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EVALUATION OF NRL GLASS FOR FIBER OPTICS, PHASE I.(U)
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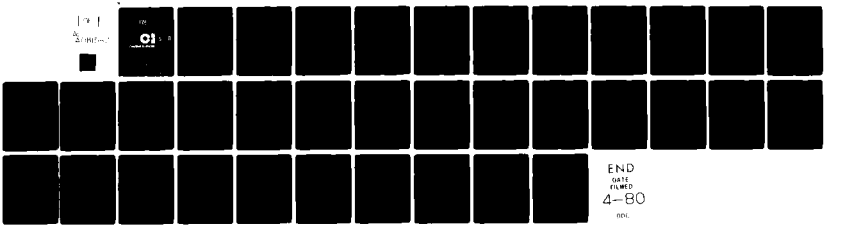

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A REPORT FROM THE TECHNICAL CENTER TOLEDO, OHIO, 43666

- INTERIM REPORT -

EVALUATION OF NRL GLASS FOR FIBER OPTICS - PHASE I

NRL CONTRACT NO. N00173-78-C-0242

ABSTRACT

This interim report covers a Phase I activity to develop glasses for medium loss fiber optic waveguides. The requirements of the final fiber are that it should have: (1) a diameter of 75 to 125 μm with a 50 to 75 μm core; (2) a numerical aperature of 0.4 to 0.6; (3) losses of approximately 100 dB/km; and 4) radiation resistance.

The Phase I activities included the evaluation of a special NRL core glass composition, development of several promising clad glasses and fabricating a test fiber utilizing one of the clad glasses over the NRL core. Meeting the loss requirement was not an objective in Phase I.

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I. INTRODUCTION

The objective of this program is to advance the development of a special NRL core glass composition and to design a clad glass that has properties which match important corresponding properties of the core so that the pair will perform as functional fiber waveguides. The fibers are intended to have a numerical aperture (NA) greater than 0.4 with potential for moderate loss applications and exhibit improved resistance to intense radiation.

Phase I work was started October 1978, and continued through June 15, 1979. The NRL composition was determined to be suitable for large scale melting activity. Several promising clad compositions were identified and one borosilicate was converted into a test fiber as the cladding over the NRL core. Fiber strength limitations indicate the need for better clad glass stability at the draw temperature and an improved property correspondence to the core glass. A lower melting temperature is also desired for the clad glass so that controlled atmosphere melting can be safely performed in special commercial types of melting units. A Phase II program is recommended to complete development and provide suitable fiber for radiation test purposes.

II. RESULTS & CONCLUSIONS

1. The NRL core glass is stable, has sufficient fluidity at melting temperatures for casting and does not have any devitrification tendency in casting or redrawing. Its fluidity at melting temperatures aids in fining. Volatilization of B_2O_3 at the surface leads to inhomogenity, but this can be overcome if melted in a proper unit such as an enclosed melting system with bottom delivery for casting.

2. Cladding glasses in the silicate system were identified having indices that yield a numerical aperture (NA) greater than 0.4, when paired with NRL core glass. Silicate glasses with indices low enough to give NA of 0.6 were not obtained. The glass having the lowest index in this study was KG-33, a commercial borosilicate (n_D = 1.47), which would result in a NA of 0.58 but it is too viscous for consideration.

3. The "best" cladding glasses developed under this study were GS-368, GS-381, and GS-382. GS-382 was the primary choice but was abandoned when large samples (~1-1/4 x 1-1/4 in cross section) phase separated during redraw. The second choice candidate glass (GS-381) was used for the cladding trials although it showed a tendency for devitrification during redraw when higher draw temperatures were used. The third choice glass, (GS-368) was thought to be a little too "soft" for redrawing over the NRL core glass.

4. A cladding glass such as those developed in this study, under proper conditions, can be applied over the NRL core glass to produce a clad fiber.

5. Continuous waveguide fibers were produced by a two-draw process. The first draw reduced the 2" diameter preform approximately 10:1 and completed the seal of the core to the clad. The second draw converted the sealed composite to the final diameter fiber.

6. The first draw of the selected cladding (GS-381) over NRL core glass did not seal well to the core because of poor vacuum during the draw. A second trial with adequate vacuum yielded a good seal between core and cladding.

7. Second redraws produced resin coated fibers in the desired size range of ~0.005" diameter. These fibers were characterized as "brittle" --due to slight devitrification of cladding and/or fairly large difference in expansion (~19 points).

III. RECOMMENDATIONS

1. The NRL core glass should be retained as the core for the proposed clad fiber with minor modification to reduce melting temperature and fining time.

2. The selected cladding glasses, or ones similar to them, should be modified to achieve: 1) lower viscosity for melting in a commercial type of atmosphere furnaces; 2) more stability to prevent devitrification or phase separation during redraw; and 3) increased thermal expansion to obtain an improved expansion match with the core.

3. The NRL core glass and modified cladding glass, using ultra high purity materials, should be melted in a small laboratory melting unit suitable for obtaining a limited amount of the desired glass product for test. Such a melting unit should be an enclosed system to provide the required atmosphere for reduction of cerium and to deliver a cast product having low seed count and good homogeneity.

