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# INVESTIGATION OF THE KINETICS OF CRYSTALLIZATION OF MOLTEN BINARY AND TERNARY OXIDE SYSTEMS

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by

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Investigation of the Kinetics of Crystallization of

Molten Binary and Ternary Oxide Systems

Quarterly Status Report No. 14 - December 1, 1968 through February 28, 1969

Contract No. NASW-1301

SUMMARY

This report is based on the research studies carried out in the fourteenth quarter of Contract NASW-1301, which started December 1, 1968 and ended February 28, 1969. Twenty-five new glass compositions were originated, prepared, and partially characterized in this period. Similar characterization measurements comprising density, Young's modulus, and fiberizability measurements were completed for some of the compositions reported earlier.

At the time of this report, a total of eleven glass compositions have been developed with values for Young's modulus in excess of twenty million psi and another two dozen with values for Young's modulus lying between eighteen and twenty million psi. Possibly the most significant of these glasses is UARL 350, which while not having the highest value for Young's modulus since it has a Young's modulus of 19.8 million psi, has a specific modulus of 197 million inches. These values can perhaps best be interpreted by comparison with other common structural materials.

	<u>Young's Modulus</u> <u>(millions psi)</u>	<u>Specific Modulus</u> <u>(10<sup>7</sup> inches)</u>
E glass	10.5	11.4
Owens-Corning Pilot Plant X-2285	14.95	16.65
Steel	29	10.3
Molybdenum	52	14.1
Boron nitride	13	18.8
UARL 350	19.8	19.7

Experiments on the fiberization of these new glasses are not yet complete. At report time the best data on the experimental glass compositions in fiber form is for UARL 331 which gave a value for Young's modulus of 19.8 million psi and a specific modulus of 150 million inches.

## INTRODUCTION

This is the fourteenth quarterly report for Contract NASW-1301 entitled "Investigation of the Kinetics of Crystallization of Molten Binary and Ternary Oxide Systems". The fourteenth quarter started December 1, 1968 and ended February 28, 1969 and forms the first quarter of the fourth nine month extension to the contract, an extension which runs from December 1, 1968 through August 30, 1969.

The primary objective of the contract is to gain a better understanding of the mechanisms of glass formation by measuring the rate at which crystallization occurs and the effect of antinucleating agents on the observed crystallization rate for systems which tend to form complex three-dimensional structures. The molten oxide systems selected for study, the reasons for their selection, and the methods used to prepare them form the first major section of our report.

Characterization of the experimental glasses produced as bulk specimens is the subject of the second major section of the report. Such characterization is largely achieved by measuring the density and Young's modulus for bulk samples of the experimental glasses. Originally, specimens for measuring Young's modulus of bulk glasses were made by first casting a glass slab, annealing it, and then cutting rectangular bars from the slab by precision optical grinding techniques. Now, however, we form samples suitable for modulus determinations by pulling molten glass into fused silica tubes by means of controlled suction supplied by a hypodermic syringe so that measurements of Young's modulus are both much cheaper and easier to make.

Observations of the fiberizability of the various melts and determinations of Young's modulus on mechanically drawn fibers by measurements on the usual tensile test equipment form the final major section of the report.

An abstract of an article submitted for presentation at the joint meeting of the International Commission on Glass and the Canadian Ceramic Society to be held in Toronto, Canada next September is also included.

SELECTION AND PREPARATION OF GLASS SYSTEMS  
FOR PRELIMINARY EVALUATIONLow Atomic Number Oxide Components are Primary  
Though Not the Only Choice

Since the ultimate long-range objective of this program is the attainment of high-modulus, high-strength-to-density continuous vitreous fibers, research will be largely concentrated on complex molecules composed of low atomic number oxides such as those shown in the tabulation below (Ref. 1).

## Moduli-Density Values for Several Low Atomic Number Oxides

Oxide	Young's Modulus ( $10^6$ psi)	Density (gms/cm <sup>3</sup> )	Maximum Strength (5% strain - $10^6$ psi)	Modulus Density Ratio $10^6$ psi/gms/cm <sup>3</sup>
Al <sub>2</sub> O <sub>3</sub>	76	4.0	3.8	19
BeO	51	3.0	2.6	17
MgO	35	3.5	1.8	10
SiO <sub>2</sub>	10.5	2.2	0.53	5
MgO Al <sub>2</sub> O <sub>3</sub>	35	3.6	1.8	10
TiO <sub>2</sub>	41	4.26	2.06	9.6

For this reason, specific molecular systems considered have been silicates (since silica is the best-known glass former) such as cordierite, Al<sub>3</sub>Mg<sub>2</sub>(Si<sub>5</sub>Al)<sub>0</sub><sub>18</sub>, known to have a ring crystal structure with ions arranged in sheets but not a layer structure (Ref. 2); benitoite, BaTiSi<sub>3</sub>O<sub>9</sub>, likewise a complex three-dimensional structure (Ref. 2); and beryl, Be<sub>3</sub>Al<sub>2</sub>Si<sub>6</sub>O<sub>18</sub>, a ring structure like that of cordierite. The low value for silica in contrast to the other oxides indicates that the percentage of silica in such a glass must be held at a minimum if high moduli are to be achieved. The major constituent, silica, which provides the glass structure and the desired viscosity characteristics, can be regarded as a necessary evil and the positive direction for modulus improvement clearly lies in using no more silica than necessary and so leads to the consideration of invert analog glasses as we show in a later section.

Although low atomic number oxides are the most promising ingredients for high modulus glass fibers, they are not the only allowable constituents. In our earlier report, UARL G910373-10, we showed that based on UARL experimental data and the method of calculation introduced by C. J. Phillips (Ref. 3) contributions per mol % to Young's modulus of several of the heavier elements were high:

Oxide	Contribution to Young's Modulus per mol % (kilobars)
SiO <sub>2</sub>	7.3
Al <sub>2</sub> O <sub>3</sub>	12.1
CaO	12.6
Li <sub>2</sub> O	7.0
B <sub>2</sub> O <sub>3</sub>	7.2
ZnO	1.72 and rising with increasing R <sub>2</sub> O
TiO <sub>2</sub>	13.3
BeO	19.0
ZrO <sub>2</sub>	18.9
MgO	12.0 and rising with decreasing R <sub>2</sub> O & SiO <sub>2</sub> to 14.8
Ce <sub>2</sub> O <sub>3</sub>	18.6
Y <sub>2</sub> O <sub>3</sub>	24.3
La <sub>2</sub> O <sub>3</sub>	22.4

The judicious use of several of these heavier oxides is obviously to be considered in cases where they improve the viscosity, surface tension, working range and other characteristics contributing to fiberizability.

#### Results with Cordierite Glasses are Promising

A glass field in which UARL has carried out extensive research is that of the cordierite glass system to which rare earths have been added as major constituents. Cordierite or  $Mg_2Al_4Si_5O_{18}$  is a three-dimensional ring former, as discussed in earlier UARL reports (UARL E910373-4, for example) and these glasses include major quantities of one of the rare earths such as lanthana, ceria, or yttria which, as UARL has shown (UARL G910373-11), actively delay the onset of devitrification in cordierite glasses while at the same time increasing their elastic modulus. In addition to the rare earths, zirconia has been found to have similar beneficial results. The compositions of the glasses are shown in Table I. In Table III the results for cordierite-type glasses are marked with the superscript 3. It will be noted that UARL 125 with no toxic ingredients has a Young's modulus of 16.1 million pounds per square inch and a specific modulus of 161 million inches. Corresponding numbers for other nontoxic cordierite-base glasses are UARL 304 with 19.2 and 147; UARL 337, 20.9 and 147; UARL 363 with 19.3 and 150.5. With beryllia added to the cordierite-base, UARL 323 shows a Young's modulus of 18.4 million pounds per square inch and 184 million inches for its specific modulus; UARL 344 has 20.3 and 168 and UARL 345 gives 21.1 and 174.5. The fibers made from these glasses have not yet been evaluated.

#### Glasses Analogous to "Invert" Glasses Show Best Results to Date

The very new UARL experimental glasses considered in this section of the report belong to a region of glass composition analogous to Stevels' "invert" glasses.

The concept developed by J. M. Stevels and his associates from 1954 on was that by a proper combination of oxides, stable metasilicate glasses could be obtained. A typical example cited by Stevels is 50 mol %  $SiO_2$  and 12.5 mol % of the four materials  $Na_2O$ ,  $K_2O$ ,  $CaO$ ,  $BaO$ . Dr. Stevels explained this "anomalous" case of glass formation by saying that "by choosing a batch with a great number of network modifiers the 'glue' between the chains is so irregular that crystallization is prevented." Obviously, by using a combination of alkali oxides and a combination of alkaline earth oxides as preferred by Stevels, the liquidus temperature can be lowered and the field of glass formation can be increased.

