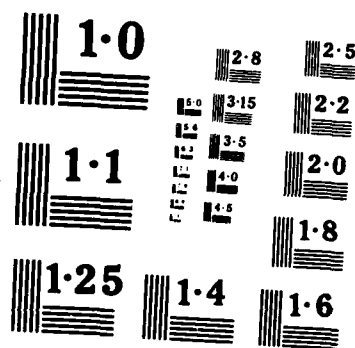


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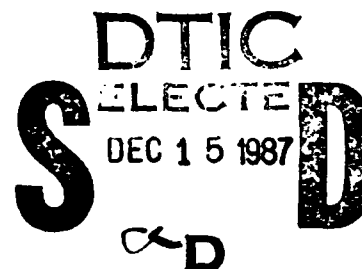
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Final Technical Report
August 1987



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SBIR - LONG FLUORIDE FIBER

SpecTran Corporation



Raymond E. Jaeger and Lubos J. B. Vacha

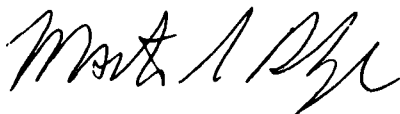
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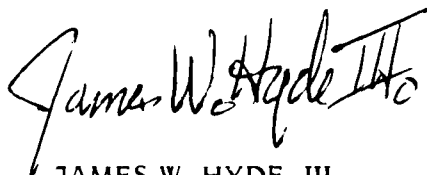
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19 ABSTRACT (Continue on reverse if necessary and identify by block number) This report summarizes results obtained under Phase I SBIR program aimed at developing new techniques for fabricating long lengths of heavy metal fluoride glass (HMFG) optical fiber. A new method for overladding conventional HMFG preforms with a low melting oxide glass was developed, and improvements in the rotational casting method were made to increase preform length. The resulting composite glass canes consist of a fluoride glass core which carries the optical energy, a fluoride glass optical cladding, and an oxide glass overcoat layer to enhance strength and chemical durability. To show feasibility, prototype optical fiber preforms up to 1.6 cm in diameter with lengths of 22 cm were fabricated. These were drawn into optical fibers with lengths up to 900 meters. f				
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1.0 Introduction

The development of heavy metal fluoride glass fibers is now approaching the stage of limited practical use. Their potential performance, such as minimum attenuation in the range of 10^{-2} to 10^{-3} db/km and excellent tolerance to exposure to nuclear radiation makes them an interesting longterm alternative to silica fibers. In addition to long distance repeaterless transmission lines, military applications of interest include IR image and light transmitting flexible bundles. While these applications require the ability to draw long, strong and durable lengths of fluoride fiber, to date only relatively short lengths (~ 100-200m) have been achieved.

The objective of this program (SBIR Phase I) was to demonstrate the feasibility of drawing fiber lengths in excess of 500m from a single preform. To our knowledge, this report describes the first example of a continuous fluoride fiber $165\mu\text{m}$ in diameter with a length in excess of 800 meters.

The program has been conducted by Spectran Corp. with funding through Rome Air Development Center at Hanscom AFB.



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2.0 Experimental

2.1 Equipment

The glass melting and forming equipment used in this program has been described earlier in the Final Report for Contract #F 30602-84-C-0198 entitled Preparation of Heavy Metal Fluoride Glass in Bulk Form. The facility shown in Fig. 1 basically consists of an atmosphere controlled chamber in which batching, melting, casting and annealing processes are conducted. At the present time, the batch size is limited to ~ 1.5 kg and the maximum furnace temperature is about 950°C.

A new fiber drawing tower has been equipped with a Beta Instruments, Inc. laser diameter gauge including automatic feedback to assure good diameter control. The take-up has been provided with tension control enabling the spooling of the fiber with very low tension (1-5g). The fiber draw furnace has a gas inlet for N₂ or He, depending upon whether the drawn fiber is coated with FEP Teflon or drawn bare.

As the first step toward increasing the drawable fiber length, a new preform mold 12.5mm in diameter x 220mm

long was fabricated. The higher surface to volume ratio of this mold was chosen due to the low heat conductivity of fluoride glasses and earlier success in preparing shorter preforms of the same diameter.

A second approach, based on an idea that was not originally proposed, was to overclad the preform with an oxide glass. This allowed for an increase in mold diameter to 16mm and a corresponding increase in preform volume by almost a factor of 2.

To accommodate the longer brass mold (Fig. 2), a larger mold preheating chamber was fabricated. The chamber design consisted of an alumina tube for insulation purposes lined with a resistance heated copper tube.

An earlier mold cover containing a hole for pouring the cladding glass was replaced with a specially designed plug mounted to a rotating chuck (Fig. 3) that functions as a cover, preventing glass leakage during mold rotation. This arrangement makes it easier to pour the glass melt into the mold without any restrictions and also helps to keep the mold centered during the spinning operation. At rotation speeds of 5000 rpm, the 2-piece molds showed a tendency to leak at the joint, resulting in a distinct seam along the

preform. This seam caused surface hydrolysis and crystallization at the interface between the glass cladding and Teflon coating during the fiber draw from coated preforms.

In order to avoid difficulties caused by seams, a single piece mold was designed. We tested different mold materials with lower linear expansion coefficients, such as Pyrex, silica and stainless steel. Although silica and Pyrex released the glass nicely and the preforms did not possess a longitudinal seam, they often cracked due to the low heat capacity of the thin mold material. Inserting these glass molds into a massive brass heat reservoir did little to improve the situation, primarily as a result of problems associated with the design of this composite mold.

A stainless steel mold with $\alpha = 10 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ released the first cast preform nicely, but a second casting had to be forced out in pieces, damaging the highly polished inside mold surface. It was, therefore, decided to provide this mold with a 1mm taper (difference between the top and bottom inner diameter). This approach has resulted in the greatest success to date.

During the casting of these preforms, we observed a prominent crystal pattern inside the core along the direction of glass flow. Condensed material, deposited inside the melting crucible above the melt line, was suspected to be the origin of the crystal growth. As a result, we designed a platinum bottom drain crucible and plunger to avoid pouring these condensed particles along with the glass melt (Fig. 4). This only partially eliminated the observed crystal pattern. Upon further investigation, it was determined that the main reason for the crystallization of the core was the remelting of the clad tube and the mixing of it into the core as the molten core was poured into the solid cladding tube. This technique caused the cladding glass to pass through the dangerous crystallization temperature range (T_x - T_g) 3 times during preform fabrication as shown in Fig. 4.

2.2 Glass Preparation

2.2.1 Compositions

Our experiments began with the typical ZBLAN core and ZHBLANI clad given in Table I that have been previously reported in the literature. (1)

The use of PbF_2 as a core dopant, as originally proposed, was eventually dropped due to its apparent tendency to promote crystallization. As the program developed, modifications to the ZHBLANI composition produced a superior core-clad combination for which we are presently applying for patent protection.

TABLE I

FLUORIDE FIBER COMPOSITIONS

(mole %)							
	ZrF_4	HfF_4	BaF_2	LaF_3	AlF_3	NaF	InF_3
ZBLAN (core)	53		20	4	3	20	
ZHBLANI (clad)	39.55	13.24	17.92	3.98	2.99	21.90	0.42

2.2.2 Oxide Overclad Glass

A new idea, not previously reported, has been utilized to improve fiber drawing properties and fiber strength. The optical properties of fluoride glass with chemical properties of oxide glass have been combined to prepare a waveguide fiber of superior quality.

Glass compositions from the alkali phosphate group with a very low softening point were identified in the literature [2,3,4,5]. Oxide melts were made by calcination of dihydrogen phosphate, sodium carbonate and zinc oxide in a Pyrex beaker at ~ 300°C. The calcined batch was transferred into a Pt crucible, melted at ~ 950°C for 1 hour and cast into a mold held at ~ 200°C. A cullet of this glass was weighed into a Pt crucible, remelted at 800°C, cast into a preform mold and spun at 5000 rpm. This tube was subsequently filled with coreglass or clad and core using conventional rotational casting technique.

Initially the most suitable overclad

composition was sodium-zinc phosphate glass having a glass transition temperature (T_g) of 265°C and thermal expansion coefficient (α) of $18.5 \times 10^{-6}^{\circ}\text{C}^{-1}$. This appeared to be a good candidate when compared to the ZHBLAN clad glass which has a $T_g = 264^{\circ}\text{C}$ and $\alpha = 18.6^{-6}$.

Unfortunately, these composite preforms had to be drawn at higher draw temperature ($\sim 400^{\circ}\text{C}$) than the usual 340°C resulting in core glass crystallization. A search was, therefore, conducted to identify an oxide glass composition which would not only match T_g and α , but also have similar viscosity at fiber drawing temperatures.

A series of compositional modifications based on mixed alkalis were evaluated in order to fine tune viscosity characteristics. Thermal expansion data and T_g 's for a selected group of compositions are shown in Figs. 5-11. The viscosity characteristics of the optimum oxide/fluoride pair were compared and are shown in Fig. 12. The activation energy was measured to be 700 KJ/mol for the oxide glass and 900 KJ/mol for the fluoride glass.

Differential scanning calorimeter tests (DSC) on both glasses separately and in composite form, shown in Figs. 13 and 14, exhibited no tendency for increased crystallization upon reheating. This absence of crystallization would be expected during fiber drawing as well.

Preforms prepared using the oxide overclad (P-1) and the fluoride core and clad glasses given in Table I did not crack and fiber drawn from these preforms did not crystallize. The results of over 50 castings are summarized in Table II. A typical fluoride/oxide fiber refractive index profile is given in Fig. 15. As shown in the figure, the oxide overclad index is higher than fluoride core and, as such, is presently unsuitable for an optical clad. This profile was measured on a preform cross section as shown in Fig. 16.

2.2.3 Chemical Durability of the Oxide Glass

At this time, the chemical durability of the oxide overclad glass is fairly low. As shown in Table III, it is actually worse than the

fluoride clad glass and thought to be due to its high alkali content. It is known, however, that this can be improved dramatically through the addition of oxides, such as Al_2O_3 . Future experiments to modify the basic composition are expected to improve the glass durability by approximately 2 orders of magnitude.

2.3 Fiber Draw

Fiber has been drawn using the draw tower described in an earlier government report (#N00014-85-C-2494) on a contract sponsored by Naval Research Laboratory. The drawing parameters were dependent on the type of preform to be drawn and the desired coating.