IV. TECHNICAL DISCUSSION

1. NRL Core Glass Evaluation

Melts of the NRL core glass were made in 90%Pt.-10%Rh. crucibles, in gas-fired furnaces, and under moderate reducing conditions. Reagent grade batch materials having low iron content were used for all melts, including the cladding glasses described later. A typical batch calculation, for the NRL core glass, is shown in Table I. The melts are summarized in Table II. The NRL core glass melts easily and is quite fluid at melting temperatures of 2700°-2850°F. High temperature viscosity values and other property data are shown in Table III. The core glass is also stable--no sign of devitrification was observed in any samples made. This was further verified by gradient boat tests as summarized in Table III. No phase separation was observed, but some surface devitrification was observed at 64 hours. This time span, however, is far beyond redraw time intervals.

Because of the moderately high B_2O_3 content, a B_2O_3 deficient surface layer exists (see B_2O_3 loss in chemical analysis, Table IV) and contributes to inhomogeniety in the cast rods. The glass tends to fine up quite nicely, although no special effort was made to obtain a seed-free glass in the early melts. In melt 6-1, an effort was made to obtain a sample of improved quality. This glass was melted from water quenched frit in a covered crucible.

A one-inch diameter rod from Melt 3 was redrawn. The core glass has sufficient working range for redrawing, and no devitrification was observed in 0.050"; 0.020"; and 0.005" diameter samples nor in the root of the one-inch rod.

2. Cladding Glass Development

The purpose of this portion of the study was to develop cladding glasses which, when redrawn with the NRL core glass, would produce fibers with a large numerical aperature (NA), moderate loss, and be radiation resistant.

2.1 Numerical Aperature

The numerical aperature (NA) of a clad fiber is defined as, NA = $\sqrt{(n_1)^2 - (n_2)^2}$ where n_1 is the core refractive index and n_2 is the cladding refractive index. For this NRL contract, numerical aperatures of 0.4 to 0.6 have been requested. With the NRL core glass, which has an n_D of 1.58, a compatible silicate cladding glass with an n_D of 1.529 is needed for an NA of 0.4. Glasses with refractive indices in this range should be fairly easy to obtain. But for a numerical aperature of 0.6, a glass with a refractive index of 1.462 is needed. This will be difficult in the silicate system since the refractive index of pure fused silica[1] is 1.458, which gives an NA of 0.609. For refractive indices lower than 1.458, other systems such as fluorophosphates or fluorides[2] would be required.

One of the lowest refractive indices in the silicate system (other than fused silica) is 1.47 for KG-33 (Kimax) which gives a NA of 0.58.

2.2 Cladding Glasses

Thirty-one candidate cladding glasses were evaluated and some physical properties are listed in Table V. Ten of these glasses have some degree of opalescence, and hence, properties were not measured.

2.2 Cladding Glasses (cont'd.)

Complete physical property measurements were made on twelve of the glasses. The remaining nine glasses did not have properties measured because they had low NA's, or high melting temperatures. In most instances any missing properties could be interpolated from neighboring glasses.

The thirty-one glasses are divided into a number of systems which can be thought of as a continuum or a solution between two end glasses. For example, one system has the NRL core glass at one end of the continuum with KG-33 borosilicate at the other extreme. Each of these systems are discussed below. The properties of end members are listed in each series for comparison. All of these glasses were 500g in size and were melted in electrically heated furnaces for 16-20 hours. Small discs were cast, annealed and used for obtaining property data.

NRL - Commercial Borosilicate (KG-33)

A band of opalescence was observed in glasses in the NRL-KG-33 system as shown in Table V (a). Those glasses which are close in composition to either the NRL glass or KG-33 were clear glasses. This opalescence or phase separation can be shown to be directly related to the CaO-B_2O_3-SiO_2 phase diagram and it will be discussed later.

Glasses close to KG-33 in composition had NA's of 0.56 and 0.57 while those close to NRL glass had NA's of less than 0.4. The intermediate compositions had some degree of phase separation or opacity.

2.2 Cladding Glass (cont'd.)

NRL - Commercial Aluminosilicate (EZ-1)

Two glasses which were intermediate in composition to the NRL glass and EZ-1 have properties listed in Table V (b). It appears that the high Al_2O_3 and MgO content is stabilizing the glasses. Glasses in the NRL-KG-33 were phase separated in the range of 2 to 10 Wt. % Al_2O_3. NRL-EZ-1 glasses had Al_2O_3 contents of 16 to 18 Wt. %. However, NA's of the NRL-EZ-1 glasses were 0.385 or less.

NRL - Borosilicate Crown

The borosilicate crown (BSC) glass was shown from the literature to have a low refractive index. It also appears to be compatible with the NRL core glass from compositional and physical property standpoints.

There was a band of opalescence between these two glass end members, as shown in Table V (c). However, it appears that glasses close in composition to either end member are stable (i.e., will not phase separate).

The NA of the BSC end member glass is 0.52.

NRL - Commercial Pharmaceutical Container Glass (KG-34)

Two stable glasses in the NRL-KG-34 system were found and physical properties are listed in Table V (d).

Numerical aperature values for the two glasses and KG-34 are 0.41, 0.52 and 0.53.

Commercial Borosilicate (KG-33) - Borosilicate Crown

The KG-33-BSC system had no phase separation and all intermediate glasses were clear stable glasses and some physical properties are listed in Table V (e).

2.2 Cladding Glasses (cont'd.)

Commercial Borosilicate (KG-33) - Borosilicate Crown (cont'd)

The reason for these clear glasses will be explained in the discussion section of the $CaO-B_2O_3-SiO_2$ system.