Weyl (Ref. 4) states further that Trap and Stevels characterized the coherence of their silicate glasses by a structural parameter  $Y$  denoting the average number of bridging ions per  $SiO_4^{4-}$  tetrahedron. This parameter may be calculated from the expression

$$Y = 6 - \frac{200}{P} \quad \text{where } P = \text{mol \% } SiO_2$$



Table I

New Experimental Glass Batches  
Actual Ingredients in Grams

<u>Actual Ingredient</u>	<u>330</u>	<u>331</u>	<u>332</u>	<u>333</u>	<u>334</u>	<u>335</u>	
Silica	129.2	148.8	123.8	142.1	117.9	124.3	
Aluminum Oxide (C.P.)	67.4	77.5	64.7	74.0	85.8	90.4	
Magnesium Oxide (C.P.)	---	---	53.4	60.9	67.8	---	
Yttrium Oxalate	---	460	---	439	---	---	
Lanthanum Oxalate	466	---	447	---	395	416	
Beryllium Carbonate	98.3	113	---	---	---	121.8	
Zinc Carbonate (C.P.)	82.9	95.4	79.6	91.0	70.2	74.2	
	<u>336</u>	<u>337</u>	<u>338</u>	<u>339</u>	<u>340</u>	<u>341</u>	
Silica	140.7	107.6	93.8	98.5	106.2	109.5	
Aluminum Oxide (C.P.)	102.5	91.2	79.3	83.5	72.2	61.9	
Magnesium Oxide (C.P.)	---	72.1	62.8	---	56.8	48.9	
Yttrium Oxalate	405	450	---	---	421	---	
Lanthanum Oxalate	---	---	459	480	---	428	
Beryllium Carbonate	143.1	---	---	116.9	75.3	42.7	
Zinc Carbonate (C.P.)	84.2	93.3	81.3	85.5	88.4	75.9	
Lithium Carbonate (C.P.)	---	---	---	---	52.2	45.0	
	<u>342</u>	<u>343</u>	<u>344</u>	<u>345</u>	<u>346</u>	<u>347</u>	
Silica	127.4	111.6	181.0	156.9	78.0	190.9	
Aluminum Oxide (C.P.)	71.9	63.1	102.3	105.9	66.0	53.9	
Magnesium Oxide (C.P.)	28.4	24.9	40.5	---	26.1	---	
Yttrium Oxalate	427	---	405	418	391	---	
Lanthanum Oxalate	---	435	---	---	---	371	
Zirconium Carbonate(nominal)	---	---	---	---	89.8	---	
Beryllium Carbonate	100.7	88.1	71.8	148.0	92.2	113.3	
Zinc Carbonate (C.P.)	88.2	77.5	---	---	81.1	66.5	
Lithium Carbonate (C.P.)	52.1	45.6	---	---	47.9	---	
	<u>348</u>	<u>349</u>	<u>350</u>	<u>351</u>	<u>352</u>	<u>353</u>	<u>354</u>
Silica	182.9	190.9	144.7	209.5	94.0	111.5	128.5
Aluminum Oxide (C.P.)	51.8	53.9	133.1	108.3	63.9	62.0	62.3
Magnesium Oxide (C.P.)	---	---	52.7	42.8	35.4	25.1	12.4
Yttrium Oxalate	---	---	---	---	378	374	369
Lanthanum Oxalate	357	---	---	---	---	---	---
Zirconium Carbonate(nominal)	70.0	---	---	---	86.7	85.7	84.8
Beryllium Carbonate	108.3	113.3	174.5	137.5	45.7	44.0	43.7
Zinc Carbonate (C.P.)	---	66.5	---	---	78.4	77.7	76.7
Lithium Carbonate (C.P.)	---	---	96.2	78.4	41.2	45.8	45.2
Calcium Carbonate (C.P.)	---	---	130.8	106.2	---	---	---
Cerium Oxalate	---	371	---	---	---	---	---

Table I (Cont'd)

Actual Ingredient	<u>355</u>	<u>356</u>	<u>357</u>	<u>358</u>	<u>359</u>	<u>360</u>
Silica (SiO <sub>2</sub> )	138.9	157.0	128.1	146.7	137.6	153.0
Alumina (Al <sub>2</sub> O <sub>3</sub> )	72.4	82.4	66.8	76.5	71.8	79.8
Magnesia (MgO)	57.9	65.2	52.7	60.4	56.8	63.1
Yttrium Oxalate	358	---	91.2	378	355	394
Zirconium Acetate	---	220	---	---	---	---
Cerium Oxalate	125.3	142.6	---	---	---	---
Rare Earth Oxalate	---	---	385	---	---	---
Zinc Carbonate	44.5	50.7	41.2	47.0	---	49.2
Lithium Carbonate	26.2	28.8	24.1	27.4	21.6	28.9
Vanadium Pentoxide	---	---	---	34.2	---	---
Zinc Tungstate	---	---	---	---	92.8	---
Cobaltous Carbonate	---	---	---	---	---	31.4
	<u>361</u>	<u>362</u>	<u>363</u>	<u>364</u>	<u>365</u>	<u>366</u>
Silica (SiO <sub>2</sub> )	148.1	152.2	154.0	182.1	144.5	159.0
Alumina (Al <sub>2</sub> O <sub>3</sub> )	77.0	80.0	80.2	95.2	75.4	82.4
Magnesia (MgO)	60.9	62.9	63.5	75.2	59.5	---
Beryllium Carbonate	---	---	---	55.3	26.4	120.5
Yttrium Oxalate	382	393	396	---	372	---
Zirconium Acetate	---	---	---	---	---	268.0
Cerium Oxalate	---	---	---	164.5	130.3	143.3
Zinc Carbonate	47.5	48.8	---	58.5	---	76.5
Lithium Carbonate	27.9	28.8	29.1	34.4	27.3	---
Ferric Oxide	30.2	---	---	---	---	---
Fused Boric Acid	---	---	48.7	---	---	---
Copper Oxide	---	15.6	15.7	---	---	---
	<u>367</u>	<u>368</u>	<u>369</u>	<u>370</u>	<u>371</u>	<u>372</u>
Silica (SiO <sub>2</sub> )	154.8	149.7	150.8	147.7	150.0	128.7
Alumina (Al <sub>2</sub> O <sub>3</sub> )	80.8	78.2	78.8	77.2	78.3	67.2
Magnesia (MgO)	31.9	15.5	---	---	10.3	---
Beryllium Carbonate	117.5	114	114.7	112.3	114.3	97.9
Yttrium Oxalate	396	385	389	382	387	---
Lanthanum Oxalate	---	---	---	---	---	77.3
Cerium Oxalate	93.2	90.1	90.7	88.8	90.3	---
Rare Earth Oxalate	---	---	---	---	---	388
Zinc Carbonate	---	47.9	---	47.5	32.2	82.7
Calcium Carbonate	---	---	77.2	37.7	25.5	---

Table I (Cont'd)

<u>Actual Ingredient</u>	<u>373</u>	<u>374</u>
Silica (SiO <sub>2</sub> )	153.9	151.3
Alumina (Al <sub>2</sub> O <sub>3</sub> )	80.3	84.0
Beryllium Carbonate	104.2	122.3
Yttrium Oxalate	396	416
Cerium Oxalate	92.6	---
Zinc Carbonate	---	57.8
Lithium Carbonate	29.0	30.4
Copper Oxide	---	11.0
Calcium Carbonate	39.4	---

so that when  $P = 33 \frac{1}{3}$ ,  $Y = 0$  and  $\text{SiO}_4^{4-}$  groups are isolated; when  $P = 40\%$ ,  $Y = 1$  and on the average  $\text{SiO}_4^{4-}$  groups appear in pairs. Commercial silicate glasses, on the other hand, have  $Y$  values between 3.0 and 3.5 in agreement with the Zachareisen rules for stable glass formation which state that a stable silicate glass should consist of  $\text{SiO}_4^{4-}$  tetrahedra sharing at least three of their corners with other  $\text{SiO}_4^{4-}$  tetrahedra. On the other hand, the "invert" glasses developed by Trap and Stevels have  $Y$  values lower than 2.0 in direct contradiction of the accepted rules for stable glass formation.

