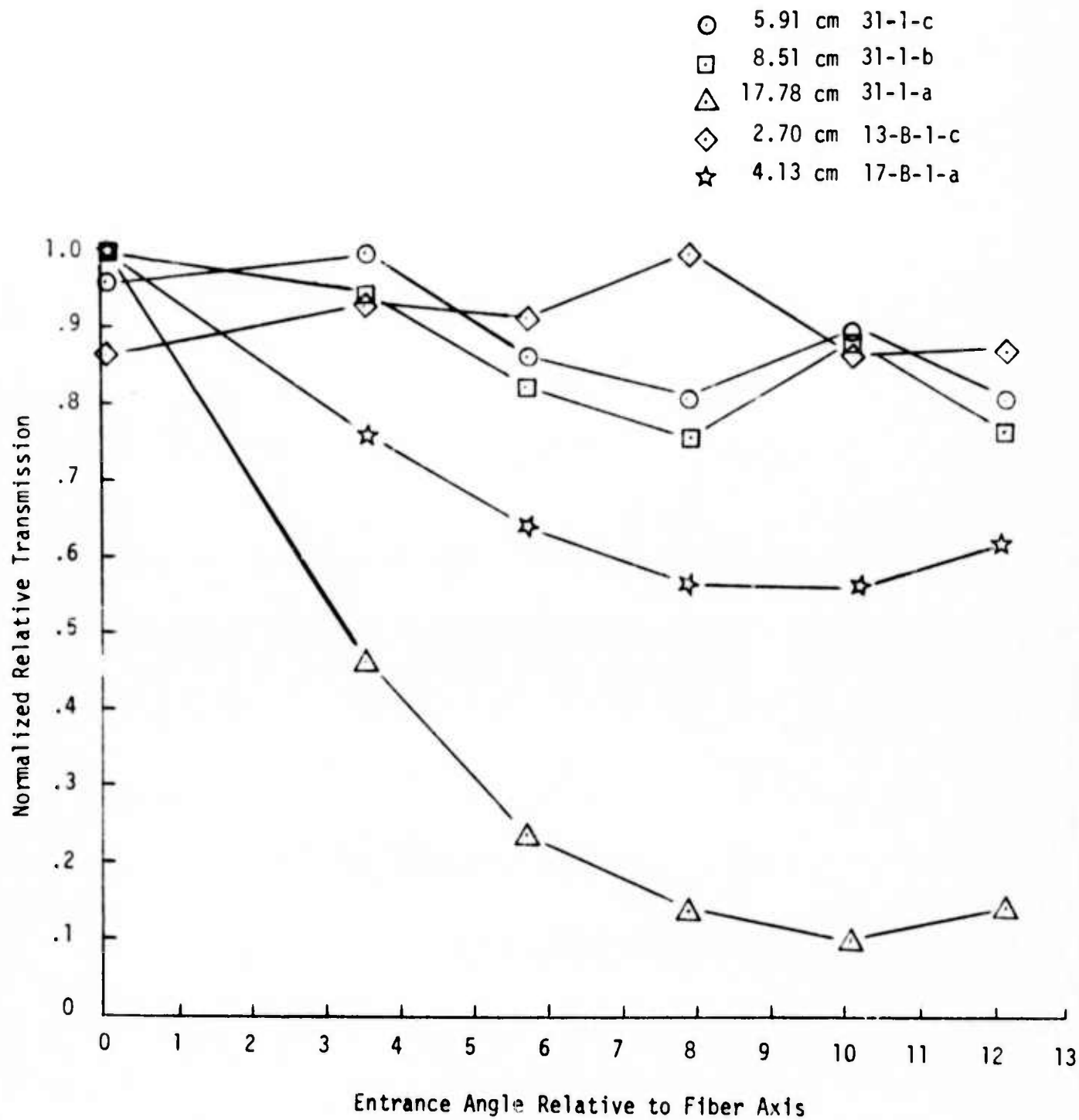


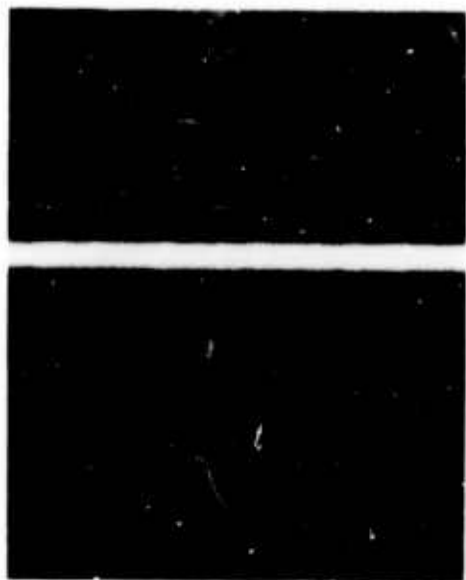
FIGURE 6 NUMERICAL APERTURE REPRESENTATION