Numerical aperatures for these glasses ranged from 0.52 to 0.56 with a NA of 0.58 for KG-33.

$CaO-B_2O_3-SiO_2$ System

Reference 3 provides a phase diagram (Fig. 646) for the $CaO-B_2O_3-SiO_2$ ternary system. A simplified version of this system is shown in Fig. 1. There is a large two liquid region on the $B_2O_3-SiO_2$ side which extends from about 1% CaO to about 40% CaO. Any ternary glasses which are located in this two liquid region could be phase-separated. However, if the glass becomes a quaternary system,or more, the region of phase separation is not as well known. For example, the NRL core composition without Al_2O_3 and normalized to the $CaO-B_2O_3SiO_2$ ternary system is located in the two liquid region. This composition, however, is quite stable with the addition of Al_2O_3. Al_2O_3 makes the phase diagram three dimensional so that in going from certain points to others in three dimensions (e.g., from NRL to KG-33) in opal region is intercepted.

In Fig. 1 compositions normalized to the $CaO-B_2O_3-SiO_2$ system are denoted as circled dots. The main glasses (NRL, BSC, and KG-33) are shown normalized as circled dots also. Thus, it can be seen that the KG-33-BSC system is outside the two liquid region entirely and all compositions are glasses. The other two major joining lines from NRL to KG-33 or NRL to BSC pass through the two liquid or opal region.

2.2 Cladding Glasses (cont'd.)

CeO_2

Cerium oxide (CeO_2) in the amount of 1.6 wt. % was added to all the cladding glasses to prevent radiation darkening.[4] When the glasses were melted under oxidizing conditions, the glasses were dark amber in color due to blue absorption of Ce^{4+} ions. When these glasses were melted in gas-fired furnaces, under moderate reducing conditions, the amber intensity varied depending upon the composition (Table VI).

Based on the preliminary cladding melts, four glass compositions (GS-347, GS-357, GS-363 and GS-368) were selected as being promising and were melted in larger quantities. In addition, a new composition, GS-380, was also melted. GS-380 is essentially KG-33 (commercial borosilicate) plus 1.6 wt. % CeO_2 and was melted for comparison to GS-357, a modification of KG-33. These glasses were melted in 90%Pt.-10%Rh. crucibles in a gas-fired furnace and under moderate reducing conditions (1.5% combustibles).

The melt sizes were 5 to 6 kilograms. Melting temperatures and times were 2950°F and 24 to 26 hours, respectively. The results are summarized in Table VI. Based on these results, GS-347 was ruled out because of the light opalescence in the cast billets as was GS-357 and GS-380 because of their high melting viscosities. GS-380 was also rejected because of the results obtained from a preliminary redraw trial (see Section IV.3).

Of the two remaining glasses, GS-368 (essentially borosilicate crown plus 1.6 Wt. % CeO_2) was considered a better cladding candidate than GS-363 because of the apparent slightly greater fluidity at melting and casting temperature.

In order to have the option of several candidate cladding glasses, two additional compositions were melted. These were GS-381 which is

2. Cladding Glass Development (cont'd.)

essentially KG-34 (commercial pharmaceutical container glass) plus 1.6 wt. % CeO_2, which has a higher softening point than GS-368. The other was GS-382, a modified GS-347 which hopefully would eliminate the light opalescence observed in the GS-347. The results of these melts, which were made under similar conditions described earlier, are also shown in Table VI.

Based on all of the melting results, we concluded that the three "best" cladding glass candidates were GS-382, GS-381 and GS-368 in that order. GS-382 was the lightest in color and was the "hardest" glass in terms of softening point. A higher softening point for the cladding glass is desirable during the redraw process.

Some initial redraw work was done on the three cladding glasses to check for phase separation. Initially, 3/8-inch unpolished rods, core drilled from billets were redrawn to 5-10 mil. Redraw temperature was 1700°F. No devitrification or phase separation was seen in the starting glass, the fibers, or the root of GS-382, GS-381 or GS-368. A second series of redraw trials was again made on the three glasses, this time using 1-1/4" x 1-1/4" x 6" polished square bars cut from the larger billets. With the larger cross section requiring longer residence time in the furnace, phase separation was seen in the GS-382 root section. No devitrification or phase separation was observed in GS-381 or GS-368. Because of the phase separation encountered with GS-382, gradient tests were made on GS-381, GS-382, and GS-368. GS-382 showed the phase separation encountered during redraw. GS-381 had no phase separation and no devitrification after 2 hours. However, heavy surface

2. Cladding Glass Development (cont'd.)

devitrification was seen after 64 hours. GS-368 showed no devitrification or phase separation after 2 hours. After 64 hours, there appeared to be some very thin patches of surface devitrification. These results are tabulated in Table VII. As a result of the redraw and gradient boat studies, GS-381 became the first choice cladding glass and GS-368 the second choice.

3. Evaluation of KG-33 for Cladding

In a preliminary experiment, a clad assembly consisting of KG-33 commercial tubing over NRL core glass was redrawn. The assembly consisted of a six-inch length of 3/8 O.D. x 7/32 I.D., commercial KG-33 tubing and a six-inch length of NRL 6-1 polished rod. Five mil fiber was redrawn at approximately 1900^{o}F. No cracks were observed in the fibers due to the large difference in thermal expansion. Cracking was observed, however, in the larger cross sectional areas (0.025" O.D.) of the assembly root after cool down. Because the redraw temperature was near the liquidus temperature (1980^{o}F) of KG-33, devitrification occurred on the outer surface of the cladding. For this reason, KG-33 (GS-380) was rejected.