For the uncoated preform, we used dry He as protective atmosphere. For the FEP Teflon coated preform/fiber, we used dry N_2 , since He had a tendency to overheat the Teflon and degrade the fiber strength. The oxide overcladded fluoride preform (FLOX) did not require any protective atmosphere at all. This represents a significant improvement since uncoated fluoride preforms show severe surface degradation upon heating in air. When drawn in an air atmosphere, needle like crystal deformations on the surface of the preforms

have been observed to propagate through the neckdown region and into the fiber, resulting in decreased fiber strength. The FLOX fiber design eliminates this problem.

To improve fiber strength, fluoride preforms were etched in a 0.4N ZrOCl_2 or 0.4N H_3BO_3 solution in 1N HCl for a few minutes. The same idea was applied in the case of FLOX preforms using 1N HF. Bare fibers drawn from both fluoride and FLOX preforms were coated in-line with UV curable epoxy resin. Typical coating thickness was $75\mu\text{m}$ on $165\mu\text{m}$ OD fiber. The total length of $165\mu\text{m}$ fiber drawn from a 12mm OD preform and 220mm length was 800m. 16mm OD FLOX preform yielded ~ 900m of fiber with $150\mu\text{m}$ diameter. The result of tensile testing of these fiber designs is given in Fig. 17 and indicates the improvement in strength exhibited by the FLOX fiber. A photograph of the 220mm fluoride preform is shown in Fig. 18.

Conclusion

We have proved that, indeed, it is possible to draw a long continuous length of fluoride fiber with good strength from a single preform. This goal was met by either increasing the length of a normal diameter (12.5mm) preform or by

overcladding the fluoride preform with oxide glass. A 150 μ m fiber, ~ 900m in length was drawn from a 16mm diameter FLOX preform and showed higher strength than its fluoride counterpart.

We are confident that, in the future, even longer FLOX preforms can be fabricated. The composition of the oxide overclad glass will be modified to lower the expansion and increase chemical resistance, thus, yielding a stronger more durable fiber exhibiting increased reliability and longer life.

We wish to thank our consultants on this program, Prof. Cornelius T. Moynihan and Dr. Steve Crichton of RPI for their contribution of ideas, advice and measurements during this program.

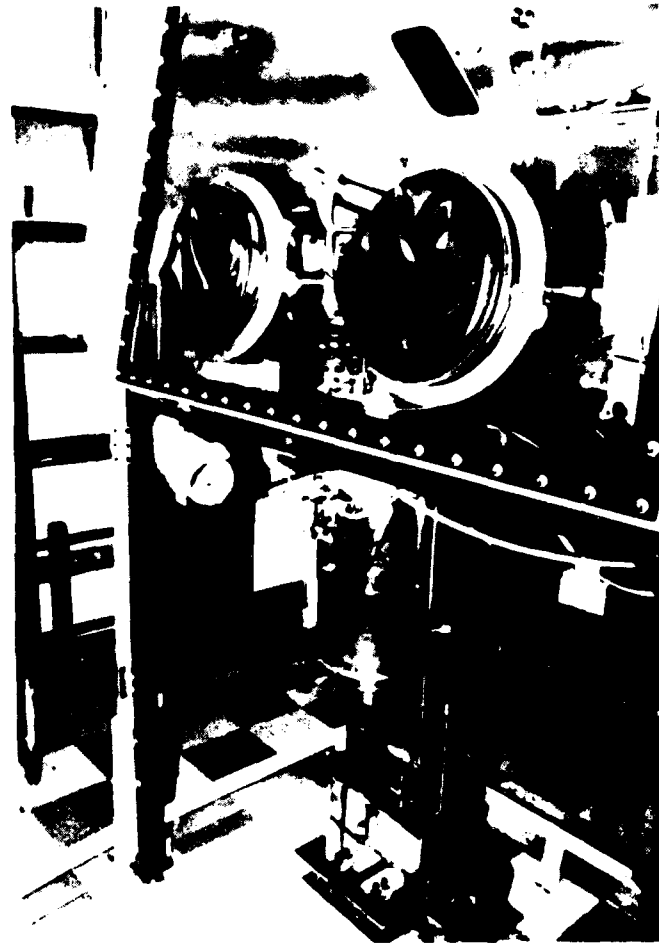
We also wish to thank Dr. Martin Drexhage and Dr. Al Drehman of Hanscom AFB for their guidance, support and measurements.

Finally, we wish to thank Mr. Chris Matson of SpecTran's technical staff for his daily contribution in carrying out the details of the experimental program.

REFERENCES

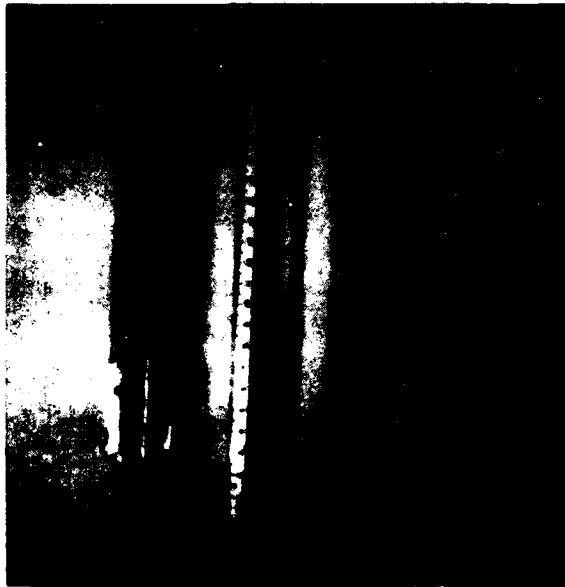
- (1) U.S. Patent #4,445,755, "Infrared Transmitting Optical Waveguide", Takahashi, Shibata, Ohsawa; May 1, 1984.
- (2) Wilder, J. A., "Property Variation in Alkali-Alkaline-Earth Metaphosphate Glasses". Journal of American Ceramic Society, Vol. 67, No. 6 (1984), pages 438-444.
- (3) Bunker, B.C., Arnold, G.W., Wilder, J.A., "Phosphate Glass Dissolution in Aqueous Solutions", Journal of Non-Crystalline Solids 64, (1984), pages 291-316.
- (4) Ray, N.H., Plaisted, R.J., Robinson, W.D., "Oxide Glasses of Very Low Softening Point, Part 4", Glass Technology, Vol. 17, No. 2, April 1976.
- (5) Wilder, J.A., "Development of High Thermal Expansion Glasses for Sealing to Aluminum Alloys", Report from Sandia Laboratories SAND 78-1964.

FIGURE 1.



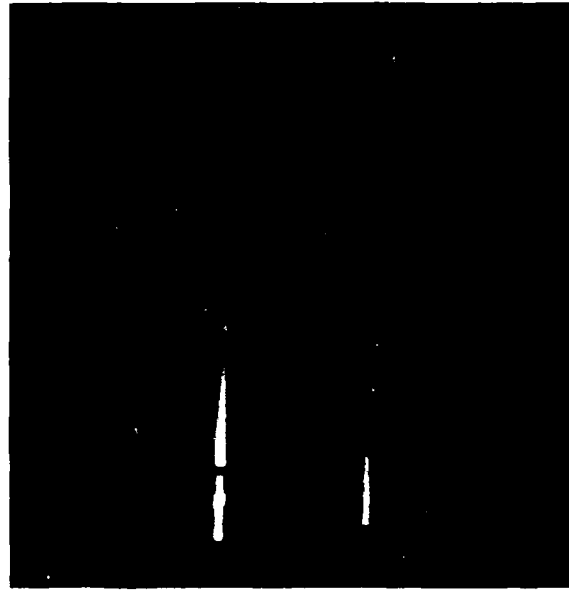
MELTING AND FORMING EQUIPMENT

FIGURE 2.



LONG PREFORM MOLD WITH 12MM BORE

FIGURE 3.



PHOTOGRAPH (above) OF THE REDESIGNED MOLD WITH COVER.

SCHEMATICS OF THE SAME (below)