When the glass composition was changed to lower and lower  $\text{SiO}_2$  concentrations, Trap and Stevels (Ref. 4) found that some properties such as thermal expansivity, electrical deformation losses, and viscosity go through maxima or minima reaching extreme values as the parameter  $Y$  passes through the value 2.0. It is the feeling at this laboratory, UARL, that the modulus of elasticity may likewise achieve a decided maximum. However, extensive research with the "invert" glasses in the first eight quarters of this contract failed to yield any marked change in modulus. The ninth and tenth quarters, however, yielded a sixty percent increase in modulus while research in the eleventh quarter resulted in an invert glass with an elastic modulus in excess of twenty million psi as will be shown in a later section and recent quarters have produced another eight "invert" glasses with equally high or higher moduli.

Stevels' "invert" glasses comprised silica, two or more monovalent oxides, usually  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ , and two or more alkaline earth oxides. The UARL glasses, on the other hand, although analogous to Stevels' invert glasses, consist of silica, lithia, two or more divalent oxides or fluorides, one or more trivalent oxides, and may include a second tetravalent oxide or a pentavalent oxide. This combination of divalent and trivalent oxides has proven equally effective in blocking crystallization while yielding higher moduli. The glasses of Table I, compositions 266 through 277, may be considered typical UARL invert analogues.

In Table III the results for "invert" analogue glasses are marked with a superscript 1. As is evident from the table, "invert" analogue glass 329 with no toxic ingredients has a measured Young's modulus of 20.7 million pounds per square inch and a specific modulus of 189 million inches while for 337, also a nontoxic composition, the corresponding numbers are 20.7 million pounds per square inch and 147 million inches. When beryllia is added to the "invert" analogue glasses these numbers are for UARL 325, 20.2 million pounds per square inch and 158 million inches; for 331, 20.9 and 158; for 336, 21.0 and 166; for 340, 20.9 and 163; for 343, 19.4 and 140; for 347, 21.6 and 164; for 350, 19.8 and 197; and for 352, 20.0 and 146. Fibers with a Young's modulus of 19.8 million psi have been produced for UARL 331 while fibers from the others have yet to be measured and in some cases yet to be made.

Just as in the case of our earlier experimental compositions, once a composition has been selected, 500-gram batches of the specified raw materials are melted in high purity (99.9%) alumina crucibles in air using kilns heated by Super-kanthal hairpin electrical resistance elements. Starting materials used are 5-micron particle-size high purity silica, high purity alumina of 325 mesh, laboratory reagent grade magnesium oxide, 99.9% lanthanum oxalate, and

other comparable materials such as reagent grade zinc carbonate or calcium carbonate. These materials customarily yield a water-white optical grade glass free of seed, stone and bubbles when properly compounded and held at temperatures of 1000-1650°C for at least two hours. Less commonly, glasses may be prepared in beryllia crucibles in air and in the same kilns, or in platinum crucibles in air in the platform kiln which is heated by the high-temperature variety of Superkanthal heating elements and can reach temperatures of 1700°C, or in tungsten crucibles in purified argon or vacuum atmospheres. Alumina crucibles of even very slightly lower purity, i.e., 99.3 to 99.7% have not proven useful for this type of glass research. In some cases where marked departure occurs from the more usual optical clarity, the melt may be ground and remelted in platinum before forming the rods used for Young's modulus measurement and before drawing any fibers. In the future the technique of pouring from one crucible to another as suggested by Stirling and Miselbach (Ref. 5) will be used in the case of any glasses that fail to appear homogeneous.

#### CHARACTERIZATION OF UARL EXPERIMENTAL GLASSES

Most of the experimental glasses prepared in this and earlier quarters have been characterized by measuring the density and Young's modulus on bulk samples. The reproducibility of the Young's modulus measurements made on the aspirated rods has also been examined.

##### Density Measurements

Density of the experimental glasses is determined for UARL by the Glass Testing Division of the Hartford Division of the Eshart Corporation. For samples with densities less than 3.00 gms/cm<sup>3</sup> the heavy-liquid-of-known-density comparison procedure is used while for samples with densities greater than 3.00 gms/cm<sup>3</sup> the Archimedean method is employed. The results of all density measurements are shown in Table II in both metric and English units. The observed densities range from 2.20 to 5.22 gms/cm<sup>3</sup>. Possibly the only noteworthy deviation from predicted density is provided by UARL 284 where a partial substitution of copper for zinc lowers the density from 3.52 to 3.23 gms/cm<sup>3</sup> but numerous small deviations in density are apparent when the density is plotted against molal sum and straight line relationships are only approximately obeyed by these experimental glasses.

##### Young's Modulus for Glass Rods Formed by Aspiration Directly from Melt

Samples for modulus measurement are prepared using the simple and inexpensive technique of drawing samples directly from the crucibles of molten glass into fused silica tubes previously dusted lightly with powdered magnesia. Controlled suction for pulling the sample into the fused silica tube is supplied by a hypodermic syringe. The fact that all of the experimental glasses have coefficients of

Table II

Summary of all Density Determinations for Bulk  
Specimens of UARL Experimental Glasses

<u>Number</u>	<u>Density gms/cm<sup>3</sup></u>	<u>0.03613 Density Pounds/in.<sup>3</sup></u>	<u>Number</u>	<u>Density gms/cm<sup>3</sup></u>	<u>0.03613 Density Pounds/in.<sup>3</sup></u>
25	2.5672	0.0938	125	2.7818	0.1000
38	2.6415	0.0965	126	3.4634	0.1250
40-3	2.9574	0.1067	127	3.2557	0.1173
56	2.4368	0.0879	131	3.1386	0.1132
62-3	2.7404	0.0989	134	3.0671	0.1107
63-1	2.6847	0.0970	135	2.6303	0.0946
64-1	2.6818	0.0968	136	2.8035	0.1012
65-1	2.7197	0.0983	137	3.0834	0.1113
66	2.6112	0.0943	138	3.5498	0.1282
66-1	2.6784	0.0968	140	3.678	0.1329
67-3	2.6499	0.0957	151	3.2541	0.1175
68-2	2.6295	0.0950	155	3.5452	0.1282
69	2.5910	0.0935	157	2.6962	0.0972
69-3	2.5952	0.0935	159	3.2216	0.1163
70-1	2.7526	0.0993	160	3.2211	0.1161
71	2.6627	0.0962	161	3.4523	0.1247
72-2	2.8877	0.1043	162	3.6150	0.1307
73-2	3.0152	0.1088	163	3.1876	0.1152
74	2.9983	0.1082	164	4.05973	0.1465
75	2.6342	0.0951	165	3.3088	0.1196
82-3	2.5875	0.0935	166	2.6295	0.0946
83	2.8376	0.1026	167	3.4085	0.1232
93	3.1167	0.1124	168	3.2047	0.1157
96	2.9676	0.1071	169	3.6355	0.1313
97	2.8426	0.1027	170	4.202	0.1518
98	2.9168	0.1053	171	3.810	0.1377
99	3.186	0.1152	172	3.934	0.1420
102	2.9188	0.1050	173	4.525	0.1633
103	2.9089	0.1048	174	3.472	0.1253
106	3.6859	0.1332	175	3.189	0.1152
107	3.3799	0.1222	176	3.151	0.1137
108	3.1140	0.1133	177	4.196	0.1513
110	2.6128	0.0939	178	3.613	0.1303
113	3.5298	0.1275	179	4.331	0.1553
114	3.2237	0.1163	188	3.2548	0.1174

Table II (Cont'd)