4. Melts for Preforms

Eight-kilogram melts of GS-381 and five-kilogram melts of NRL core glass were made in preparation for preform fabrication. High purity (low iron - 5 to 7 ppm) batch materials were used for each composition. These materials were the same as, or equivalent to, those shown in Table I. The melting conditions were the same as those described earlier for the NRL glass and large cladding melts. Rods 1-1/2"

4. Melts for Preforms (cont'd.)

diameter x 18" long were cast from the NRL glass and 2-1/2" x 3" x 18" billets were cast from GS-381 glass. Three melts of each composition were made. The physical properties of these melts are shown in Table VIII.

5. Preform Fabrication

The core and cladding were finished to size using standard optical finishing techniques. The core glass (1-1/2" diameter x 18" long billet) was turned and then polished to a final size of 1.273" O.D. x 13-1/2" long. The cladding was fabricated from a 2-1/2" x 3" x 18" long billet. The billet was core drilled, ground, fine-ground, and polished to a final size of 2.000" O.D. x 1.283" I.D. x 15" long. Figure 2 is a sketch of the finished assembly showing a 1/4" hole at the upper end for a 1/4" pin which provided the support for the core. As shown, the core was shorter than the cladding by about 1" at the upper end to allow for vacuum connection and 1/2" at the lower end to provide a good draw down over the core at the start of the redraw.

6. Fabrication of Clad Fibers (GS-381 over NRL Core)

6.1 Primary Draw

The core rod and cladding were first washed in hot water using a mild detergent. The washing was followed by a hot water rinse, three rinses in distilled water and finally two rinses in propanol. The tube and rod were then placed on end to dry. After drying, the tube and rod were assembled. Vinyl gloves were worn for all handling of the assembly during and after washing. A 1/4" pin was inserted through the support hole, as shown in Figure 2, and the ends were sealed with silicone rubber. A vacuum connection consisting of a

6.1 Primary Draw (cont'd.)

stopper and copper tubing was placed in the upper end of the cladding tube and sealed with silicone rubber.

The hanger for the assembly consisted of modified hose clamps having silicone rubber on the inner faces for good grip to the outer surface of the cladding and having extensions on the outer faces for attachment to the redraw tower head. All redrawing was done on a standard redraw tower consisting of a down feed mechanism, a pulling mechanism with a set of rubber rolls and a small electric furnace.

The assembly was hung with approximately 3" of the lower portion of the assembly extending into the furnace. The remaining portion of the assembly above the furnace was surrounded by a 3" I.D. ring of insulation brick to form a chimney. The redraw arrangement is shown in Figure 3.

Heat-up to drawing temperature of 1650^{o}F took place over 6-1/2 hours. At 1600^{o}F., drawing of the assembly began. As soon as the assembly was pulled down and in the draw rolls, the vacuum pump was turned on. The first portion of the draw appeared to be devitrified but cleared up as the draw proceeded. The interface seal between cladding and core was poor. Power to the furnace elements was increased to maximum limits. Although furnace temperature did not apparently increase (as indicated by the recording controller) the interface seal did improve. Shortly, thereafter, bubbles were noticed at the interface indicating insufficient vacuum. The silicone seals and other seal areas were beginning to fail due to the high temperatures encountered. At this point, the run was aborted and the assembly was slow-cooled to room temperature.

6.1 Primary Draw (cont'd.)

Nineteen pieces of redraw stock, each approximately three feet in length, were obtained ranging in size from .050" diameter to .220" diameter. Target diameter for the primary draw was ~.200" diameter. Five pieces were obtained in the target diameter range of 0.160" diameter to 0.220" diameter. All pieces showed some spalling of the cladding from the core. However, sufficient lengths of material in the larger diameters were obtained to permit secondary redraw trials. Microscopic examination showed a poor interface as evidenced by an almost continuous layer of small bubbles at the interface. No devitrification was seen on the rods of interest; i.e., three of the large diameters which were obtained just prior to aborting the run.

The remaining portion of the undrawn assembly (~6" length) was drawn on a second attempt using a different drawing support head. In this case, the exterior of the upper end of the cladding fitted into the inner surface of a heavy walled stainless steel tube. A support pin passed through the support hole in the core and cladding and into the walls of the stainless steel tube. The exterior of the tube was threaded, enabling a large nut to be passed over the assembly and screwed onto the stainless tube. O-ring seals were placed at the upper and lower shoulders of the nut to provide for the vacuum seal. The vacuum source was attached at the upper end of the stainless steel tube. Radiation shields were placed around the core and cladding assembly immediately below the large stainless steel nut. The assembly was preheated over a 6-1/2 hour period to 1675^{o}F. where drawing commenced. A good vacuum was obtained (~30") and held during

6.1 Primary Draw (cont'd.)

the entire drawing period without any apparent leaks. The core-clad interface seal appeared to be very good as a result of the good vacuum obtained. Approximately forty feet of clad rod ranging in size from 0.100" to 0.170" diameter was obtained. The desired diameter of 0.200" diameter could not be obtained because only about a 4" length of usable assembly was available for drawing. No spalling of the cladding from the core was observed. However, a very thin layer of devitrification was observed on the outside of all of the redrawn rods.