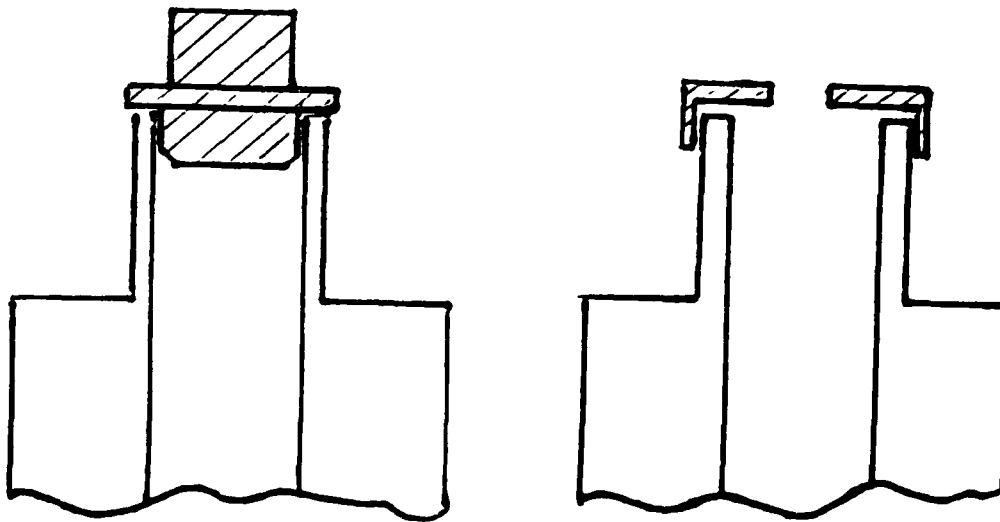
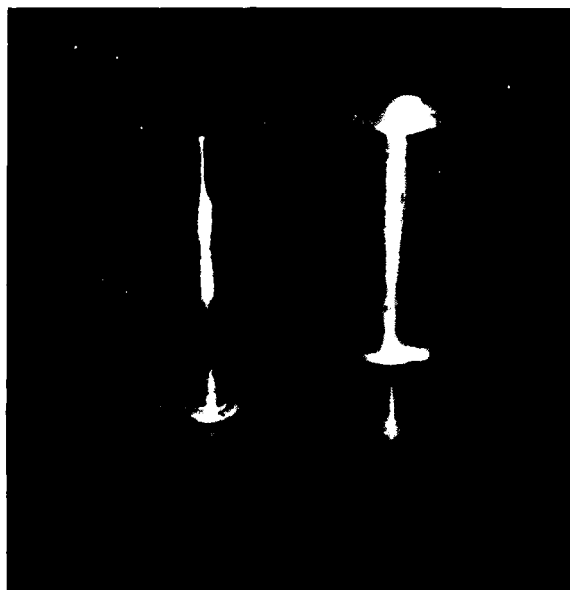
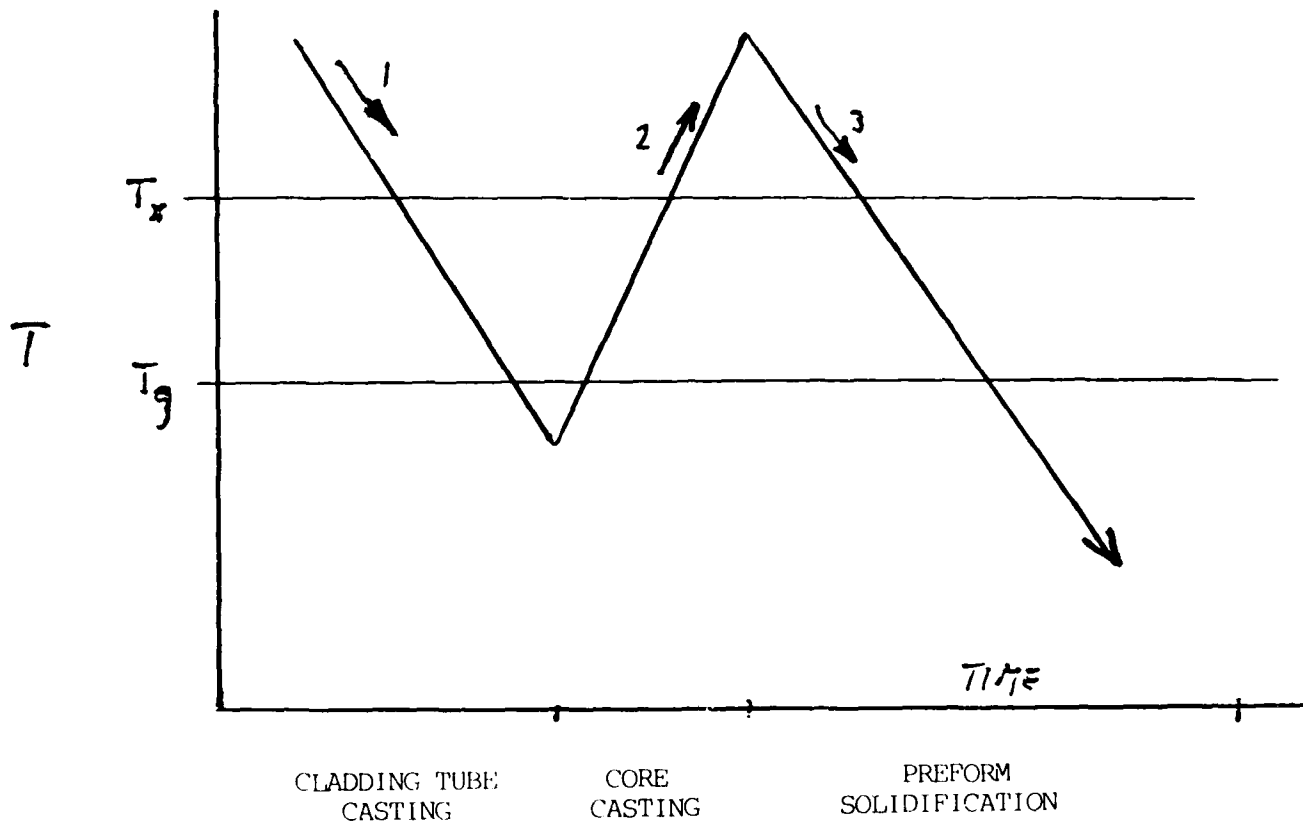


FIGURE 4a.



Pt BOTTOM DRAIN CRUCIBLE WITH PLUNGER

FIGURE 4 b.



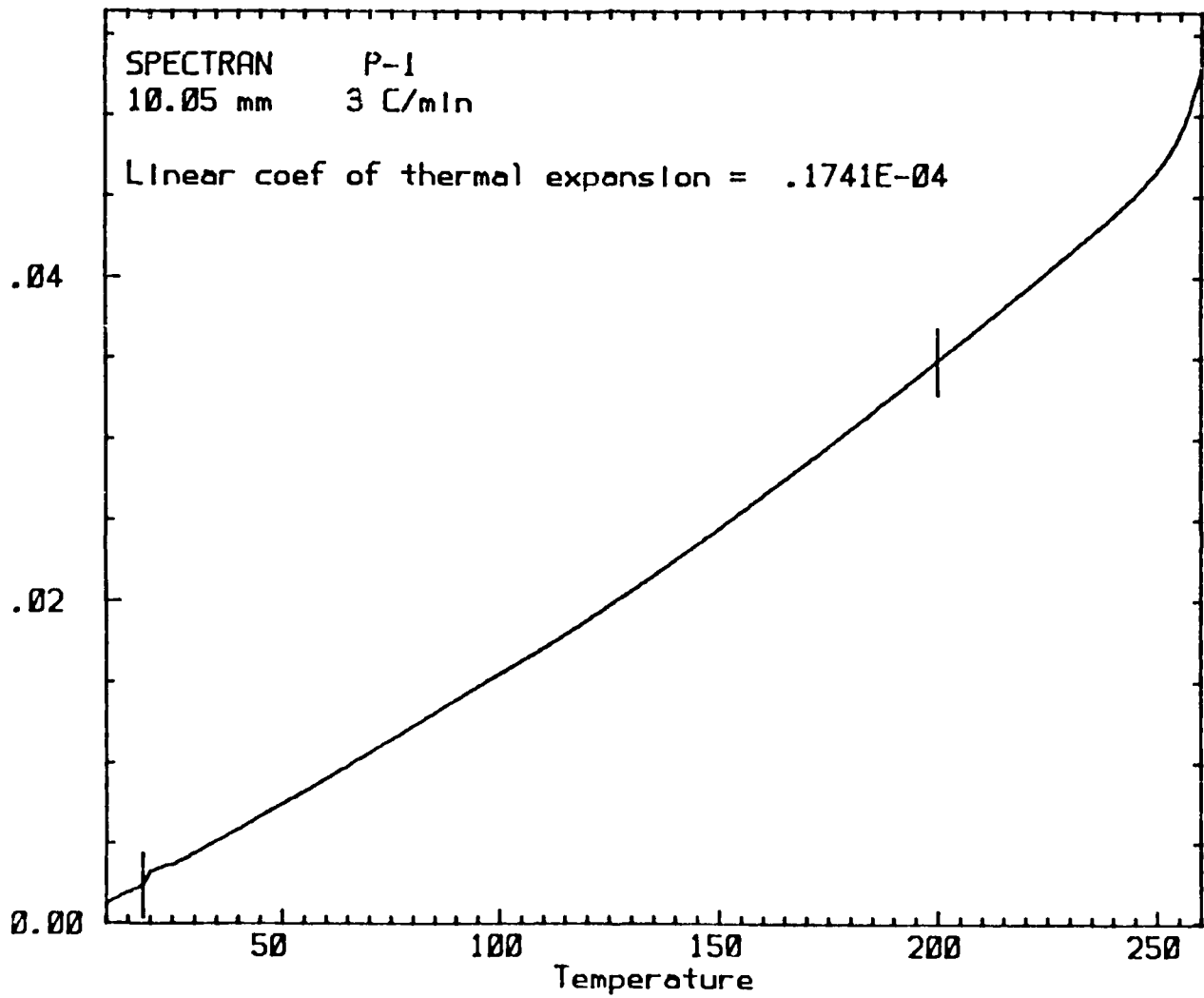
TEMPERATURE VERSUS TIME DURING THE CASTING OF THE PREFORM

DISSOLUTION RATES IN WATER AT 20°C

ZBLAN	0.88 mg/cm ² /h
Oxide Overclad	1.11 mg/cm ² /h
Window Glass	$7.2 \cdot 10^{-6}$ mg/cm ² /h