<u>Number</u>	<u>Density gms/cm<sup>3</sup></u>	<u>0.03613 Density Pounds/in.<sup>3</sup></u>	<u>Number</u>	<u>Density gms/cm<sup>3</sup></u>	<u>0.03613 Density Pounds/in.<sup>3</sup></u>
194	4.479	0.1618	258	2.7232	0.0978
195-2	4.167	0.1503	259	2.8988	0.1043
200	3.584	0.1293	260	2.6191	0.0942
201	3.550	0.1282	261	2.5783	0.0927
202	3.769	0.1360	262	2.3180	0.0834
203	3.490	0.1259	263	4.0091	0.1447
205	4.0576	0.1463	265	3.9818	0.1439
210	3.8972	0.1405	266	3.1872	0.1153
212	3.0360	0.1095	267	2.7162	0.0976
214	2.5854	0.0930	268	3.1986	0.1155
215	3.1277	0.1138	269	3.4357	0.1242
219	2.9689	0.1072	270	3.5259	0.1275
222	4.4871	0.1620	271	3.7692	0.1360
223	5.2235	0.1887	273	2.7472	0.0988
224	5.1584	0.1860	274	2.9926	0.1077
225	4.6850	0.1690	275	3.6460	0.1318
231	3.4337	0.1240	276	3.3983	0.1227
232	3.5892	0.1297	277	3.9086	0.1413
233	3.0314	0.1093	278	2.6073	0.0942
234	3.7081	0.1338	278-1	2.6278	0.0948
235	3.3261	0.1203	278-2	2.6255	0.0947
237	3.3335	0.1203	279	2.6941	0.0972
238	3.0462	0.1098	280	2.0556	0.0742
244	3.63	0.1312	281	2.4152	0.0871
247	2.9870	0.1078	282	2.2126	0.0798
248	3.0906	0.1118	283	3.6391	0.1313
249	3.0114	0.1087	284	3.3233	0.1200
250	4.3226	0.1561	285	3.6569	0.1322
251	3.0660	0.1108	286	3.7951	0.1368
252	3.0680	0.1108	287	3.6206	0.1308
253	3.2534	0.1172	288	3.6110	0.1303
254	3.6307	0.1312	289	3.9026	0.1407
255	4.1231	0.1490	290	3.2423	0.1172
256	3.5838	0.1296	291	3.3225	0.1200
257	3.7271	0.1347	292	3.6614	0.1322

Table II (Cont'd)

<u>Number</u>	<u>Density</u> <u>gms/cm<sup>3</sup></u>	<u>0.03613</u> <u>Density</u> <u>Pounds/in.<sup>3</sup></u>	<u>Number</u>	<u>Density</u> <u>gms/cm<sup>3</sup></u>	<u>0.03613</u> <u>Density</u> <u>Pounds/in.<sup>3</sup></u>
293	3.2873	0.1187	329	3.0380	0.1095
294	3.3745	0.1217	330	4.0522	0.1462
295	3.1942	0.1152	331	3.6638	0.1322
296	3.2892	0.1187	331A	3.6293	0.1308
297	3.5426	0.1278	332	4.2390	0.1528
298	3.9706	0.1432	333	3.7066	0.1338
299	3.1904	0.1150	334	3.9453	0.1422
300	2.8883	0.1042	335	3.8562	0.1392
301	3.8131	0.1372	336	3.5098	0.1265
302	3.7684	0.1358	337	3.9452	0.1423
303	3.7256	0.1343	338	4.1815	0.1508
304	3.6248	0.1307	339	4.1550	0.1500
305	3.6629	0.1320	340	3.5589	0.1283
306	3.6654	0.1322	341	3.9190	0.1413
307	3.6950	0.1331	342	3.5616	0.1322
308	3.5651	0.1284	343	3.8365	0.1383
309	3.5951	0.1296	344	3.3901	0.1225
310	3.6864	0.1327	345	3.3434	0.1208
311	3.7008	0.1337	346	3.5527	0.1282
312	3.2789	0.1183	347	3.6345	0.1312
314	3.7178	0.1342	348	3.6505	0.1317
315	3.5831	0.1293	349	3.5261	0.1273
316	3.8017	0.1373	350	2.7817	0.1006
317	3.8051	0.1375	351	2.7814	0.1003
318	2.7173	0.0978	352	3.7924	0.1368
319	3.6270	0.1310	353	3.9158	0.1413
320	2.9286	0.1057	354	3.5718	0.1288
321	3.6319	0.1313	355	3.7860	0.1365
322	2.9967	0.1080	356	3.3507	0.1210
323A	2.7711	0.0999	357	3.9424	0.1222
324A	2.9708	0.1072	358	3.4014	0.1257
325A	3.5449	0.1280	359	3.7040	0.1337
326	3.0939	0.1115	360	3.5183	0.1269
327	3.6914	0.1330	361	3.4989	0.1260
328	4.3740	0.1578	362	3.4997	0.1260



Table II (Cont'd)

<u>Number</u>	<u>Density</u> <u>gms/cm<sup>3</sup></u>	<u>0.03613</u> <u>Density</u> <u>Pounds/in.<sup>3</sup></u>
363	3.5680	0.1286
364	3.0985	0.1117
365	3.5453	0.1277
366	3.4697	0.1253
367	3.5310	0.1273
368	3.6229	0.1307
369	3.5272	0.1273
370	3.6285	0.1307
371	3.5664	0.1282
372	4.0747	0.1470
236	3.4629	0.1249
96-2	2.9298	0.1057
SF6	5.18	0.1868
LaSF3	4.90	0.177
LaSF6	6.13	0.2207
E	2.55	0.092

Table III  
 Summary of All Values for Young's Modulus Measured on  
 Circular Rods Formed Directly from Melt

Class Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Specific Modulus 10 <sup>7</sup> inches	Class Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Specific Modulus 10 <sup>7</sup> inches	Class Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Specific Modulus 10 <sup>7</sup> inches	Class Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Specific Modulus 10 <sup>7</sup> inches
40-33	0.1067	15.5	14.5	1353	0.0946	14.3	15.1	2343	0.1338	18.1	13.5	2343	0.1338	18.1	13.5
62-33	0.0989	14.2	16.2	1363	0.1012	14.4	14.2	2353	0.1203	17.4	14.4	2353	0.1203	17.4	14.4
67-33	0.0957	14.4	15.0	1373	0.1113	13.3	12.0	2363	0.1249	17.8	14.3	2363	0.1249	17.8	14.3
68-33	0.0950	14.1	14.8	1383	0.1282	15.3	12.0	2373	0.1203	18.3	15.2	2373	0.1203	18.3	15.2
69-33	0.0935	14.2	15.2	1403	0.1329	15.6	11.7	2383	0.1098	16.6	15.1	2383	0.1098	16.6	15.1
72-23	0.1043	14.0	13.4	151	0.1175	16.9	14.4	2471	0.1078	15.1	14.0	2471	0.1078	15.1	14.0
83	0.1026	16.0	15.6	1553	0.1282	15.7	12.2	2481	0.1118	15.7	14.0	2481	0.1118	15.7	14.0
96	0.1071	16.33	15.24	1573	0.0972	13.3	13.7	2491	0.1087	15.9	14.6	2491	0.1087	15.9	14.6
971	0.1027	15.5	15.1	1593	0.1163	16.2	13.9	2501	0.1561	14.8	9.47	2501	0.1561	14.8	9.47
991	0.1152	10.5	9.12	1663	0.0946	12.5	13.2	2511	0.1108	15.9	14.3	2511	0.1108	15.9	14.3
1021	0.1050	15.0	14.3	1743	0.1253	16.5	13.2	2521	0.1108	14.9	13.5	2521	0.1108	14.9	13.5
1081	0.1133	14.8	13.1	1753	0.1152	16.1	14.0	2531	0.1172	12.3	10.5	2531	0.1172	12.3	10.5
1103	0.0939	14.6	15.5	1763	0.1137	15.2	13.4	2561	0.1296	17.9	13.8	2561	0.1296	17.9	13.8
1143	0.1163	16.7	14.4	1793	0.1553	14.9	9.6	2571	0.1347	18.4	13.7	2571	0.1347	18.4	13.7
1253	0.1000	16.1	16.1	194	0.1618	14.7	9.2	258	0.0978	13.5	13.7	258	0.0978	13.5	13.7
1263	0.1250	16.8	13.4	219	0.1072	14.8	13.8	259	0.1043	13.2	12.6	259	0.1043	13.2	12.6
1273	0.1173	16.1	13.7	222	0.1620	14.8	9.1	2633	0.1447	14.5	10.0	2633	0.1447	14.5	10.0
1293	0.1193	16.5	13.8	2313	0.1240	18.05	14.55	2651	0.1439	16.3	11.3	2651	0.1439	16.3	11.3
1313	0.1132	14.0	13.5	2323	0.1297	18.1	13.9	2661	0.1153	16.7	14.5	2661	0.1153	16.7	14.5
1343	0.1107	15.4	13.9	2333	0.1093	15.86	14.5	2671,2	0.0976	15.3	15.7	2671,2	0.0976	15.3	15.7

Table III (Cont'd)

