RECEIVED

MAY 25 1995

OSTI

FINAL PROJECT REPORT
JUNE, 1992

PROJECT NUMBER: 9-XTI-W7962-1

TITLE: A NEW U.S. MANUFACTURING CAPABILITY OF GLASS PREFORMS FOR
FIBER OPTICS IN DEFENSE PROGRAMS

SUBCONTRACTOR: POLYMICRO TECHNOLOGIES, INC.
PHOENIX, ARIZONA

PRINCIPAL INVESTIGATOR: DILIP K. NATH
(602) 272-7437

SUMMARY

The present project is a part of the program to develop "A New U.S. Manufacturing Capability of Fiber Optics in Defense Programs." The scope of the program extends beyond the limit of defense needs, impacting profoundly on important national issues such as, health industry vis-a-vis medical and insurance infrastructure as well as a great segment of commercial-industrial complex. At present, the glass preform - critical raw material to produce the optical fiber, is 100% imported from Germany and Japan. Objectively, to create a domestic source, a cooperative project participated by Los Alamos and Polymicro began in the summer of 1991 to develop the cladding part of the glass preform. The goal was achieved by developing 0.2NA preform, that was fabricated by Modified Chemical Vapor Deposition (MCVD) of boron and fluorine doped silica glass as cladding layer on a silica substrate tube and collapsing the tube on a solid silica rod as the core. The preform was finally drawn into 200 micron core optical fiber and delivered to Los Alamos National Laboratory.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED
MASTER
GH

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

1.0 INTRODUCTION:

A key objective of this program was to create a competitive domestic source for 0.2 NA step-index multimode glass preforms and fibers now supplied by foreign companies currently manufacturing the fiber optics that is considered important as a critical raw material used in nuclear weapons test program, laser ordnance ignition, industrial process control, laser welding and cutting as well as a strongly emerging field of medical application of Minimally Invasive Surgery and Therapy. Heraeus of Germany and Mitsubishi of Japan are the two world suppliers for this type of preforms. Both companies have their own patented processes to manufacture these preforms. The purpose of this project was to develop a similar type of preform based on the patent docketed by D. K. Nath at Polymicro to

- reduce the cost of production of optical fiber by at least 50%;
- improve the performance of currently used fibers by enhancing the quality and design of preforms;
- market these preforms domestically and internationally; and,
- ultimately make new kinds of preforms for application in other fields.

The resultant preforms can then be specifically designed to produce radiation-hard, high laser power, UV and IR transmitting fiber.

2.0 TECHNICAL PROGRESS

The project progressed through four milestones as follows:

- Facility
- Equipments
- Patent
- Process Development

2.1 FACILITY:

Polymicro had no facility to make glass preform. A warehouse-type facility was converted to build

- preform room,
- glass cleaning and storage room,
- computer interface room,
- scrubber room, and
- hydrogen gas storage facility.

2.2 EQUIPMENTS:

A complete set of Modified Chemical Vapor Deposition (MCVD) preform making unit costs about \$350K. Because of budget restriction, old equipments were bought and refurbished. The only new equipment bought was a scrubber. The following equipment-related tasks were performed.

- Refurbished - lathe, control consoles, chemical delivery system and computer interface
- Bought/Installed - scrubber, torch system, exhaust, gas lines
- Debugged - lathe system, chemical delivery system, scrubber, glass cleaning

2.3 PATENT:

A patent has been docketed by D. K. Nath at Polymicro to fabricate preform of the present project. Because other foreign companies have patents on similar type of glass preforms, a thorough patent search was made. One German and two Japanese companies were identified. The results are shown below.

HERAEUS

- US Patent 4,082,420, April 4, 1978
- RF Plasma Processed
- Highest NA Preform With F Doping
- Used By Most Manufacturers Internationally
- Usable in UV
- Radiation Hard
- Patent Expires April 4, 1995

MITSUBISHI

- US Patent 4,733,939, March 29, 1988
- CVD Processed Preform
- B and F Doping
- OH Content 0.1 to 300 ppm
- 60% More Expensive Than Heraeus
- Radiation Hard
- Not Usable in UV
- Patent Expires March 29, 2005

SUMITOMO

- Jap. Patent J56036606, April 9, 1981
- CVD Processed Preform
- B and F Doping
- OH or OD Content At Least 40 ppm
- Claims on X-rays, Gamma Rays and Radioactive Environment
- Not in UV
- Patent Expires April 9, 1998

RECOMMENDATION BY ATTORNEY

"Limited Patent Protection to a Conventional Optical Fiber Having a High OH Content May Be Available."