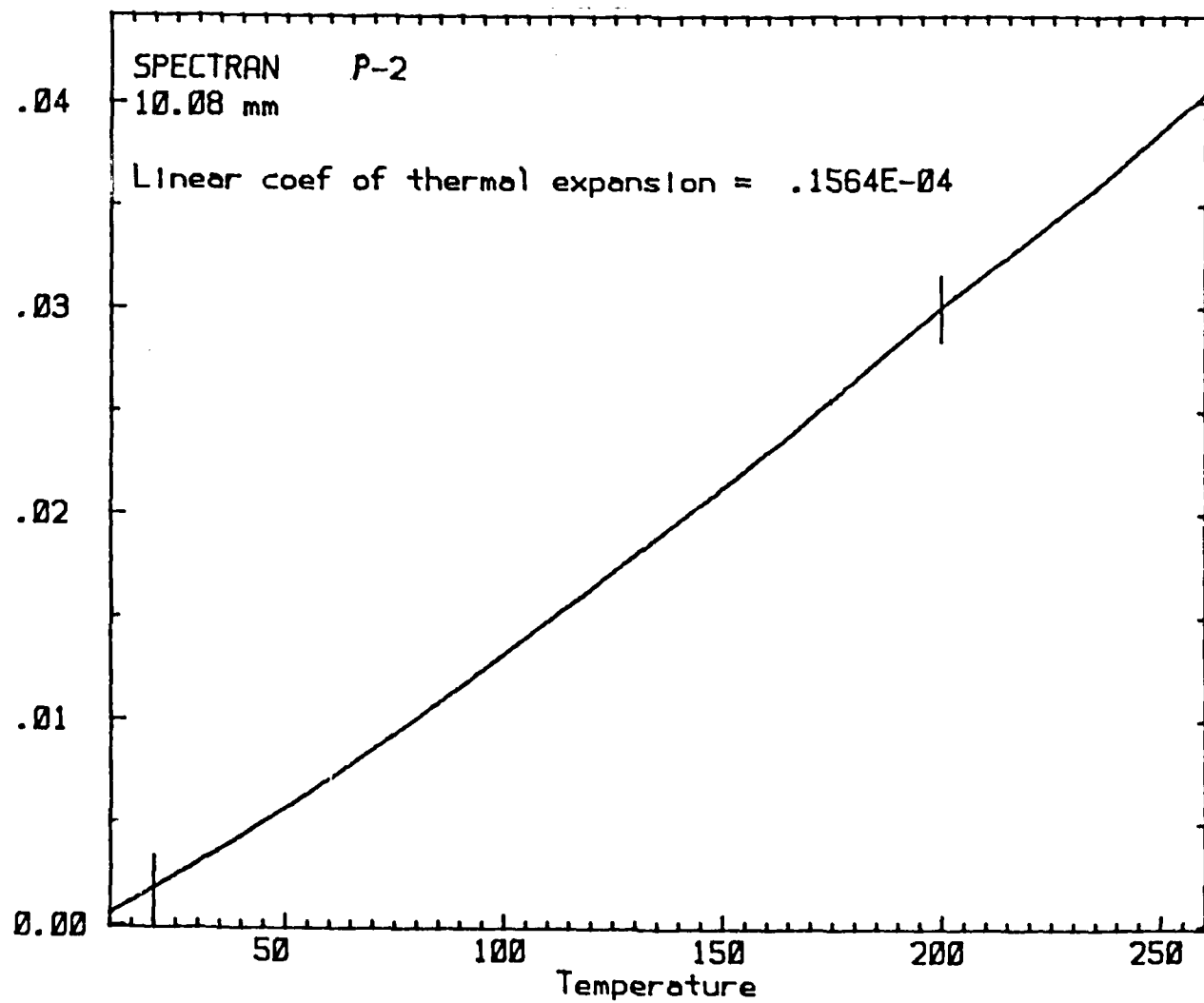
TABLE III

FIGURE 5.



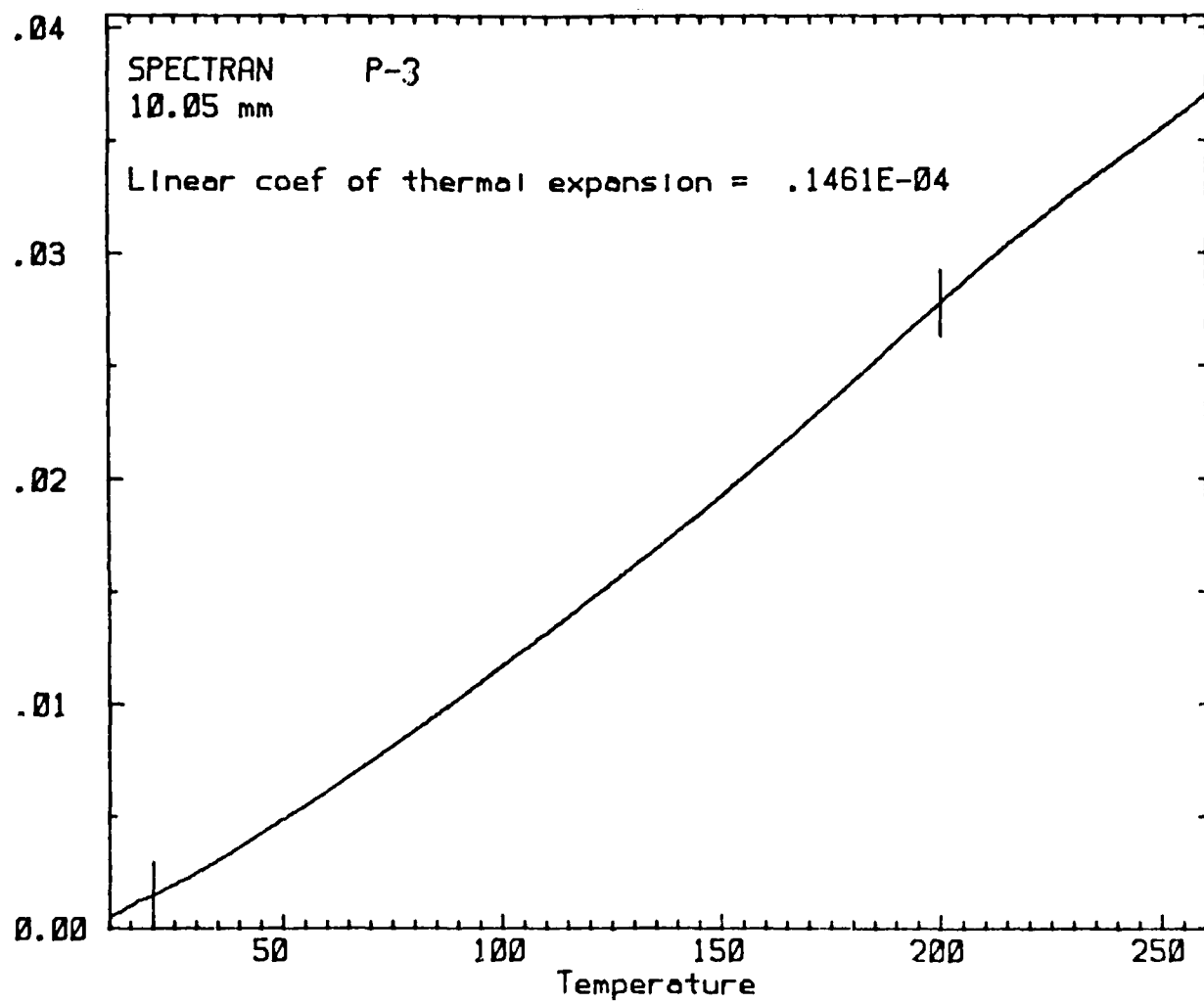
LINEAR EXPANSION OF OXIDE GLASS

FIGURE 6.



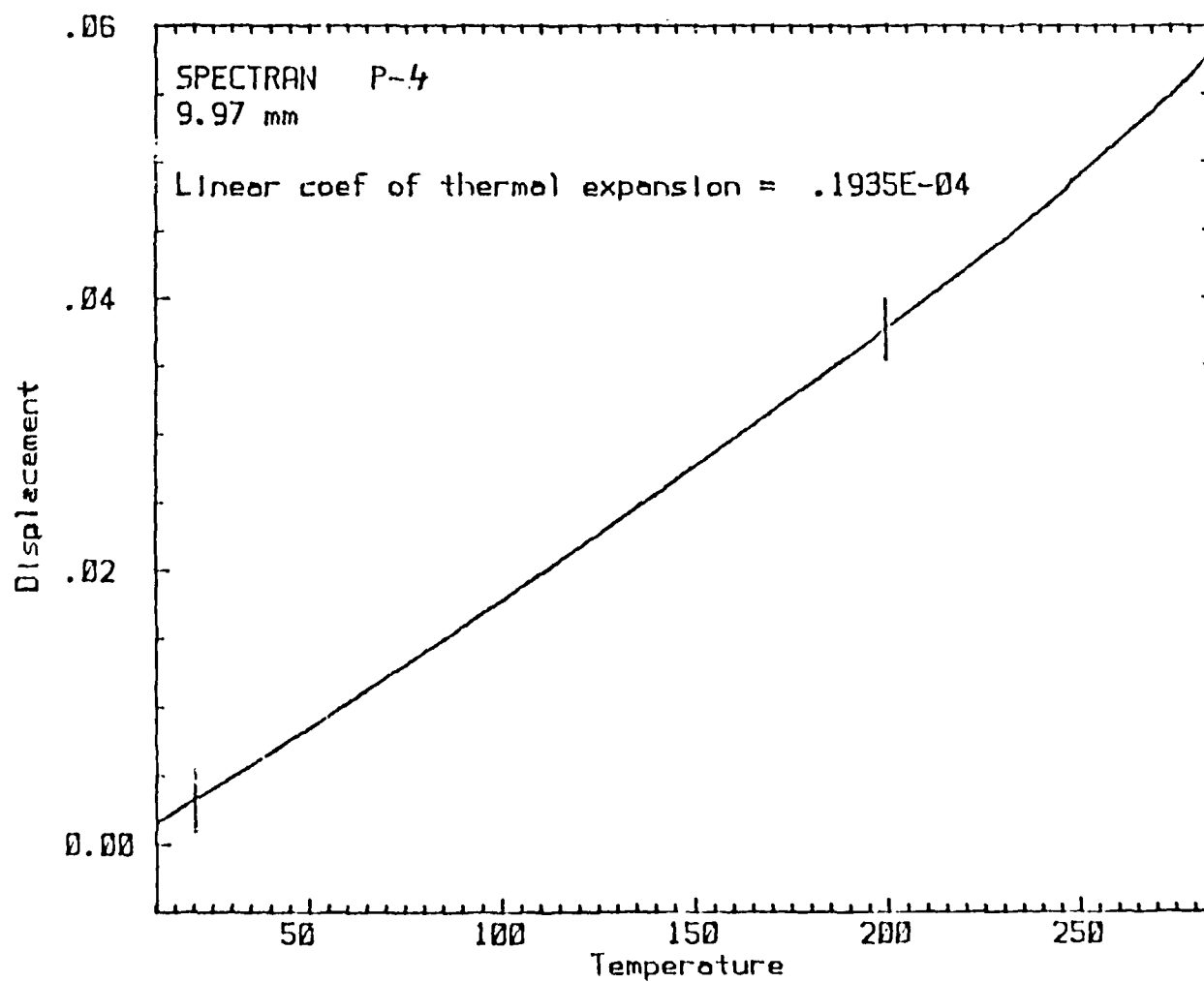
LINEAR EXPANSION OF OXIDE GLASS

FIGURE 7.