signal through the 17.78 cm length was approximately 3 times lower than the value expected from extrapolating the results for the two shorter sections. The full length section exhibited an approximate 85% decrease in transmitted signal at 12° angle of incidence while there was a 10-15% drop in the relative transmission versus input angle for shorter sections. Other fibers exhibited similar trends: those fibers which showed large decreases in relative transmission versus input angle exhibited low total transmissions. It is felt that the localized losses result from surface defects.

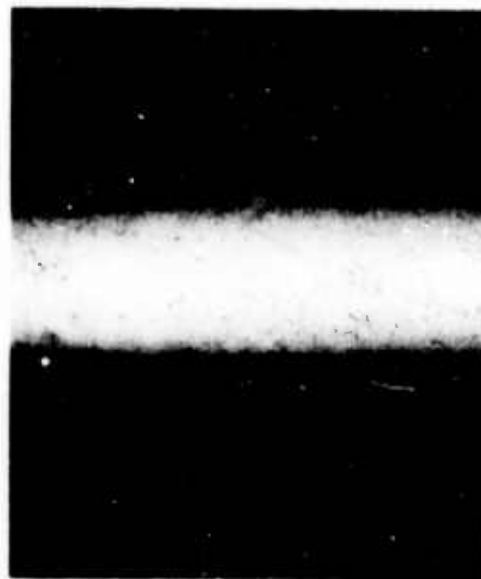
Microscopic examination of the Ge fibers revealed surface defects which appear to be embedded particles. The size and concentration of these defects varied; however, they were found on most fibers. It is probable that these surface defects result from the impurity which caused the large defects at facets on the initial fiber growth runs from the as-received feed stock. Repeated zone melting apparently reduced, but did not eliminate, the impurity.

Fiber 17-B-1-a exhibited average total transmission. The photomicrograph of the fiber shows that the surface is smooth and facet lines are straight (Figure 7A) but second phase particles are embedded in the fiber surface (Figure 7B). Photomicrographs of fiber 22-B-2, which also exhibited lower than average transmission had larger second phase particles (Figure 7C). Other types of defects, such as the growth defect shown in Figure 7D for fiber 13-B-1-b, contributed to low overall transmission. It is felt that the surface defects probably accounted for the difference between the best absorptivities measured on the fibers ($\sim 0.065 \text{ cm}^{-1}$ @ $10.6 \mu\text{m}$) and the best bulk absorptivities reported for Ge ($\sim 0.02 - 0.03 \text{ cm}^{-1}$ @ $10.6 \mu\text{m}$). The poor transmission values obtained for fibers 13-B-1-b and 20-B-1 resulted primarily from growth defects.

Although it is likely that the surface defects are responsible for the difference between observed and bulk absorptivities, it is not known whether the losses result from absorption or scattering mechanisms. The



A. Section of Fiber 17-B-1-a
(100X)



B. Surface of Fiber 17-B-1-a
(200X)



C. Surface of Fiber 20-B-2
(200X)



D. Growth Defect in Fiber 13-B-1-b
(100X)

FIGURE 7 MICROGRAPHS OF FIBER SURFACES

high index of Ge makes radial leakage minimal but does make total internal reflection at the ends likely. Any ray which strikes a Ge-air surface at an angle of incidence greater than 14.5° will be totally reflected into the fiber. Thus, any ray whose path deviates from ideal, whether caused by scattering at a surface particle or intercepting a surface irregularity, may become entrapped in the fiber lowering the measured transmission. This means the quality of the as-grown surfaces must be maximized since claddings will not improve transmission when the principal loss mechanism is surface defects on the core.

F. Conclusions

Single crystal germanium fibers 0.007 to 0.020 inch in diameter have been grown successfully by the CO_2 laser-heated, floating zone fiber growth process. Optical absorption measurements with the fibers indicated that overall transmissions at $10.6\mu\text{m}$ were within a factor of 2 - 3 of the best bulk absorptivities reported for Ge. The fibers are useful for $10.6\mu\text{m}$ light pipes; however, their transmitted mode structures were not investigated. Optical losses appear to be caused by occasional growth defects and a persistent surface defect. By selecting feed stock and carefully adhering to determined preparation and growth procedures, the concentration of the surface defects were reduced significantly. Fibers grown with the final processing conditions exhibited lower concentration of defects and improved transmissions.

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