2.4 Process Development:

The experimental work started on January 6, 1992. The detailed activities are shown in program schedule (Appendix 1). In essence, the process consists of chemical vapor deposition of fluorine-and-boron-doped silica glass inside a silica tube as cladding layer and then collapsing the tube onto a pure silica rod, which acts as core for guiding light. The resultant glass rod, called preform, is drawn into hair-thin optical fiber to be used as waveguide. The success of the process development effort can be best described by the following achievements.

- A. A worldwide search of core rods was completed and samples from Heraeus, Nippon, Mitsubishi, Thermal American, and General Electric were evaluated by Los Alamos National Laboratories. Heraeus and Nippon core rods were found to be the most suitable materials for radiation hardness. Heraeus core rods was used for the deliverable.
- B. Boron and fluorine doped silica glass deposition process has been fully developed. Data sheet for the fabrication of the preform that was used for the deliverable is given in Table I.
- C. The collapsing technique of the doped silica tube on silica core rods, which act as waveguide, has been developed and optimized via four techniques as given in Appendix 1, Activity No. 15.

D. The fabricated glass preform was drawn into 200 micron core optical fiber. The properties of the fibers are as follows:

<u>Lot No.</u>	<u>(MICRON)</u>				<u>NA</u>	<u>Attn. Loss (dB/km) at</u>	
	<u>Core</u>	<u>Clad</u>	<u>Glass O.D.</u>	<u>Polyimide</u>		<u>308μm</u>	<u>800μm</u>
DBS-01&02	199.2	214.9	265.7	280.7	0.20	90	15.8
DEQ-01	201.4	216.2	268.1	291.1	0.20	98	15.3

The spectral attenuation curves of these fibers in the range 250 - 1120 μ m are shown in Figures 1 - 4.

3.0 BUDGET STATUS

ITEM	TOTAL COST (\$)	FUNDED BY:	
		PTI (\$)	LOS ALAMOS (\$)
Capital	41,142	23,355	17,787
Material	19,357	10,645	8,712
Labor	16,652	10,729	5,923
Overhead	41,355	27,359	13,996
Facility	25,000	15,947	9,053
TOTAL(S)	143,506	88,035	55,471

LANL subcontracted this project to PTI for \$55,471.

TABLE I.

PREFORM I.D. E-1025

OPERATOR

START/FINISH

START DATE

START 08:00FINISH 12:30COMPLETED? Y 2 N

WHY?

RECIPE NO. # BF3/1012

CORE ROD NO.

CORE OD

LITTON 9665-1 FILE # 3 (600)

SPINDLE POT:

DEPOSITION 435 % COLLAPSE %SUBSTRATE O.D. 25.62 WALL 1.53 CSA 115.65

BUBBLER LEVEL

TRANSFILLED?

BEFORE START

SiCl₄ 21.3 cm Y NGeCl₄ I cm Y NGeCl₄ II cm Y NPOCl₃ cm Y N

OPER	TIME	H ₂ O	FLAME			BY PASS O ₂		"IN" O.D.	MD O.D.	EX O.D.
			H ₂ LPM	POT	O ₂ POT	LPM	POT			
	8:48	.14	42.0	21.0	80%	1.21	24.0			