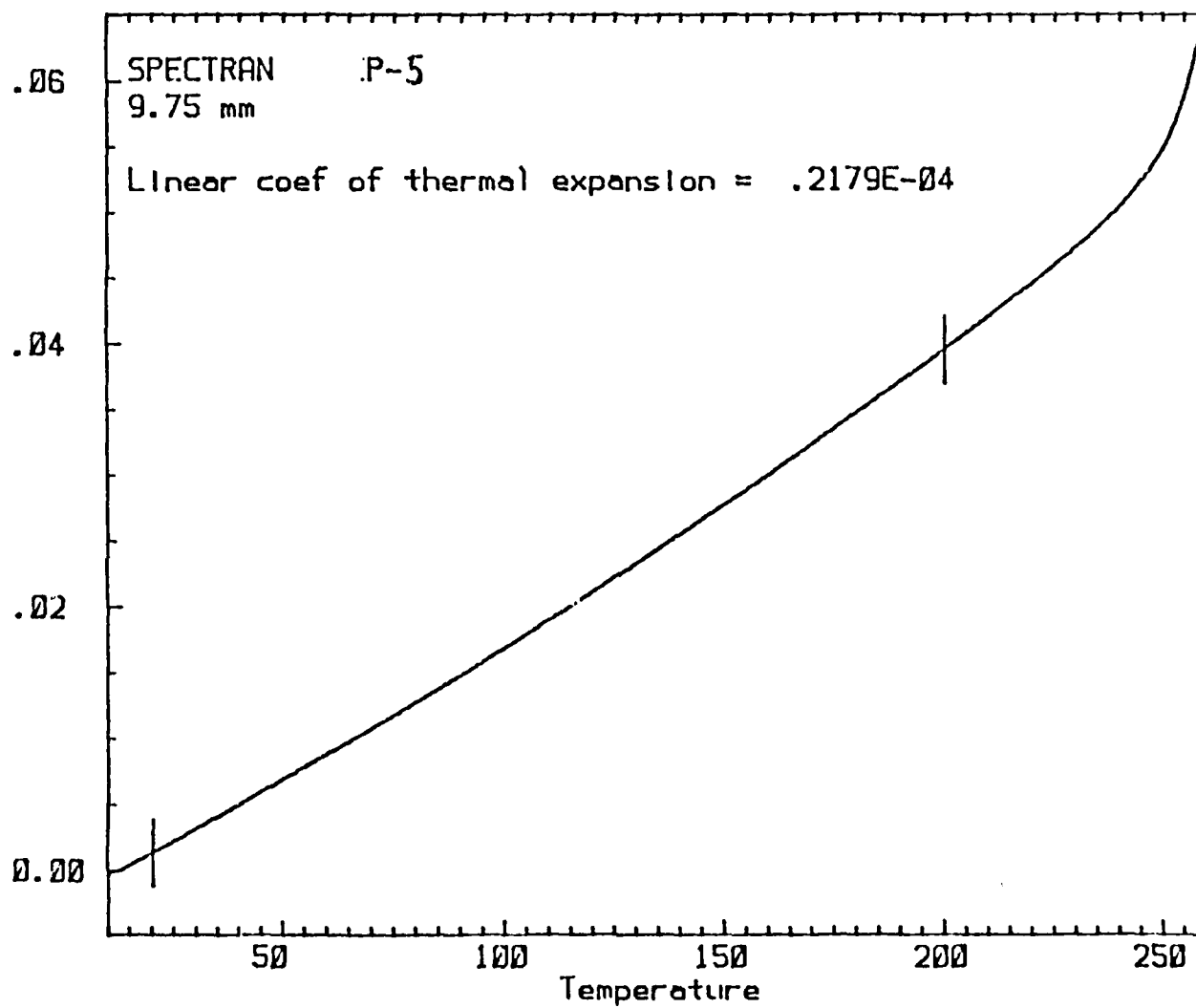
LINEAR EXPANSION OF OXIDE GLASS

FIGURE 8.



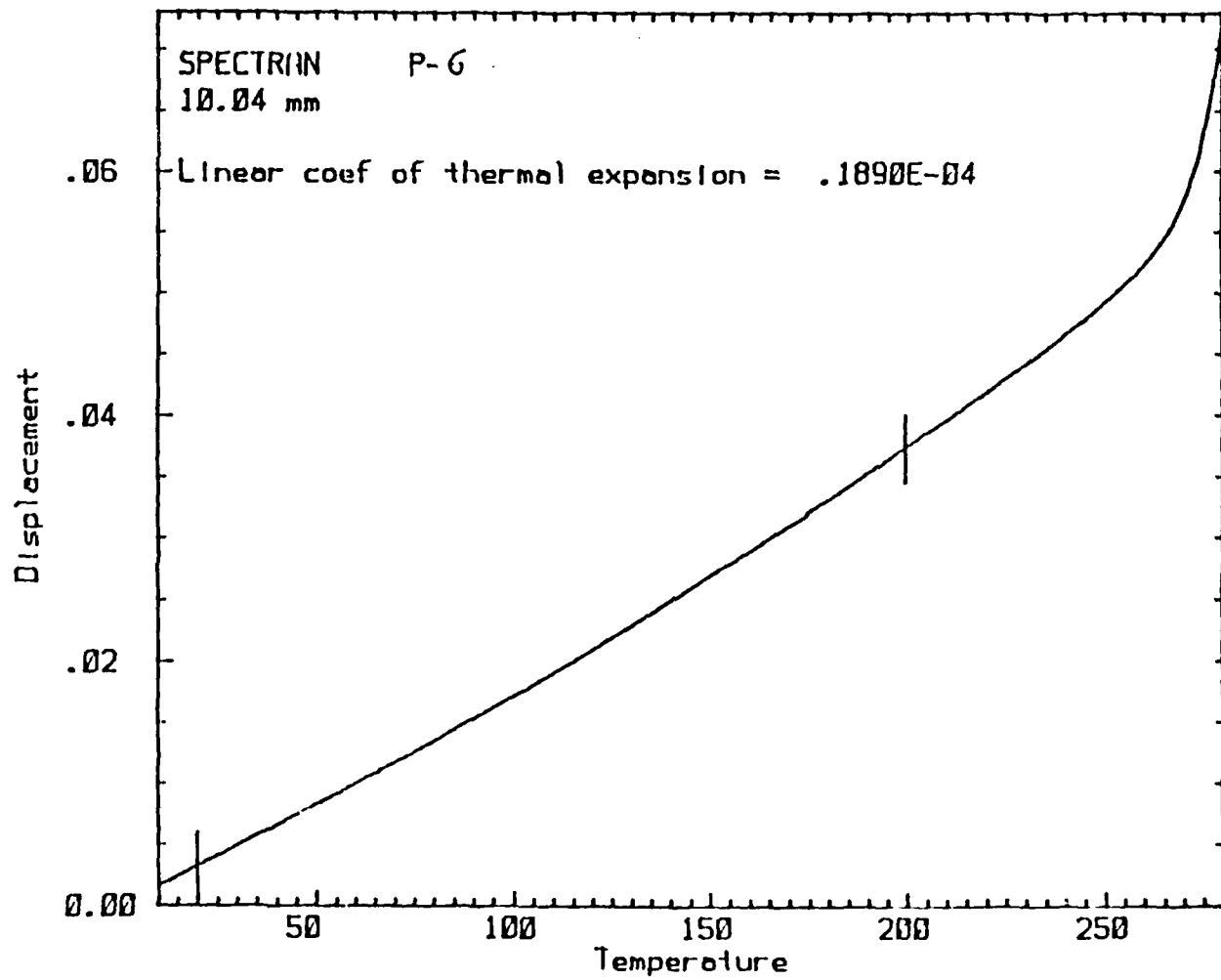
LINEAR EXPANSION OF OXIDE GLASS

FIGURE 9.



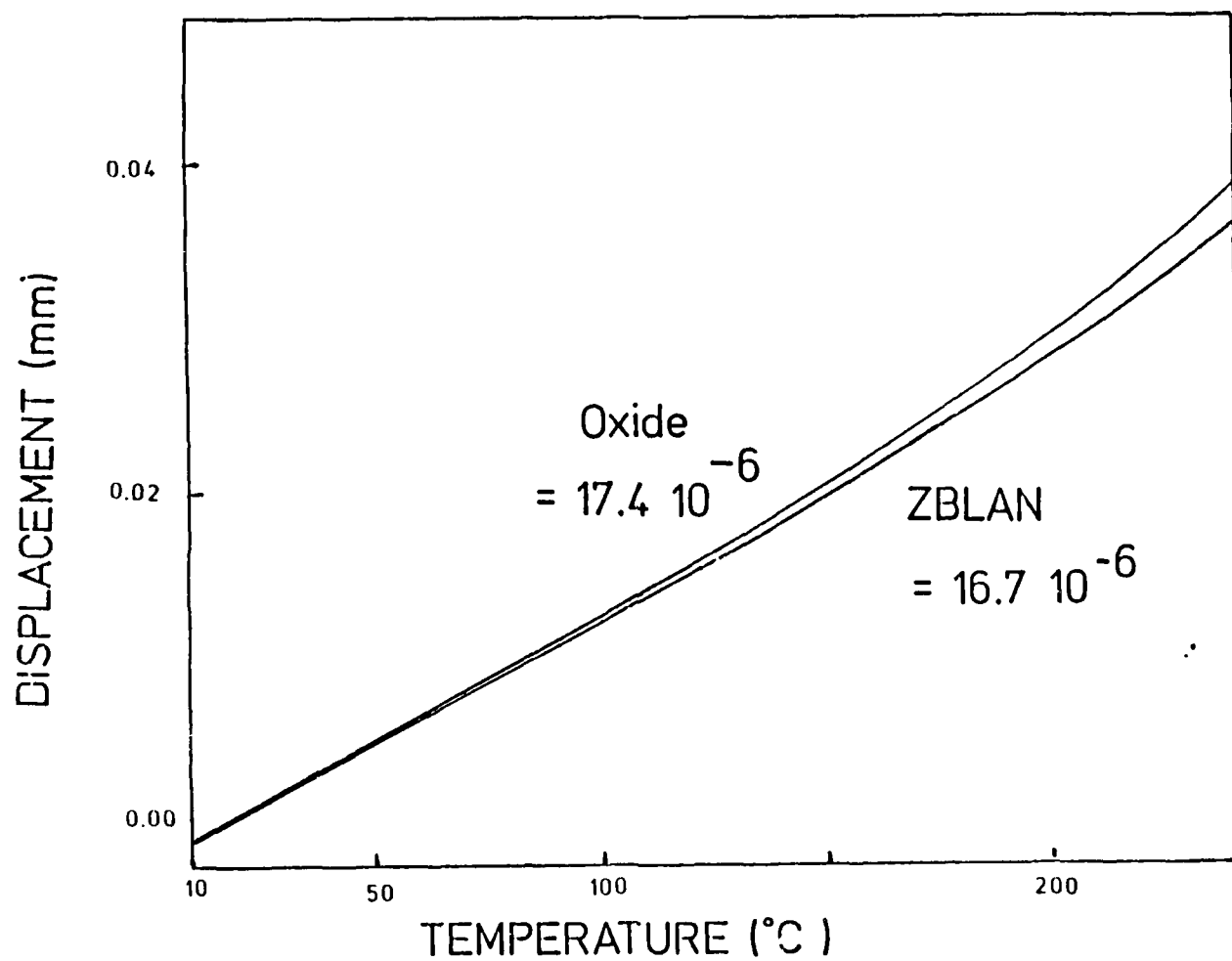
LINEAR EXPANSION OF OXIDE GLASS

FIGURE 10.