6.2 Second Draw

Redraws of primary rods were made on a redraw tower similar to that used for the primary draws. A more elaborate arrangement was required however because of the requirements to provide a continuous fiber on a spool and to jacket the drawn fiber with a plastic coating. The redraw arrangement is depicted in Figure 4. The clad rod from the primary redraw was suspended from the redraw tower head by notching the rod and attaching it to the fixture by stainless steel wire. The furnace was of the same type (3" I.D.) used for the primary redraw using a simple cover to prevent heat loss. Below the furnace a laser micrometer was used for continuous readout of fiber diameter. The drawn fiber then passed through a glass laboratory funnel with a rubber nipple attached for applying the plastic coating (Dow Corning Sylgard^{R} 184). The coated fiber then passed through a 24" long tube furnace which accelerated curing of the resin. The fiber was initially pulled without the coating by the drawn rolls until stabilized in size (0.004" to 0.005" diameter). The fiber was

6.2 Second Draw (cont'd.)

then transferred to the collection spool and the resin then introduced into the funnel to begin coating the fiber.

A total of three primary rods from the initial redraws of the clad assembly were redrawn. A summary of these runs is shown in Table IX. An attempt to redraw a rod from the second group of primary redraws were made but no fiber was obtained. This fiber was very brittle and broke frequently. The brittleness is attributed to surface devitrification observed on the primary rods which may have increased during redraw.

V. REFERENCES

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2. W. D. Kingery, Introduction to Ceramics, John Wiley and Sons, (1960), 781 pp.

3. E. M. Levin, G. R. Robbins and H. F. McMurdie, Phase Diagrams for Ceramists, Am. Cer. Soc.

4. E. J. Friebele, G. H. Sigel, Jr., and M. E. Gingerich, "Nuclear Radiation Effects on Fibers", Laser Focus, pg. 51-56, Sept. 1978.

TABLE I

NRL CORE GLASS BATCH CALCULATION

OWENS ILLINOIS - BATCH CALCULATION 09/26/78

COMPOSITION- NRL-5 (10-26-78) PROJECT- 2041 REQUESTED BY- C.E. SCHOTT

RAW MATERIAL	MATERIAL CODE	GMS MATL	COST/GM -¢	COST-$
MAGNA PLUS QUARTZ 28	7 14 16 505	2299.3511	0.003	6.945
CALCIUM OXIDE 648-145	3 20 6 505	1315.2380	0.036	47.119
INSTEL ALUMINA 1368	5 13 4 505	669.6647	0.0	0.0
BORIC ACID 5M15E5	5 5 10 505	1162.8301	0.001	0.692
CERIUM OXIDE 40A	16 58 2 505	81.2285	0.030	2.418

TOTAL BATCH WT- 5528.3086 GMS TOTAL GLASS WEIGHT- 5000.0000 GMS FUSION LOSS- 528.3123 GMS

TOTAL BATCH COST- 57.17 COST/GM OF GLASS- 0.01 REL. OXIDE RATIO = 1.0000

OXIDE NO.	OXIDE NAME	TARGET MOLE PCT.	TARGET WT. PCT.	WEIGHT OXIDE	WT. PCT. OXIDE	CHEMICAL ANALYSIS
2103	LI2O	0.0	0.0	0.0023	0.0000	
2111	NA2O	0.0	0.0	0.0058	0.0001	
2119	K2O	0.0	0.0	0.0023	0.0000	
2220	CAO	29.7000	25.9107	1295.5093	25.9102	
2305	B2O3	12.1000	13.1051	655.2544	13.1051	
2313	AL2O3	8.5000	13.4826	674.1292	13.4826	
2326	FE2O3	0.0	0.0	~~0.0175~~ .02245	0.00045	
2414	SIO2	49.1000	45.8951	2294.7520	45.8950	
2458	CEO2	0.6000	1.6066	80.3268	1.6065	
		100.0000	100.0000			

SIGNED- William H. Grant DATE- 9-26-78

READ, UNDERSTOOD AND WITNESSED BY- Daisy Hall DATE- 9/26/78

TABLE II

SUMMARY OF CORE GLASS MELT DATA

Melt No.	Melt Size	Temp. (°F)	Time (Hours)	Furnace ATM	Fe_2O_3 (ppm)	Samples Made	Comments
1	400g	2700	20-3/4	1.5%*	7	2 2-inch dia. discs	Annealing pt. determination
2	5000g	2700	7-3/4	1%	7	2 1-inch Ø x 18" rods 2 1½-inch Ø x 18" rods	Scale up melt. Very cordy. Large blisters. Casting temp. too low
3	5000g	2850	7	1%	7	1 1-inch Ø x 18" rod 1 1½-inch Ø x 18" rod 1 2½-inch Ø x 10" rod	Better quality. Samples to NRL
4	5000g	-------Not Melted - Opt for better quality raw materials-------					
5	5000g	2850	7	1.1%	4.5	2 1-inch Ø x 18" rods 2 1½-inch Ø x 18" rods	About same quality as Melt 3. Samples to NRL
6	5000g	2850	6	1%	4.5	Water Frit	Base for Melt 6-1.
6-1	3500g Frit From Melt 6	2800	77-1/4	1.3%		Cooled in situ for core drilling	Seed-free. Better homogeniety than Melts 3 and 5. Samples to NRL.