Glass Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Spectric Modulus 10 <sup>7</sup> inches	Glass Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Spectric Modulus 10 <sup>7</sup> inches	Glass Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Spectric Modulus 10 <sup>7</sup> inches
268 <sup>1</sup>	0.1155	16.9	14.6	285 <sup>1</sup>	0.1322	15.1	11.4	310 <sup>1,2</sup>	0.1327	16.7	12.6
269 <sup>1</sup>	0.1242	17.2	13.8	286 <sup>1</sup>	0.1368	15.6	11.4	311 <sup>1,2</sup>	0.1337	15.9	11.1
270 <sup>1</sup>	0.1275	20.3	15.9	287 <sup>1</sup>	0.1308	15.1	11.5	312 <sup>1,2</sup>	0.1183	16.5	14.0
273-1 <sup>1,2</sup>	0.0988	18.4	18.6	288 <sup>1</sup>	0.1303	14.3	11.0	314 <sup>1</sup>	0.1342	17.0	12.6
273-2 <sup>1,2</sup>	0.0988	17.2	17.4	289 <sup>1</sup>	0.1407	15.0	10.7	315 <sup>1,2</sup>	0.1293	16.6	12.8
274 <sup>1,2</sup>	0.1077	17.2	15.9	290 <sup>1</sup>	0.1172	14.5	12.3	316 <sup>1,2</sup>	0.1373	16.5	12.0
275-1 <sup>1,2</sup>	0.1318	16.7	12.7	291 <sup>1</sup>	0.1200	15.7	13.1	317 <sup>1,2</sup>	0.1375	16.3	11.9
275-2 <sup>1,2</sup>	0.1318	16.8	12.7	292 <sup>1</sup>	0.1322	15.4	11.6	318 <sup>1,2</sup>	0.0978	16.0	16.4
276 <sup>1,2</sup>	0.1227	15.8	12.9	293 <sup>1</sup>	0.1187	16.0	13.5	319	0.1310	17.9	13.7
277 <sup>1,2</sup>	0.1413	17.9	12.6	294 <sup>1</sup>	0.1217	17.6	14.4	320	0.1057	16.0	15.1
278 <sup>4</sup>	0.0942	13.3	13.6	295 <sup>1</sup>	0.1152	15.2	13.2	321 <sup>3</sup>	0.1313	18.7	14.2
278 <sup>4</sup>	0.0942	15.2	16.1	296 <sup>1</sup>	0.1187	16.5	13.9	322 <sup>1,2</sup>	0.1080	16.9	15.65
279	0.0972	12.4	12.7	297 <sup>1</sup>	0.1278	17.1	13.4	323 <sup>1,2</sup>	0.0999	18.4	18.4
280	0.0742	5.56	7.5	299 <sup>1</sup>	0.1150	14.6	12.7	324 <sup>1,2</sup>	0.1072	17.8	16.6
280 <sup>4</sup>	0.0142	6.23	8.40	300 <sup>1</sup>	0.1042	14.5	13.9	325 <sup>1,2</sup>	0.1280	20.2	15.8
281	0.0871	6.77	7.77	302 <sup>1</sup>	0.1358	17.2	12.6	326 <sup>1,2</sup>	0.1115	17.0	15.3
282 <sup>4</sup>	0.0798	4.80	6.00	304 <sup>3</sup>	0.1307	19.2	14.7	327 <sup>1</sup>	0.1330	18.4	13.8
282 <sup>4</sup>	0.0798	6.22	7.80	305 <sup>3</sup>	0.1320	17.7	13.4	328 <sup>1</sup>	0.1578	18.5	11.7
283 <sup>1</sup>	0.1313	15.5	11.8	306 <sup>3</sup>	0.1322	18.9	14.3	329 <sup>1</sup>	0.1095	20.7	18.9
284 <sup>1</sup>	0.1200	14.9	12.4	309 <sup>1,2</sup>	0.1296	16.9	13.0	330 <sup>1,2</sup>	0.1462	18.6	12.7

Table III (Cont'd)

Glass Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Spectric Modulus 10 <sup>7</sup> inches	Glass Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Spectric Modulus 10 <sup>7</sup> inches	Glass Number	Density lbs/in <sup>3</sup>	Young's Modulus millions psi	Spectric Modulus 10 <sup>7</sup> inches
331 <sup>1,2</sup>	0.1322	20.9	15.8	351 <sup>1,2</sup>	0.1003	17.0	17.0	X-2285	0.0899	14.95	16.65
332 <sup>1</sup>	0.1528	17.3	11.3	352 <sup>1,2</sup>	0.1368	20.0	14.6	SiO <sub>2</sub>	0.079	10.5	13.3
333 <sup>1</sup>	0.1338	18.9	14.1	353 <sup>1,2</sup>	0.1413	19.4	13.7	"F" <sup>2</sup>	0.092	10.5	11.4
334 <sup>1</sup>	0.1422	17.5	12.3	354 <sup>1,2</sup>	0.1288	18.4	14.3	"S"	0.090	12.6	14.0
335 <sup>1,2</sup>	0.1392	19.0	13.6	355 <sup>3</sup>	0.1365	18.1	13.2				
336 <sup>1,2</sup>	0.1265	21.0	16.6	356 <sup>3</sup>	0.1210	13.9	11.5	Steel	0.280	29	10.3
337 <sup>1</sup>	0.1423	20.9	14.7	357 <sup>3</sup>	0.1222	17.6	14.4	Al <sub>2</sub> O <sub>3</sub>	0.114	25	21.9
338 <sup>1</sup>	0.1508	----	----	358 <sup>3</sup>	0.1257	13.1	10.4	ZrO <sub>2</sub>	0.175	50	28.6
339 <sup>1,2</sup>	0.1500	19.4	13.0	359 <sup>3</sup>	0.1337	17.6	13.2	BW <sup>2</sup>	0.069	13	18.8
340 <sup>1,2</sup>	0.1283	20.9	16.3	360 <sup>3</sup>	0.1269	18.5	14.6	MO	0.369	52	14.1
341 <sup>1,2</sup>	0.1413	18.9	13.4	361 <sup>3</sup>	0.1260	18.6	14.8				
342 <sup>1,2</sup>	0.1322	----	----	362 <sup>3</sup>	0.1260	18.0	14.3				
343 <sup>1,2</sup>	0.1383	19.4	14.0	96-2	0.1057	15.4	14.6				
344 <sup>2,3</sup>	0.1225	20.3	16.8	331A <sup>1,2</sup>	0.1310	20.07	15.35				
345 <sup>2,3</sup>	0.1208	21.1	17.45								
346 <sup>1,2</sup>	0.1282	17.66	13.8	SFS1	----	6.98	----				
347 <sup>1,2</sup>	0.1312	21.6	16.4	SF6	0.1868	6.91	3.69				
348 <sup>1,2</sup>	0.1317	17.7	13.4	LaSF3	0.177	13.3	7.52				
349 <sup>1,2</sup>	0.1273	16.8	13.2	LaSF6	0.2207	17.4	7.88				
350 <sup>1,2</sup>	0.1006	19.8	19.7								

1 Invert Analog Glasses  
 2 Contains BeO  
 3 Cordierite Base Glasses  
 4 Heat treated two phase

thermal expansion at least slightly higher than that of fused silica results in the shrinking of the aspirated bar away from the fused silica tube on cooling and allows the ready removal of the sample. Since the rods drawn in this manner represent a glass of higher fictive temperature than the former procedure employed at UARL when the samples were prepared by precision machining from massive cast and annealed slabs, the results are not directly comparable with the earlier results reported for bulk samples but more nearly approach the values found with fibers pulled from the same melt.

The results for all the experimental glasses for which Young's modulus was measured on aspirated rods drawn directly from the melt are shown in Table III. The story represented by the values in this table is most readily understood if the tabular values are examined chronologically, i.e. in the order of increasing glass number. For example, no glasses are found in the first column which have a Young's modulus as high as 18.2 million pounds per square inch but the second column shows three glasses with Young's modulus of 18.4 million pounds per square inch and one with a value of 20.3 million psi. The third column shows seven glasses with values for Young's modulus between 18.3 and 19.9 million psi and three glasses having moduli greater than twenty million psi. The fourth column has ten glasses with moduli between 18.3 and 19.9 million psi and five glasses with moduli between twenty and twenty-one million psi and two glasses with moduli greater than twenty-one million psi. Each column represents approximately ten months research so that the accelerating pace of the research program is readily apparent. This table also gives the density in English units and the specific moduli in units of ten million inches. From consideration of both Young's modulus and specific modulus, glass 350 with Young's modulus of 19.8 million psi and specific modulus of 197 million inches and glass 329 with corresponding numbers of 20.7 million psi and 189 million inches are the best glasses produced to date and compare very favorably with the best glasses reported from other companies such as Owens-Corning Fiberglas Corp. pilot plant glass X-2285 of 14.95 million psi and 166.5 million inches for Young's modulus and specific modulus respectively.