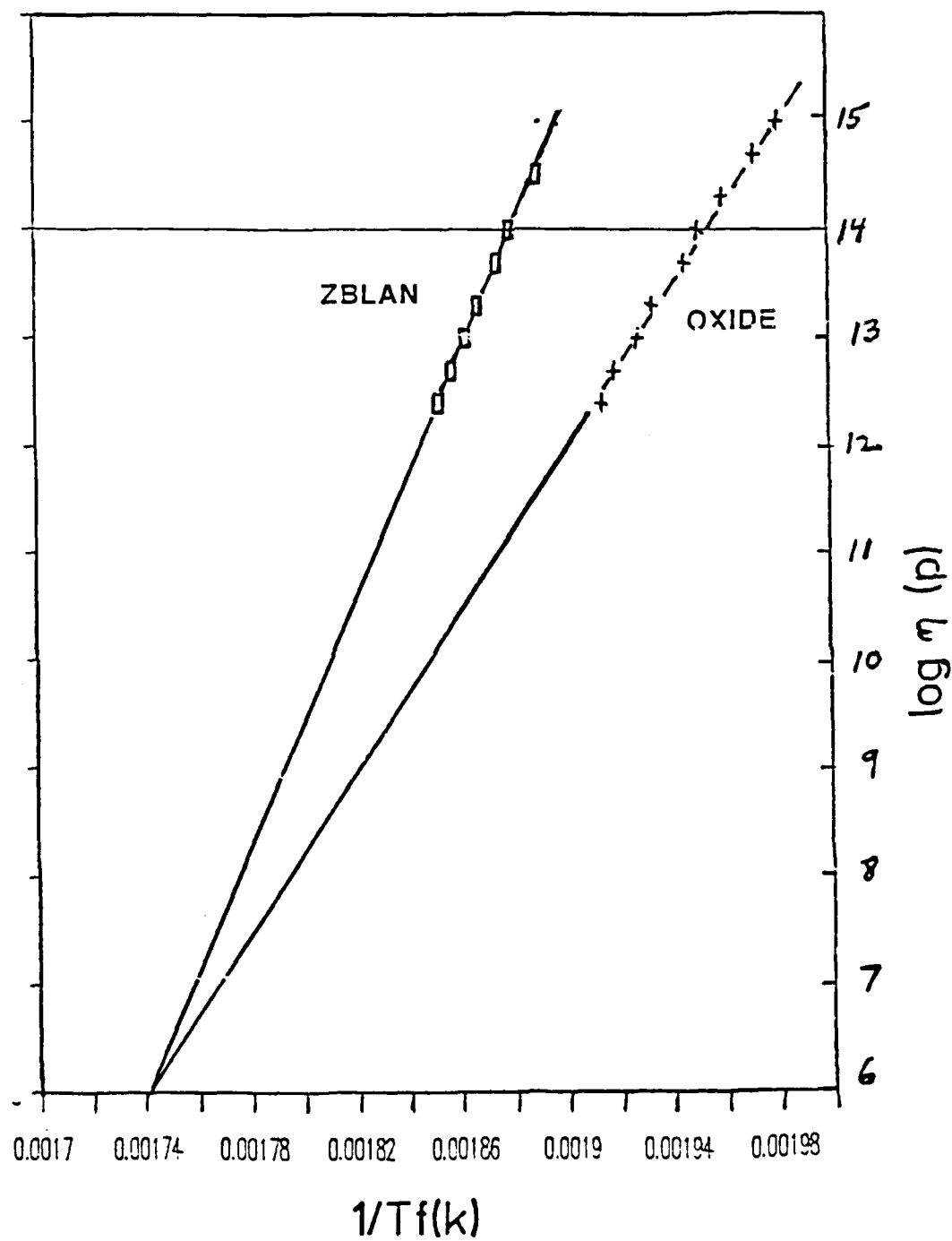
LINEAR EXPANSION OF OXIDE GLASS

FIGURE 11.



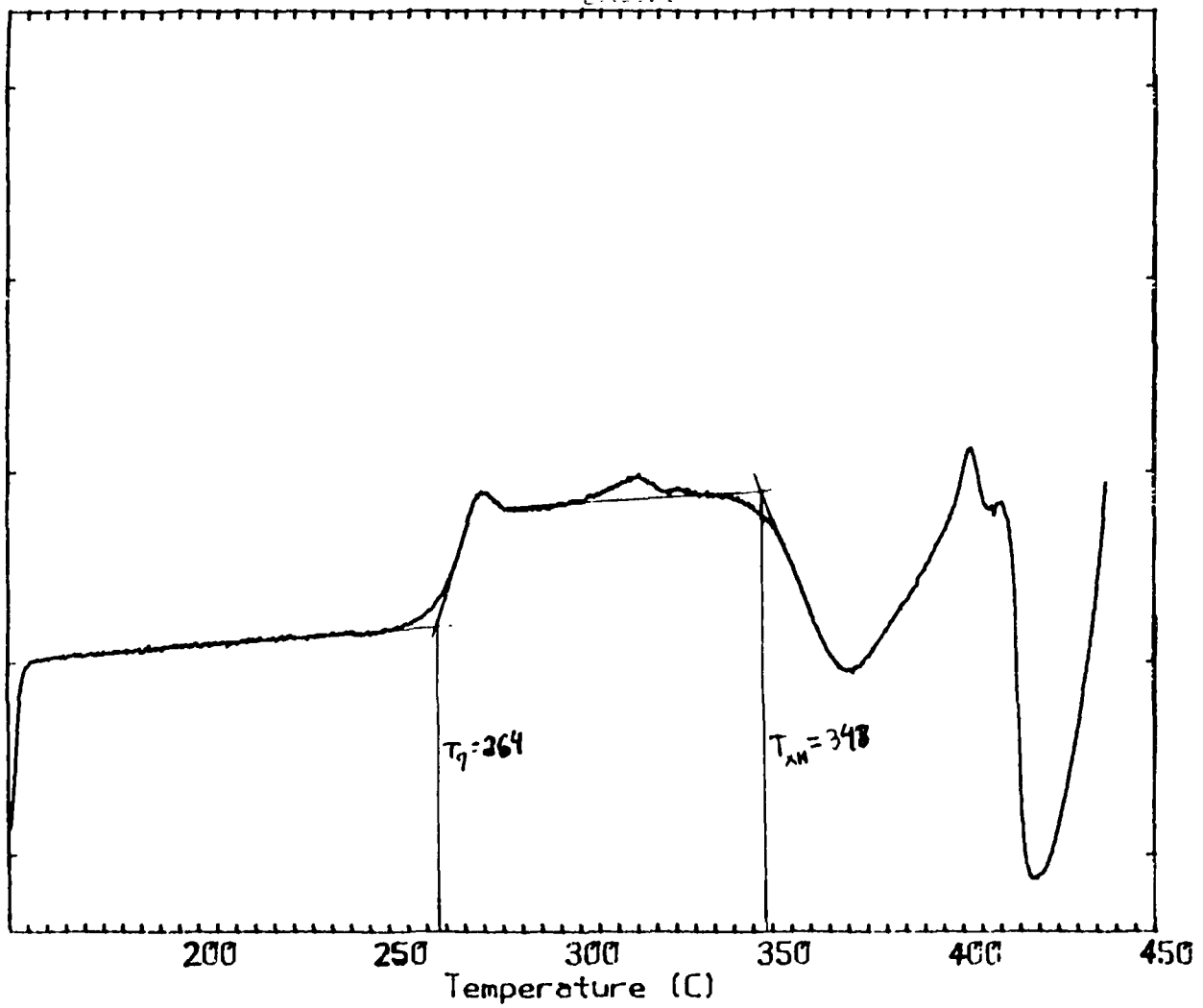
LINEAR EXPANSION CURVES FOR BEST OXIDE GLASS AND ZBLAN GLASS

FIGURE 12.