*Combustible

TABLE III

NRL CORE GLASS PROPERTIES

Annealing Point (^{o}C)	677
Strain Point (^{o}C)	637
Softening Point (^{o}C)	820
η_D	1.58
α ($\times 10^{-7}/^{o}C$) (0-300^{o}C)	60.9
α_c ($\times 10^{-7}/^{o}C$) (A.P. - Rm.T.)	83.4
ρ (g/cm^3)	2.6927
Log Viscosity (^{o}C)	
2.0	1246
2.5	1163
3.0	1099
4.0	1002
5.0	932
6.0	882
7.0	841
7.60	821

Gradient Boat Studies:

Melt No.	Temp. Range (^{o}F)	Time (Hours)	Comments
NRL-5 & NRL-9	1140-1840	16	No phase separation or surface devitrification
NRL-9	"	16	3/16" dia. rod redrawn from 1-1/2" dia. blank--no phase separation or surface devitrification.
"	"	64	3/16" dia. rod redrawn from 1-1/2" dia. blank. Surface devitrification starting at about 1500^{o}F. No apparent phase separation.
NRL-11	"	16	Sample before redraw--no phase separation or devitrification.
"	"	16	Redrawn sample (1-1/2" dia. to 0.120" dia.). No phase separation or devitrification.

TABLE V

CANDIDATE CLADDING GLASS PROPERTIES

(a)

	NRL	GS-349	GS-350	GS-351	GS-369	GS-370	GS-371	GS-372
SP (^{o}C)	820	V.L.	Opal	N.M.*		V.L.		
AP (^{o}C)	677	Opal				Opal	Opal	Opal
STP (^{o}C)	637							
n_D	1.58				1.553			
α ($10^{-7}/^{o}$C)	60.9				57.3			
C ($10^{-7}/^{o}$C)	83.4							
ρ (g/cm^3)	2.6927							
Seal Stress in NRL (psi)								
N.A.	-				.29			

(a) Cont'd.

	GS-373	GS-374	GS-375	GS-376	KG-33
SP (^{o}C)	M.	800			822
AP (^{o}C)	Opal	564		N.M.*	565
STP (^{o}C)		513			513
n_D		1.477	1.475		1.47
α ($10^{-7}/^{o}$C)		34.6	34.7		32
C ($10^{-7}/^{o}$C)		44.4			38
ρ (g/cm^3)		2.2619			2.23
Seal Stress in NRL (psi)					
N.A.		0.56	0.57		0.58

*N.M. = Not Measured
V.L = Very Light
M. = Medium

TABLE V (CONT'D.)

CANDIDATE CLADDING GLASS PROPERTIES

(b)

NRL to EZ-1 Series

	NRL	GS-345	GS-346	EZ-1
SP (^{o}C)	820			911
AP (^{o}C)	677	688	696	707
STP (^{o}C)	636	649	654	658
n_D	1.58	1.550	1.541	1.5327
α ($10^{-7}/^{o}$C)	60.9	50.5	41.5	43.3
C ($10^{-7}/^{o}$C)	83.4	67.6	54.7	55
ρ (g/cm^3)	2.6927	2.5898	2.5350	2.52
Seal Stress in NRL (psi)				
N.A.	-	0.307	0.35	0.385

(c)

NRL to BSC Series

	NRL	GS-364	GS-365	GS-366	GS-367	GS-368	B.S.C.
SP (^{o}C)	820	Opal	Opal	M	L	757	
AP (^{o}C)	677			Opal	Opal	551	
STP (^{o}C)	636					505	
n_D	1.58					1.493	
α ($10^{-7}/^{o}$C)	60.9					54.1	
C ($10^{-7}/^{o}$C)	83.4					73.2	
ρ (g/cm^3)	2.6927					2.3463	
Seal Stress in NRL (psi)							
N.A.	-					0.52	

M = Medium
L = Light

TABLE V (CONT'D.)

CANDIDATE CLADDING GLASS PROPERTIES

(d)

	NRL to KG-34 Series			
	NRL	GS-347	GS-348	KG-34
SP (^{o}C)	820	856		798
AP (^{o}C)	677	652	605	576
STP (^{o}C)	636	620	563	531
n_D	1.58	1.52	1.492	1.49
α ($10^{-7}/^{o}$C)	60.9	52.6	41.7	43
C ($10^{-7}/^{o}$C)	83.4	70.5	58.2	54
ρ (g/cm^3)	2.6927	2.5324	2.3698	2.34
Seal Stress in NRL (psi)				
N.A.		0.41	0.52	0.53

(e)

	KG-33 to BSC Series								
	KG-33	GS-357	GS-358	GS-359	GS-360	GS-361	GS-362	GS-363	GS-368
SP (^{o}C)	822	788	783	783	755	753		754	757
AP (^{o}C)	565	577	551	548	540	541		540	551
STP (^{o}C)	513	507	503	499	494	495		494	505
n_D	1.47	1.477	1.479	1.483	1.485	1.487	1.491	1.487	1.493
α ($10^{-7}/^{o}$C)	32	36.1	39.7	39.7	47	47.7	49.9	43.5	54.1
C ($10^{-7}/^{o}$C)	38	46.3	52.9	52.2	61.7	58.7		60.4	73.2
ρ (g/cm^3)	2.23	2.2638	2.2764	2.2954	2.3042	2.3107		2.3184	2.3463
Seal Stress in NRL (psi)			5700T	5600T					
N.A.	0.58	0.56	0.56	0.55	0.54	0.53	0.525	0.53	0.52