#### Reproducibility of Values of Young's Modulus Obtained with Rods Formed by Aspiration

The value shown in Table III for the Young's modulus of the various experimental glasses is in general the result of averaging measurements for five different rods formed by aspiration from a melt of the glass in question. In Table IV, instead of just the average, all the results obtained for Young's modulus measurements for glass rods pulled from the melt are shown. It will be evident that, in general, the method gives excellent reproducibility. The obvious exceptions to this generalization found in the table arise from cases where the glass devitrifies so rapidly that the extent of crystallization varies from sample to sample and causes discordant results.

Table IV

Reproducibility of Values of Young's Modulus Measured  
on Rods Aspirated Directly from Melt

<u>Glass No.</u>	<u>Individual Determinations</u> (millions psi)	<u>Average</u> <u>Modulus</u> (millions psi)
40-3	15.1, 15.6, 15.6, 15.5	15.5
62-3	13.59, 14.77	14.18
67-3	14.47, 14.59, 14.24	14.43
68-3	14.4, 13.8, 13.7, 14.6	14.1
69-3	14.11, 14.37, 14.00, 13.49	14.00
72-2	14.4, 13.9, 13.8	14.0
83	16.0, 16.0, 16.0, 16.0	16.0
96	16.51, 16.31, 15.76, 15.73, 16.00, 15.92, 16.17, 15.90, 16.38, 15.93, 18.76, 16.18, 15.70, 15.67 16.94, 16.38	16.33
99	10.7, 10.5, 10.4, 10.5	10.5
102	15.00, 14.97, 14.61, 15.13, 15.50	15.04
108	14.79, 14.67, 14.84, 14.68, 14.69, 15.16	14.81
110	14.69, 14.07, 14.73, 14.99	14.62
114	16.4, 16.7, 16.8, 16.7	16.7
125	16.2, 15.8, 16.2, 16.2, 16.3	16.14
126	16.5, 17.1, 16.7	16.8
127	15.49, 16.10, 16.51, 16.06, 15.81, 16.75	16.13
129	16.9, 16.1, 16.3, 16.6	16.5
131	13.99, 13.66, 13.93, 13.80, 14.61	14.00
134	15.3, 15.4	15.4
135	14.6, 14.5, 14.6, 14.0, 14.6, 14.3, 14.2	14.3
136	14.6, 14.2, 14.4	14.4
137	13.3, 13.6, 13.3, 13.0	13.3
138	15.3, 15.2, 15.4, 15.3	15.3
140	15.1, 15.7, 15.5, 16.2	15.6
155	15.6, 15.7, 15.7, 15.7	15.7
157	13.3, 13.6, 13.3, 13.0	13.3
159	16.5, 15.9, 16.0, 16.4	16.2
166	12.9, 12.1, 12.6	12.53
179	14.6, 14.8, 14.6, 14.9	14.7
194	14.0, 14.9, 14.6, 15.2	14.7

Table IV (Cont'd)

<u>Class No.</u>	<u>Individual Determinations (millions psi)</u>	<u>Average Modulus (millions psi)</u>
219	14.87, 14.65, 14.87	14.80
222	15.3, 15.0, 14.2	14.8
231	18.63, 18.04, 17.99, 17.41, 17.25, 18.35, 17.62, 19.08	18.05
232	20.11, 17.10, 17.46, 17.87, 18.09	18.13
233	17.57, 15.38, 15.15, 15.27, 17.58	16.21
234	17.95, 17.87, 18.14, 18.22, 17.95, 18.16	18.05
237	18.00, 17.99, 18.43, 18.47, 18.61, 18.36, 16.93	18.16
247	15.0, 15.2, 15.0	15.07
248	20.5, 16.0, 15.42, 14.03	16.48
249	15.9, 15.6, 16.0, 16.0, 15.8	15.86
250	14.35, 14.86, 15.27, 14.82	14.83
251	16.08, 15.48, 16.05, 15.82	15.86
252	14.65, 15.32, 14.63	14.87
256-2	17.35, 17.68, 15.95, 17.70, 18.30, 17.92, 17.78, 17.40, 18.05	17.88
257-2	17.85, 18.65, 18.28, 17.43, 17.95, 18.28, 17.88, 18.97, 18.47, 16.82	18.35
258	13.5	13.5
259	13.08, 13.21, 14.0, 12.67	13.24
263	14.85, 14.22, 15.08, 14.30, 14.00	14.49
265	16.58, 16.68, 16.17, 15.67	16.28
266	16.62, 16.97, 17.12, 16.22	16.73
267	14.62, 15.70, 15.40, 15.6	15.34
268	17.00, 17.03, 16.75	16.93
269	17.8, 17.1, 16.95, 16.85	17.18
270-2	20.2, 20.8, 19.9, 20.1	20.25
273-1	17.90, 18.28, 18.97	18.39
273-2	16.57, 17.73, 17.88, 17.80, 17.35, 17.4	17.51
274-2	15.48, 17.47, 18.42, 16.70, 18.32, 18.25, 17.75, 17.57, 21.2, 17.3	17.23
275-1	16.48, 16.64, 16.52, 16.65, 17.21, 16.54	16.67
275-2	16.65, 17.21, 16.54	16.80
276	15.47, 15.0, 15.82, 16.48, 16.32, 15.82	15.82
277-2	17.43, 17.43, 17.55, 18.32, 18.66, 17.93, 17.57, 17.80, 17.43	17.91
278	13.12, 13.41, 13.01, 13.36, 12.77, 13.90	13.3

Table IV (Cont'd)

<u>Glass No.</u>	<u>Individual Determinations</u> (millions psi)	<u>Average Modulus</u> (millions psi)
278 H.T.	14.93, 14.44, 14.76, 15.47	15.2
279	11.93, 11.55, 12.41	11.96
280	5.47, 5.62, 5.89, 5.70, 5.25, 5.40	5.56
280 H.T.	6.186, 6.030, 6.312, 6.115, 6.215, 6.258	6.23
281	6.23, 6.40, 6.36, 6.45, 6.18, 5.37, 6.41, 6.19	6.20
282	4.25, 4.54, 5.45	4.80
282 H.T.	6.22	6.22
283	15.58, 15.36, 15.20, 15.62, 15.39, 15.67	15.47
284	14.08, 14.87, 15.33, 15.07, 14.87	14.85
285	15.13, 15.31, 15.0, 15.17, 14.91, 14.89	15.07
286	16.86, 15.48, 14.81, 15.64, 15.49, 15.28, 16.46	15.60
287	14.53, 15.50, 14.79, 14.65, 15.26, 15.63	15.14
288	14.27, 14.48, 14.14, 13.99, 14.49, 14.42	14.30
289	14.74, 14.75, 15.19, 15.21, 14.86	14.95
290	14.47, 14.27, 14.72, 14.26, 14.51, 14.89	14.52
291	16.12, 15.32, 15.96, 15.67, 15.59, 15.37	15.67
292	15.46, 15.08, 15.73, 15.17, 14.78, 15.77	15.36
293	16.28, 16.15, 17.18, 14.93, 15.30, 15.86, 16.04	15.96
294	17.71, 17.70, 17.41, 17.37, 17.89	17.62
295	15.29, 15.14, 15.47, 14.81, 15.28, 15.60, 15.02	15.22
296	16.709, 15.12, 18.51, 15.49, 16.47, 16.87	16.53
297	17.174, 17.998, 18.082, 16.996, 16.483, 16.749	17.08
299	14.74, 14.35, 14.60, 14.63, 14.84, 14.28	14.57
300	14.36, 14.40, 14.58, 14.58, 14.47, 14.22, 14.68	14.45
302	17.23, 16.73, 17.58, 17.24, 17.04	17.16
304	24.06, 18.53, 18.45, 18.30, 19.12, 18.21, 17.91	19.23
305	17.11, 18.10, 17.72, 17.79, 17.88, 16.76	17.72
306	18.67, 18.89, 18.63, 18.72, 19.44, 18.72	18.85
309	16.92, 16.43, 16.91, 18.12, 16.59, 16.21	16.86
310	16.04, 19.58, 16.59, 15.88, 16.20, 15.65	16.66
311	16.31, 14.71, 14.98, 15.98, 16.49, 16.91	15.90
312	17.26, 15.81, 16.77, 16.25	16.52
314	16.39, 17.23, 16.68, 17.07, 17.19, 17.47, 16.89	16.99
315	16.89, 17.04, 16.17, 16.97, 16.14	16.63
316	16.55, 16.57, 16.18, 16.37, 16.52, 16.26, 16.39	16.46
317	15.84, 16.32, 16.87, 16.78, 16.64, 15.53	16.33