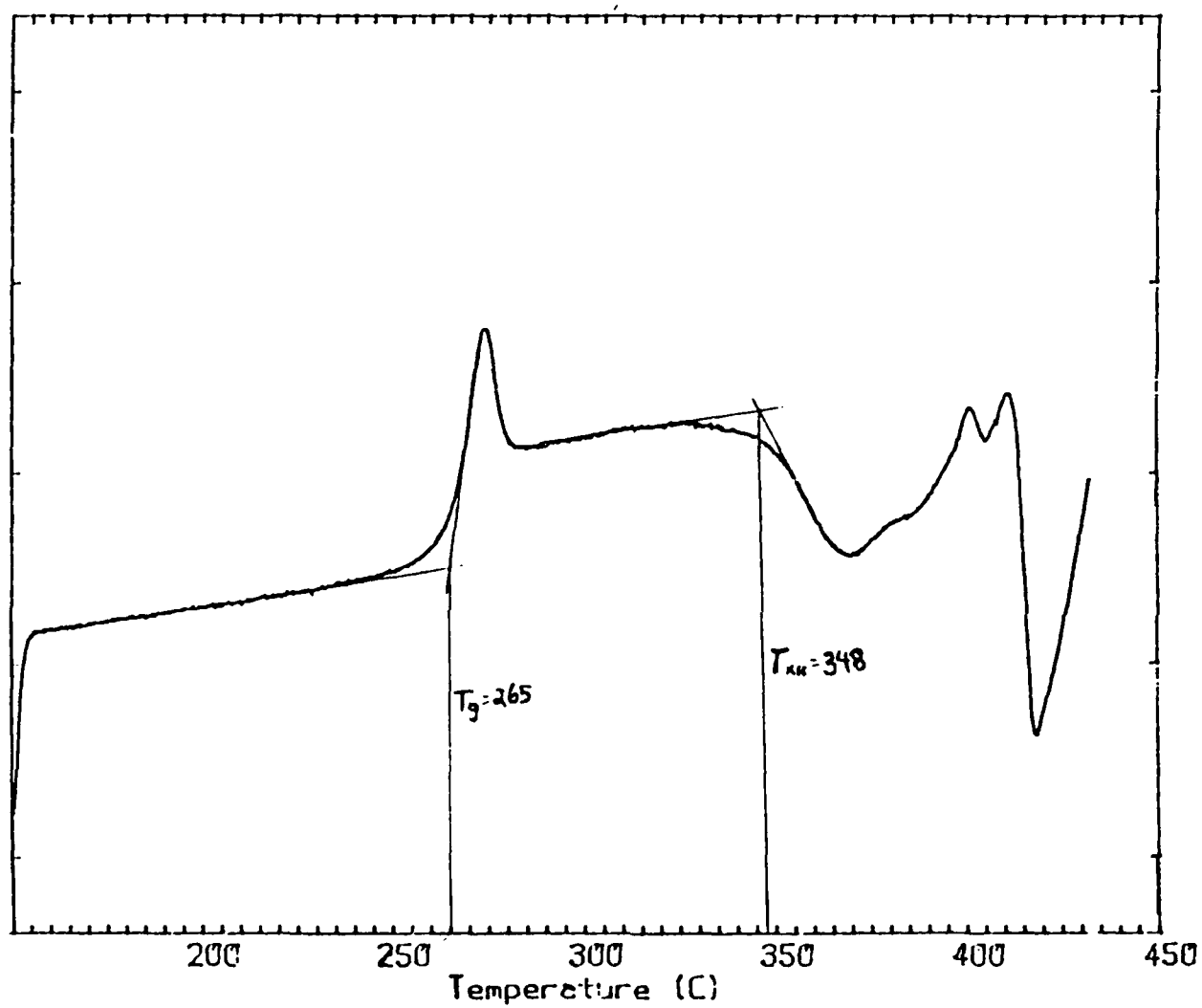
VISCOSITY CURVES FOR ZBLAN AND OXIDE GLASS

FIGURE 13.



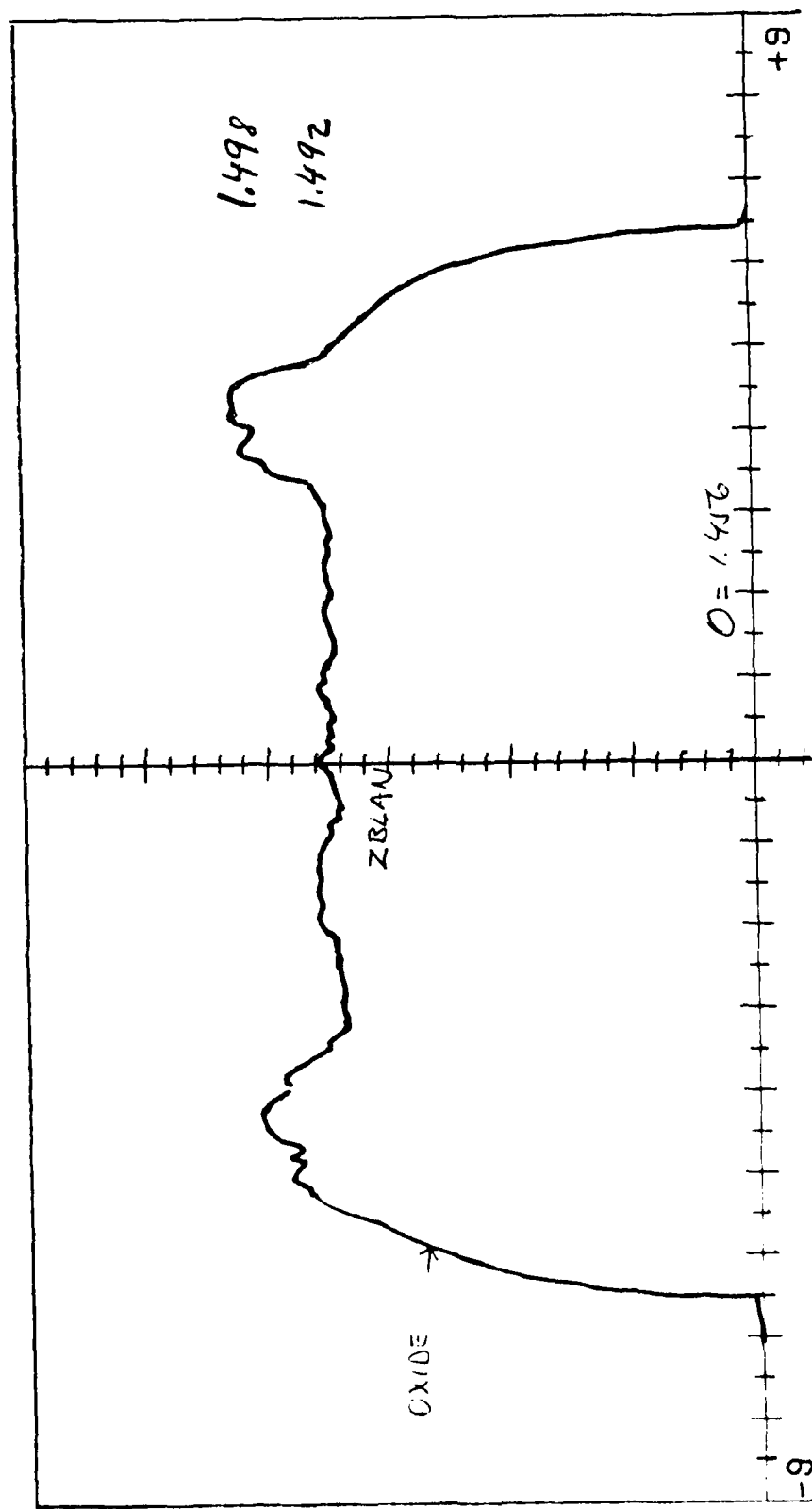
DSC CURVE OF THE FLUORIDE CORE GLASS

FIGURE 14



DSC CURVE OF FLUORIDE CORE WITH OXIDE OVERCLAD GLASS

FIGURE 15.



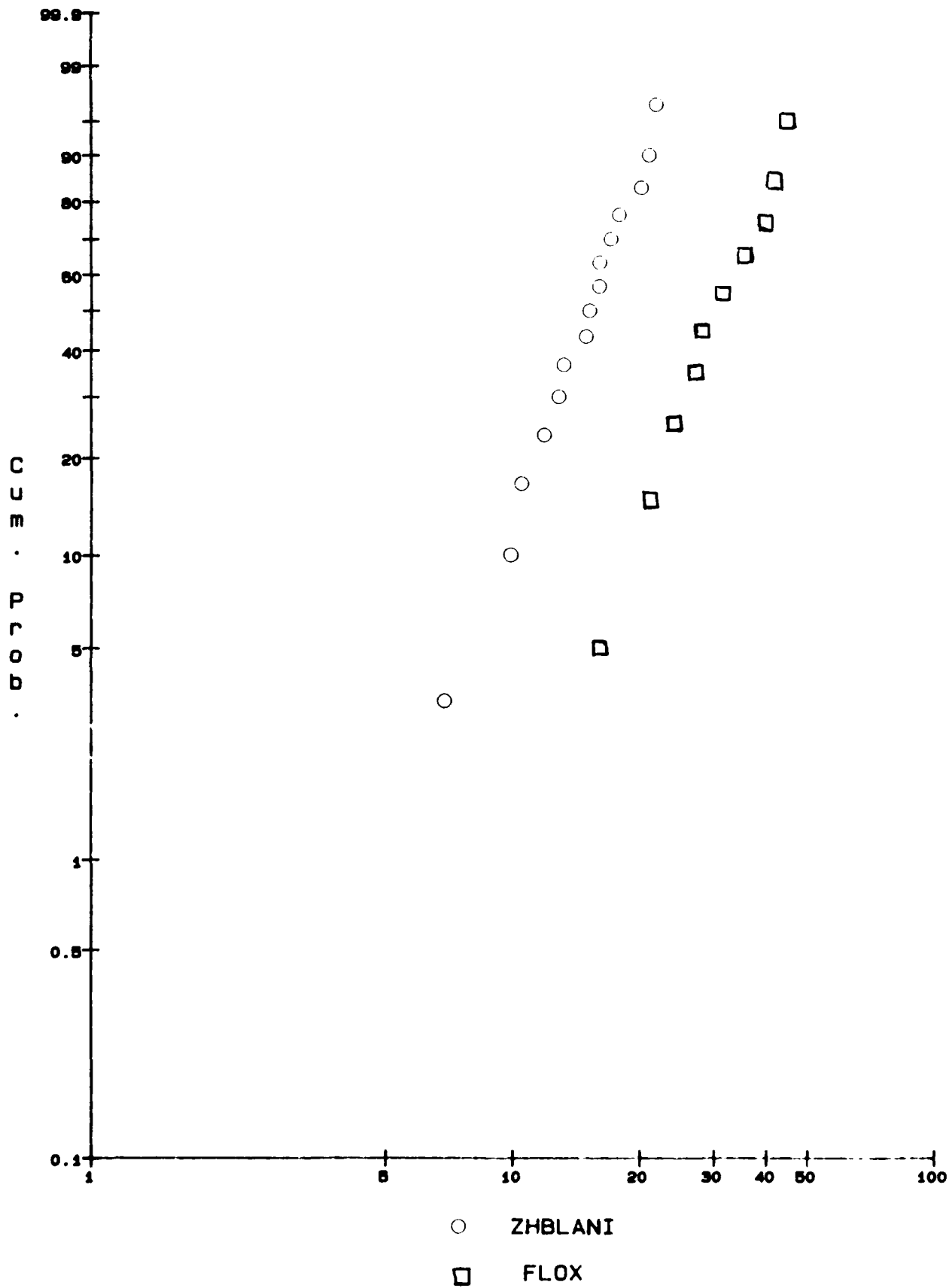
RELATIVE INDEX PROFILE OF A FLUX PREFORM

FIGURE 16.



PHOTOGRAPH OF A CROSS SECTION OF A FLUX PREFORM

FIGURE 17.



WEIBULL PLOT FOR FLUORIDE FIBER AND FLOX FIBER

FIGURE 18.