TABLE IV

CHEMICAL ANALYSIS

NRL CORE GLASS - MELT 5

	Target (Wt.%)	Analysis (Wt.%)
SiO_2	45.89	47.02
B_2O_3	13.11	11.00
CeO_2	1.61	1.62
Al_2O_3	13.48	13.51
CaO	25.91	26.37
	100.00	99.52

TABLE VI

RESULTS OF INITIAL LARGE CLADDING GLASS MELTS

Glass	Results
GS-347	Very, very light opalescence in 2-1/2" diameter billet, no NaCl refining agent.
GS-347	NaCl added as refining agent. Light opalescence in rectangular 2-1/2" x 3" x 12" billet.
GS-357	Dark amber color. Very viscous melt. Results the same with NaCl added as refining agent in both 2-1/2" diameter and 2-1/2" x 3" x 12" billet.
GS-363	Light amber. Clear glass (no opalescence), few seeds present in 2-1/2" x 3" x 12" billet.
GS-368	Light amber. Clear glass (no opalescence), few seeds present in 2-1/2" x 3" x 12" billet.
GS-380*	Dark amber color. Very viscous melt--more so than GS-357. 2-1/2" x 3" x 12" billet.
GS-381	Light amber. Few seeds in 2-1/2" x 3" x 18" billet.
GS-382	Very light glass. No opalescence. Few seeds in 2-1/2" x 3" x 18" billet.

TABLE VII

CLADDING GLASS GRADIENT STUDY RESULTS

Composition	Temp. Range (°F)	Time (Hours)	Comments
GS-368	1360-2000	2	No devitrification, no phase separation
"	1140-1840	64	Some very thin surface devitrification in range of 1500-1650°F; not continuous; due to saw cut surface??
GS-381	1360-2000	2	No phase separation but surface roughness and possible slight devitrification in areas of saw cut surfaces.
"	1140-1840	64	No phase separation but surface devitrification starting at ~1100°F over entire length. Very heavy layer starting at ~1500°F.
GS-382	1360-2000	2	Three samples run. No devitrification except on saw cut surfaces. Phase separation in all samples at ~1500°F.