Table IV (Cont'd)

<u>Glass No.</u>	<u>Individual Determinations</u> ( <u>millions psi</u> )	<u>Average</u> <u>Modulus</u> ( <u>millions psi</u> )
318	15.26, 15.68, 18.08, 15.91, 15.57, 15.44	15.97
319A	17.9, 18.02, 17.83, 16.60, 16.16, 16.48	17.17
319	15.96, 15.03, 15.86, 15.26, 15.01	15.42
320A	16.05, 15.98, 15.90, 16.12, 15.72, 16.00	15.96
320B	17.23, 19.33, 16.32, 16.31, 16.23, 16.79	17.03
321A	18.20, 17.92, 19.83, 18.09, 18.97, 19.07	18.7
322	17.56, 16.85, 17.18, 17.11, 16.71	17.08
322A	18.52, 18.11, 18.63, 18.72, 19.76, 18.87	18.60
323A	18.57, 18.14, 17.92, 20.05, 18.05, 17.79	18.42
324A	18.04, 17.99, 17.41, 17.25, 18.35, 17.62	17.78
325A	18.92, 19.75, 20.16, 21.17, 20.59, 20.55	20.19
325	19.82, 19.09, 19.05, 18.97, 19.87, 20.12	19.49
326A	16.53, 17.28, 17.05, 17.23, 16.79	16.98
326	16.89, 16.68, 17.83, 15.62	16.76
327A	18.69, 17.99, 17.88, 18.34, 18.88	18.36
328A	18.04, 19.11, 18.21	18.45
329A	20.68, 21.07, 22.91, 19.13	20.92
330	18.96, 18.73, 18.90, 19.13, 17.67, 18.38	18.63
331	20.79, 20.99, 21.02, 20.12, 20.97, 21.21	20.85
332	17.68, 16.99	17.34
333	18.79, 18.94, 18.90	18.88
334	17.25, 17.54, 17.22, 17.48, 17.42, 17.94	17.48
335	19.25, 18.88, 19.08, 18.43, 19.26	19.00
336	21.5, 21.13, 20.10, 20.97, 21.06, 20.92	20.95
337	19.58, 22.47, 21.81, 20.37, 20.32	20.92
339	19.4, 19.2, 19.1, 19.7	19.4
340	20.24, 20.50, 21.72	20.83
341	20.13, 19.83, 17.92, 18.31, 19.35, 19.4, 17.91, 18.53, 18.32, 18.28, 18.84	18.80
343	19.55, 19.45, 19.11, 19.4	19.38
344	20.27, 20.29, 20.06	20.3
345	21.6, 20.1, 21.1, 20.87, 20.79, 21.56	21.00
346	17.9, 17.6, 17.8, 17.5, 17.5	17.66
347	21.28, 22.14	21.62

Table IV (Cont'd)

<u>Glass No.</u>	<u>Individual Determinations (millions psi)</u>	<u>Average Modulus (millions psi)</u>
348	18.80, 17.50, 17.25, 18.29	17.71
349	16.73, 16.80	16.8
350	20.04, 19.32, 19.94, 19.71	19.75
351	16.59, 17.08, 17.24, 17.26, 17.17, 16.39	16.96
352	20.40, 19.60, 19.11, 20.90	20.0
353	19.86, 19.26, 19.52, 18.89	19.4
354	18.47, 18.36, 18.13, 18.44	18.4
355	18.11, 17.81, 18.50	18.1
356	13.01, 14.81	13.9
357	17.11, 17.11, 18.51	17.6
358	12.54, 12.07, 14.66	13.1
359	18.76, 17.40, 17.55, 16.72	17.6
360	18.36, 17.99, 19.18	18.5
361	18.15, 18.38, 17.72, 20.23	18.6
362	17.92, 18.06, 17.85, 18.23	18.0
363	19.74, 19.45, 18.78	19.26
364	16.97, 16.36, 16.77	16.70
365	19.04, 19.03	19.04
366A	16.77, 15.61	16.19
367	18.88, 19.03, 19.18	19.03
368	19.30, 19.00, 18.93	19.08
369	18.07, 17.74, 17.52	17.78
370	18.42, 18.56, 18.80	18.59
371	18.82, 18.09, 18.70	18.59
372	17.38, 17.28, 20.68	18.54
E	12.42, 12.64, 12.14, 12.06, 11.98, 11.93	12.2
LaSF6	17.28, 17.23, 17.63, 17.55	17.42
LaSF3	13.08, 13.55, 13.72, 13.01	13.34
SFS1	7.18, 6.74, 7.06, 6.92	6.98
SF6	7.34, 6.90, 6.58, 6.82	6.91
76	15.7	15.7
93	16.06, 15.80, 15.70, 15.64, 15.66, 15.90, 15.80, 15.82, 15.69, 15.67, 15.79, 15.45, 15.50	15.732
96-2	15.59, 15.28	15.4
97	15.52	15.5

Table IV (Cont'd)

<u>Glass No.</u>	<u>Individual Determinations</u> <u>(millions psi)</u>	<u>Average</u> <u>Modulus</u> <u>(millions psi)</u>
174	16.9, 16.4, 16.4, 16.1	16.5
175	15.9, 16.3, 16.2, 15.3	16.1
176	14.9, 16.2, 14.6, 15.0	15.2
233A	17.64, 15.15, 15.38, 15.27	15.86
235	17.5, 17.2, 17.4, 17.4, 17.4	17.4
236	18.0, 17.8, 17.4, 18.1	17.8
238	16.8, 16.8, 16.8, 16.4	16.6
331A	19.7, 19.9, 19.60, 20.53, 20.33, 20.35	20.07

## FIBERIZABILITY STUDIES

Young's Modulus for Mechanically Drawn Experimental  
Glass Fibers Evaluated by Mechanical Tests

As described in our earlier reports F910373-7, F910373-8, F910373-9, G910373-10, and G910373-11, UARL prepares mechanically drawn fibers by the use of a "poor man's bushing". The bushing consists of a 20 cm<sup>3</sup> platinum crucible with reinforced bottom and central hole which is formed by starting with a normal platinum crucible and welding several thicknesses of platinum foil to the bottom until a bottom thickness of 3/16 in. is obtained and then taper reaming a central orifice 0.088 in. at top, 0.063 in. at bottom and 3/16 in. long in bottom of crucible. The crucible is then filled with glass and introduced into the platform furnace with high temperature Super-kanthal hairpin heating elements (furnace temperatures in excess of 1700°C are obtainable) together with a ring orifice providing water cooling immediately below the crucible as well as a second ring orifice for cooling the fiber as it forms with helium jets. The equipment has now been extensively used and has proven satisfactory for the production of very nearly circular glass fiber having approximately one mil diameter at pulling rates of 4000 to 8000 ft/min.

Experimental glass fibers prepared as described above are evaluated for UARL by the Lowell Institute of Technology. Lowell Institute of Technology uses an Instron CRE tester operated with a machine speed of 0.2 in. per minute, a chart speed of 20 in. per minute, a gage length of 5 in., and a full scale capacity of 1.0 lb. The specimens were held in air actuated clamps with flat rubber coated faces.

For each fiber for which we give results in the table, a minimum of 20 specimens were taken from approximately the center portion of each spool. The specimens were 8 in. long with about one yard of fiber being discarded between each specimen. It was not always possible to select fibers in exactly this manner because many of the spools had discontinuous odd lengths of fiber, but in general, the specimens selected represent the middle 20 yds of the fiber supplied for testing. Three fiber diameter measurements were made in the middle three-inch portion of each eight-inch specimen. These measurements were made using a monocular microscope equipped with an eyepiece reticule and operated with a magnification of 774 (18x eyepiece, 43x objective). Each reticule division was equal to 0.092 mils.

The average of twenty determinations for each fiber for the cordierite rare-earth fibers is shown in Table V. As shown in our earlier report F910373-8, this data has a standard deviation of 1.82 million psi for example, so that for glass 126 one can say with 99% probability that the tabulated value at 16.4 million psi actually lies between 15.2 and 17.6 million psi. Comparative data is again supplied for Owens-Corning "E" glass, Tiede's glass 83 (example 4 of his U.S. patent 3,122,277) and glass made according to the teachings of the Houze Glass Company patent. It is immediately obvious that the outstanding glass fibers prepared to date are from UARL experimental glasses 320B3 and 331.