DEPOSITION

OPER	TIME	TYCOM STEP #	CAR. TEMP	O ₂ LPM	He SCCM	-SiCl ₄			POCl ₃			GeCl ₄ I			Pcl ₃ SCCM	FLAME		
						GPM	CARR	%	GPM	CARR	%	GPM	CARR	%		H ₂	O ₂	AUT/TE
	08 ⁵⁶	005	34.8	3.70	681											43	100%	MAN
	09 06	15	35.0	1.11	—	2.50	396	84.4							11.7	43.0	"	"
	09 36	50	35.1	1.11	—	2.51	426	78.5							11.8	46.0	"	"
	10 ²¹	105	35.0	1.11	—	2.49	457	73.6							11.8	48.0	"	"
	11 05	160	34.9	1.39	—	2.49	470	71.1	grade out						6.4	51.0	"	"
	11 12	165	34.9	1.79	—	2.50	471	70.8			"				3.5	57.0	"	"
	11	170	34.8	4.03	—	1.54	308	66.8							out	80.0	"	"

TUBE: 021/21877

AUG

STD

MIN

OUTER DIAMETER :

25.62mm

0.04mm

25.55mm

25.73mm

INNER DIAMETER :

22.57mm

0.05mm

22.46mm

22.69mm

SIDING :

0.13mm

0.00mm

0.13mm

0.14mm

BOW :

0.13mm

0.00mm

0.13mm

0.14mm

0.22mm

S1851C.S01
REGION: 1
dB/km X1E3

DBS - 01

Figure 1

S1852C.S00+ REGION: 1

S3:S3:

dB/km X1E3

DEQ - 01

NM

Figure 2

S1853C.S00+ REGION: 1

S 3:S 3:

dB/km X1E1

DEQ - 01

Figure 3

DBS - 01

518490.SW+
LOG K ^ D U / K
REGION: 1
0.00 10.00 0.20

dB/km X1E1

Figure 4

APPENDIX 1 Glass Project Schedule

Activities	Jan '92				Feb '92				Mar '92				Apr '92				May '92				Jun '92			
	6	13	20	27	3	10	17	24	2	9	16	23	30	6	13	20	27	4	11	18	25	1	8	15
1. Torch Flame Experiments w/H2																								
Tube Heating Rate	▼▼																							
Rod Heating Rate	▼▼																							
SiO2 Deposition Regime		▼▼																						
Collapsing Rate			▼▼																					
2. Test Scrubber																								
Auto PH Control		▼▼																						
Man. Chem. Control Calibration Check		▼▼																						
3. Test Chemical Delivery System																								
SiCl4/Source V,O2, He and BF3				▼▼																				
Mass Flow Controller H2 and O2					▼▼																			
4. Analyze Silica Rod																								
Send Sample to Los Alamos	—		▼																					
5. Deposition Experiments																								
Optimize Freon Etch					▼▼																			
Study Gas Flow 2/3/4/5/ Hrs.																								
SiCl4 1.0 g/min + BF3 45/SCCM							▼▼																	
SiCl4 2.0 g/min + BF3 45/SCCM								▼▼																
SiCL4 2.5 g/min + BF3 45/SCCM									▼▼															
6. Collapse on Rod										▼▼														
7. Measure Deposition Rate											▼													

01/13/92

APPENDIX 1 Glass Project Schedule

Activities	Jan '92				Feb '92				Mar '92				Apr '92				May '92				Jun '92			
	6	13	20	27	3	10	17	24	2	9	16	23	30	6	13	20	27	4	11	18	25	1	8	15
8. Draw 400um Fiber											▼													
9. Measure NA/Attn. Loss												▼												
10. Optimize Flow Rate													▼▼											
11. Measure Deposition Rate														▼▼										
12. Collapse with Quartz Rod															▼▼									
13. Draw Fiber																▼▼								
14. Measure NA/Attn. Loss																	▼							
15. Optimize Collapse																								
Freon purge before collapse											▼	▼												
Vac. collapse											▼	▼												
Without Vac. collapse											▼	▼												
He pressure colapse											▼	▼												
16. Remove Substrate																								
HF etch/Firepolish																					△	→	▼	
Fire Polish																							▼	
17. Fabricate Final Preform																								
Adjust Chem. Flow Rate																	▼							
Write Program Recipe																		▼						
Adjust Temp H2 Rate																			▼					
Write Recipe																				▼				
Fabricate a Preform																					▼			
18. Draw Fiber																						▼		
19. Measure NA/Attn. Loss																						▼		
20. Ship to Los Alamos																							▼	