TABLE VIII

PHYSICAL PROPERTIES FOR FINAL MELTS OF GS-381 AND NRL

	GS-381	NRL
STP (°C)	537	629
AP (°C)	584	670
SP (°C)	805	815
n_D	1.490	1.578
α (10^{-7}/°C)	42.1	60.9
N.A.	0.53	

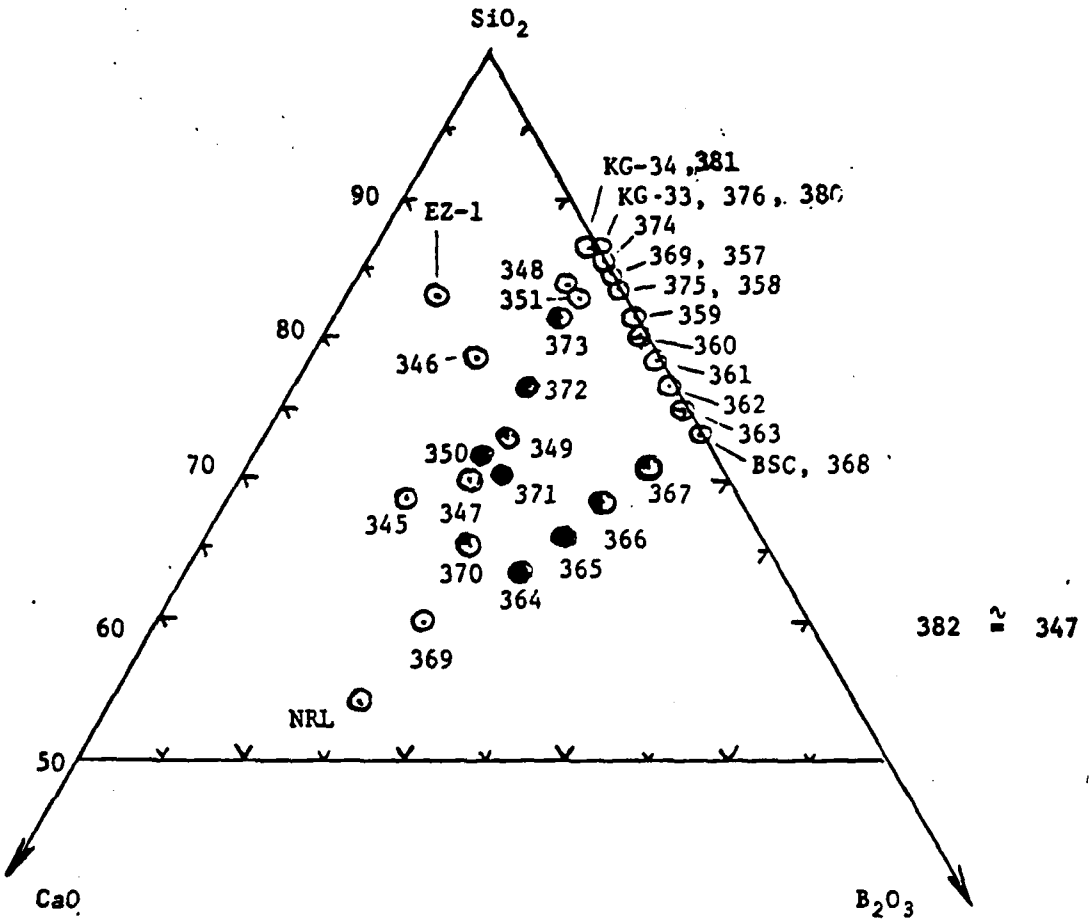
TABLE IX

SUMMARY OF SECONDARY REDRAW TRIALS

Run No.	Draw Temp (°F)	Curing Temp (°F)	~Coating Thickness (mil)	Amount Fiber Obtained (ft.)	Comments
1	1750	300	3/4-3	1100	Brittle fiber
2	1900	300	1/2-2	1900	Higher draw temperature in attempt to improve interface bond. Brittle fiber. Fiber not centered in coating. Fiber to NRL.
3	1900	550	1-10	400	Slower draw speed and higher curing temperature to allow for curing of heavy coating. Fiber not centered in coating. Brittle fiber. Fiber to NRL.

Figure 1

NRL Core Glass and Cladding
Glass Normalized to Fit
the $CaO-B_2O_3-SiO_2$ Ternary System
(wt. %)



LEGEND

- Glass
- Very Light Opalescence
- Medium Opalescence
- Opal

FIGURE 2

CLADDING AND CORE ASSEMBLY

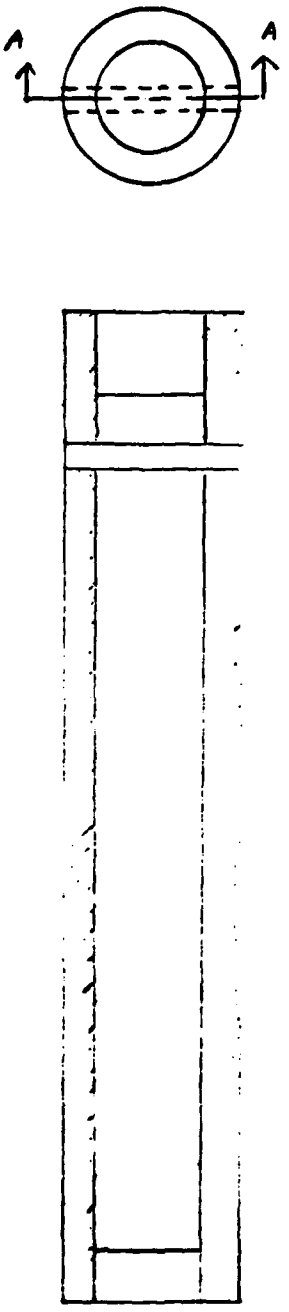


FIGURE 3

PRIMARY REDRAW ARRANGEMENT

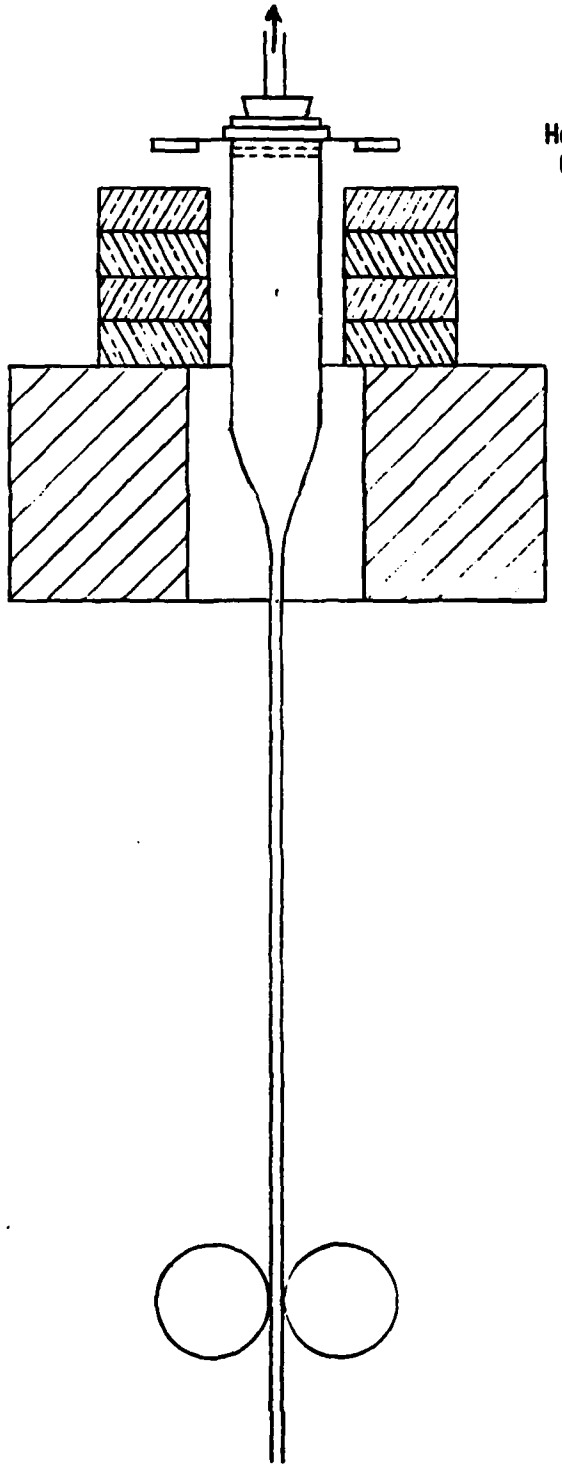


FIGURE 4

SECONDARY REDRAW ARRANGEMENT