Table V

Values for Young's Modulus on Mechanically Drawn Fibers  
of UARL Experimental Glasses as Determined by  
Measurements on Tensile Test Equipment

<u>Glass No.</u>	<u>Measurements</u>		<u>Glass No.</u>	<u>Measurements</u>	
	<u>Young's Modulus 10<sup>6</sup> psi</u>	<u>Std. Dev. 10<sup>6</sup> psi</u>		<u>Young's Modulus 10<sup>6</sup> psi</u>	<u>Std. Dev. 10<sup>6</sup> psi</u>
25	12.3	1.9	131	12.5	---
40	16.2	4.6	135	13.3	---
56	10.7	---	136	13.5	---
62	14.0	---	137	13.9	---
63	13.0	---	138	12.2	---
64	14.7	---	140	15.0	---
65	13.8	---	155	14.7	1.9
66	14.6	2.0	157	13.1	2.8
67	12.7	---	159	15.7	5.0,2.2
68	13.7	---	160	14.6	3.0
69	13.6	---	161	14.3	1.8
70	13.4	---	166	13.6	5.4
71	13.7	---	175	13.1	2.9
72	12.5	---	176	16.7	9.9
73	15.1	---	193	13.1	4.0
74	13.8	---	200	14.6	3.3
75	9.8	---	201	13.2	4.3
76	10.4	---	210	15.0	2.0
77	11.0	---	214	12.9	1.6
82*	13.3	---	215	13.3	3.0
83*	15.35	2.4,1.95	233	14.6	2.3
97	10.4	1.7	235	15.3	1.9
98	10.8	2.5	237	18.8	7.9
102	13.3	---	238	15.2	3.2
108	12.5	2.7	260	10.4	1.5
110	13.8	2.2	261	11.5	1.8
114	15.1	---	275-10	15.93	2.2
126	16.15	1.82,1.65	276	17.0	6.2
127	15.2	---	278	11.3	1.8
129	16.7	2.7,2.6	279	6.2	3.3

Table V (Cont'd)

<u>Glass No.</u>	<u>Measurements</u>	
	<u>Young's Modulus 10<sup>6</sup> psi</u>	<u>Std. Dev. 10<sup>6</sup> psi</u>
280	4.6	0.4
281	5.7	0.5
284	11.9	2.4
285	12.9	4.7
288-3	11.9	2.8
289	13.1	1.5
290-5	14.3	5.8
290-6	13.0	3.7
291	13.6	3.2
299	12.8	3.6
300	13.4	3.4
309	14.8	1.9
320B3	18.6	3.3
321A	17.4	3.6
331	18.3	2.5
E	11.8	3.9

## Panametrics Ultrasonic Check Measurements

233	14.9
275-10	15.94
320B	16.2
321A	18.2
331	19.8

82\* Houze Glass - U.S. 3,044,888

83\* Owens-Corning - U.S. 3,122,277 (#4)

All tabulated observations are the average of 20 observations except that the results for 40, 83, 126, 129, 233, 275-10, 320B, and 331 represent 60 observations

The best value for UARL 331 is probably the 19.8 million psi for Young's modulus measured by Panametrics using an ultrasonic technique. Experimental glass 237 although also showing a high value for Young's modulus has an unusually high standard deviation and the results are, therefore, not conclusive. Unfortunately, there is a very considerable time lag between the preparation of a glass fiber at UARL and its final evaluation at Lowell Institute of Technology so that no results are yet available for our more recent experimental glasses.

OTHER RESEARCH UNDER WAY AT UARL NOT SPECIFICALLY  
COVERED IN THIS REPORT

Research in several other areas intimately connected with the research described in the previous sections is under way at UARL at the present time but progress is as yet insufficient as to warrant separate report sections for these studies.

1. The UARL central computation laboratory is examining the results obtained to date for each glass composition for which we have modulus, density, and liquidus data to discern any trend. This type of analysis should be especially valuable with the invert analog glasses which possess six to eight or more components. They are employing the single factor method for determining an optimal response. To date UARL glasses 350 and 351 are the first glasses made from the recommendations of the computation laboratory.

2. Another hundred strength measurements made under the most carefully controlled circumstances using carefully cleaned and degreased glass slugs of remelted glass but still using the "poor man's bushing" described in our earlier report F910373-7 shows that it is almost impossible to obtain reproducible values for the strength of the glass fibers produced in this apparatus. Reproducible strength measurements are dependent on the installation of more standard type bushings.

3. The platinum-rhodium bushings (one hole) whose design was described in our last report H910373-13 have now been delivered and their installation is under way. The first power source, a stacked Variac welding transformer arrangement, with which we attempted to match the impedance of these bushings was able to heat the bushing to only 800°C at the output of 2800 amperes and 0.35 volts which it produced. A second power source has now been installed and trials are under way.

4. Additional two-phase glasses have been prepared but not evaluated or fiberized.

5. Microfurnace liquidus determinations continue and this work has been broadened to include not only the determination of the liquidus and working range for a given original glass but also for the same glass with three different amounts of three different additives. These additives are selected to increase the viscosity and working range of some of the glasses which had such a short working range that normal fiber production was impossible.

6. A modified, strictly nonstandard Charpy test has been set up to evaluate the relative impact strength of bulk samples of the experimental glasses.

7. A model was prepared for the use of NASA officials in their Congressional presentation.

ABSTRACT OF AN ARTICLE SUBMITTED FOR POSSIBLE PRESENTATION AT  
TORONTO MEETING OF INTERNATIONAL COMMISSION ON GLASS

We have added below the title and abstract of an article submitted for possible presentation at the jointly sponsored JUPAC meeting (International Commission on Glass & Canadian Ceramic Society) to be held in Toronto, Canada in early September.

Studies of the Young's Modulus of Magnesia-Alumina-Silica-  
Rare Earth Glass Systems with Respect to their  
Composition and Crystallization Kinetics

Glasses prepared from mixtures of magnesia-alumina-silica in such proportions that their primary crystal phase on devitrification is either sapphirine or cordierite, and to which either a rare earth oxide or zirconia have been added have been found to have unusually high values of Young's modulus. After preparation these glasses are characterized by measurement of their density, modulus, and fiberizability. Examination of the results obtained showed that the moduli of these glasses can be successfully calculated by the methods of C. J. Phillips (Ref. 1) and that the molar coefficients for the rare-earths ceria, lanthana, and yttria as well as for zirconia are as large or larger than the corrected Phillips' molar coefficient for beryllia.

Direct microscopic observations of the kinetics of crystallization of the  $MgO-Al_2O_3-SiO_2$ -rare earth systems showed that the rare-earths added to increase Young's modulus also had the beneficial result of actively delaying the onset of devitrification.

The techniques used in preparing these high melting glass compositions, in determining their fiberizability, in studying their crystallization rates, and in preparing and evaluating the samples used for modulus measurement are sketched briefly. (The results of the several experiments are tabulated.) These results show that the most favorable non-toxic composition found had a value for Young's modulus of slightly more than 20 million pounds per square inch while cordierite base compositions to which beryllia was intentionally added yielded glasses with Young's modulus as high as twenty-two million pounds per square inch.

Ref. 1 Phillips, C. J., Calculation of Young's Modulus of Elasticity from Composition of Simple and Complex Silicate Glasses. Glass Technology, Vol. 5, No. 6, Dec. 1964, pp 216-223.



## CONCLUSIONS

1. Both the UARL "invert" analog glass systems and the cordierite-rare earth (or zirconia) glass systems are capable of producing glasses with Young's moduli over twenty million psi and specific moduli of nearly twenty million.
2. The principal problem remaining for the successful production of glass fibers with moduli and specific moduli four-thirds as high as any glass now known lies in the adjustment of the composition to give increased working range without lowering the presently achieved values.
3. No evidence is available that suggests that higher modulus and specific modulus values will not be obtainable from further research. The two-phase glass compositions seem to present especially favorable opportunities to achieve higher moduli.

## PERSONNEL ACTIVE ON PROGRAM

Personnel active on the program throughout the fourteenth period have been J. F. Bacon, Principal Investigator, and Michael DiPerno and Francis Hale, Experimental Technicians. Liaison in this quarter and throughout the program has been carried out by Peter A. Stranges of the UARL Washington office. Throughout this quarter and the whole program the UARL personnel have reported progress to and received advice from James Gangler of NASA Washington Headquarters and Michael DeCrescente of UARL.

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