PHOTOGRAPH OF A 220 μ m LONG FLUORIDE PREFORM WITH 12mm OD

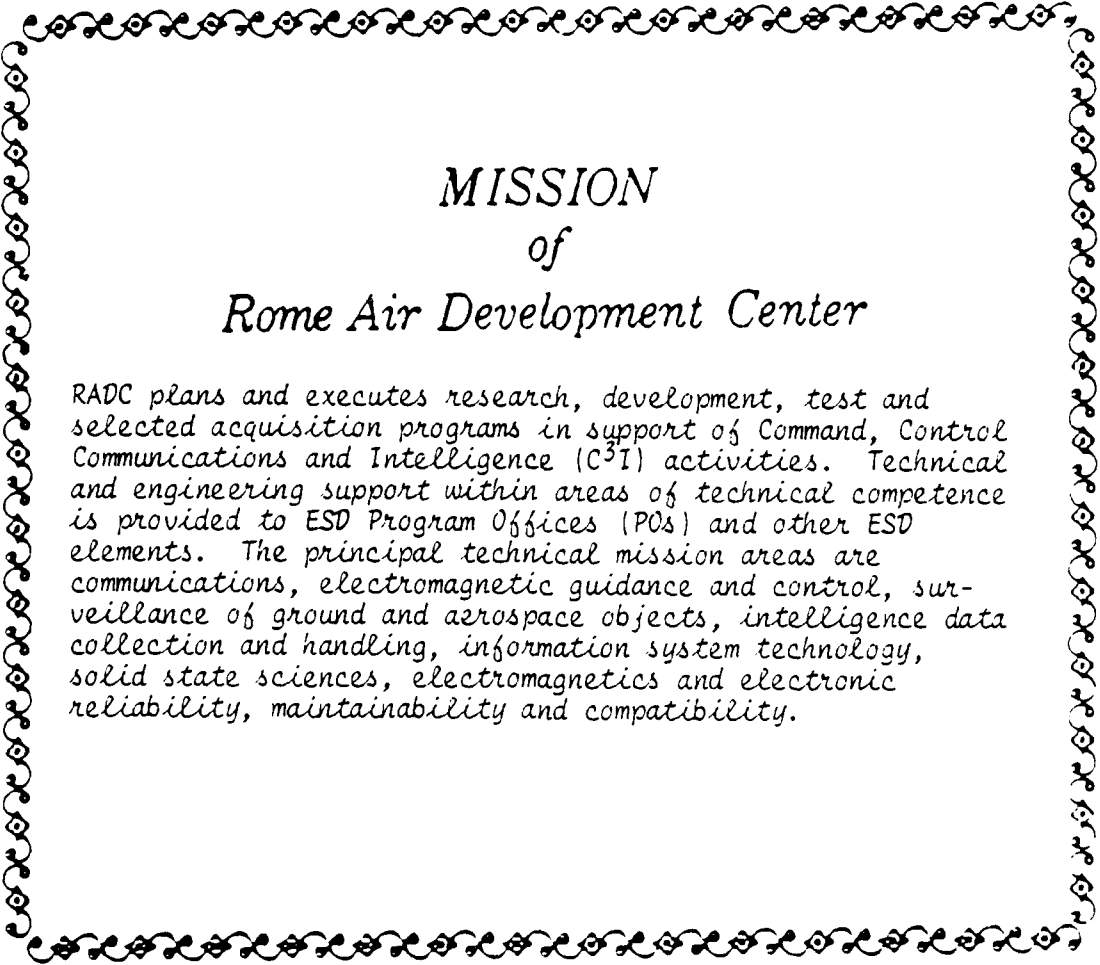
TABLE II

<u>Preform</u>	<u>Oxide</u>	<u>Clad</u>	<u>Core</u>	<u>Mold</u>	<u>Comments</u>
P110	P-1	---	ZBLANI	Br, 2 pc.	Cast the core too hot & crystallized
P111	P-1	---	ZBLANI	Br, 2 pc.	No crystals, some small bubbles.
P112	P-1	---	ZHBLANI	Br, 2 pc.	Didn't form complete tube & cracked when core was poured in.
P113	P-1	---	Hf-2	Br, 2 pc.	Preform cracked, but no crystals.
P114	P-2	---	ZBLANI	S.S., 1 pc.	Preform cracked, large amount of bubbles at interface.
P115	P-2	---	ZBLANI	S.S., 1 pc.	Made only 1/2 tube and cracked when core was poured.
P116	P-2	---	ZBLANI	Br, 2 pc.	Cracked and bubbles in clad, no crystals.
P117	P-1	---	ZBLANI	Br, 2 pc.	Some bubbles and crystals in core.
P118	P-2	---	ZBLANI	S.S., 1 pc.	Pressed clad tube instead of spinning, clad not concentric and cracked.
P119	P-1	ZHBLANI	ZBLANI	S.S., 1 pc.	Good preform, no crystals.
P120	----	ZHBLANI	ZBLANI	Br, 2 pc.	A few crystals scattered throughout & pouring patterns at top.
P121	----	ZHBLANI	ZBLANI	Br, 2 pc.	Clad glass ground in cap, made tube only.
P122	----	ZHBLANI	ZBLANI	Br, 2 pc.	Crystals at interface.
P123	----	ZBLANI	ZBLANI	Br, 2 pc.	Casting technique exp.
P124	----	ZHBLANI	ZBLANI	Br, 1 pc.	Crystals in core and casting pattern at interface.
P125	P-1	ZHBLANI	ZBLANI	S.S., 1 pc.	Nice preform, no crystals, small bubbles in core.
P126	P-1	ZHBLANI	ZBLANI	S.S., 1 pc.	Clad glass ground in cap and caused crystals in core.
P127	P-1	ZHBLANI	ZBLANI	silica tube	Core crystallized and cracked.

<u>Preform</u>	<u>Oxide</u>	<u>Clad</u>	<u>Core</u>	<u>Mold</u>	<u>Comments</u>
P128	P-1	ZHBLANI	ZBLANI	silica tube	Tube broke and glass went right through.
P129	P-1	ZHBLANI	ZBLANI	silica tube	Cracked and had casting patterns in core.
P130	P-1	ZHBLANI	ZBLANI	silica tube	Mold furnace broke in the middle of casting.
P131	P-1	ZHBLANI	ZBLANI	Silica in Br	Bad geometry in fluoride & oxide cladding, large bubbles & small crystals in core.
P132	P-1	ZHBLANI	ZBLANI	Silica in Br	Bubbles & pouring pattern throughout core.
P133	P-1	ZHBLANI	ZBLANI	Silica in Br	Pouring pattern in core
P134	P-1	ZHBLANI	ZBLANI	Silica in Br	Fluoride clad crystallized prior to casting.
P135	P-1	---	ZBLANI	S.S., 1 pc.	Preform stuck in mold, no crystals, many tiny bubbles.
P136	P-1	---	ZBLANI	S.S., 1 pc.	Didn't form complete cladding tube & condensation from crucible fell into core.
P137	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Preform stuck to mold, a couple of large pieces of junk fell into core.
P138	P-1	ZHBLANI	ZBLANI	S.S., 1 pc.	Unable to get preform out of mold without pulverizing it.
P139	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Cladding glass ground in cap and fell into tube.
P140	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Many tiny crystals or bubbles at interface.
P141	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Rotated mold while casting core; core was loaded with crystals.
P142	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Upper region of cladding crystallized when it came in contact with plug and core glass flowed crystals throughout the core.

<u>Preform</u>	<u>Oxide</u>	<u>Clad</u>	<u>Core</u>	<u>Mold</u>	<u>Comments</u>
P143	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Lowered mold temp. dramatically prior to casting core to avoid remelting of clad and causing crystallization.
P144	----	ZHBLANI	ZBLANI	tapered Br., 1 pc.	Used slightly higher mold temp. when casting core to avoid shocking clad resulted with tiny crystals and large bubbles in core.
P145	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	High purity mat'l., bubbles and flow pattern throughout and crystals in upper region.
P146	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	Almost crystal free, slight cracks in clad and bubbles; high purity core.
P147	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	No crystals; many tiny bubbles in core, cast core straight down the bore of cladding tube, tiny cracks in clad.
P148	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	A few crystals in cladding, none in core, many tiny bubbles in core.
P149	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	No crystals, minor cracks in cladding, high purity core.
P150	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	High purity core, no crystals, small to large bubbles in core.
P151	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	Used bottom drain crucible for core, no crystals in core, crystals in clad and small to large bubbles in core.
P152	P-1	---	ZBLANI	1 pc., S.S., tapered	A few small crystals in core and core only filled 1/2 of tube.
P153	P-1	ZHBLANI	ZBLANI	1 pc., S.S., tapered	Many tiny bubbles in core and hazy outside surface. Preform cracked.
P154	P-1	ZHBLANI	ZBLANI	1 pc., S.S., tapered	No crystals, bubbles in core, preform cracked.
P155	P-1	ZHBLANI	ZBLANI	1 pc., Br., tapered	Fluoride core & clad cracked, oxide overclad didn't, bubbles but no crystals in core.

<u>Preform</u>	<u>Oxide</u>	<u>Clad</u>	<u>Core</u>	<u>Mold</u>	<u>Comments</u>
P156	P-1	ZHBLANI	ZBLANI	1 pc., Br., tapered	Large bubbles in core, tiny bubbles at interface, no crystals or cracks.
P157	----	ZHBLANI	ZBLANI	1 pc., Br., tapered	Top 1/4 was crystallized, small bubbles and a few crystals in core.
P158	P-1	ZHBLANI	ZBLANE	2 pc., Br. mold	Small bubbles in core, no crystals.
P159	----	ZHBLANI	ZBLANI	1 pc., Br. mold	Thin cladding wall, large core, many tiny bubbles in core.
P160	----	ZHBLANI	ZBLANI	Cu (10" preform)	No crystals, row of many tiny bubbles down center of core.
P161	----	ZHBLANI	ZBLANI	Cu (10" preform)	Very few crystals and very few bubbles, some sort of pattern at interface that looks like air pockets.
P162	----	ZHBLANI	ZBLANI	Cu (10" preform)	Medium to large bubbles extending 2" up from bottom, no crystals in that region. A few crystals scattered from that point to the top of preform.
P163	P-1	ZHBLANI	ZBLANI	1 pc., S.S.	Oxide tube cracked and peeled off of fluoride preform.



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