

UNCLASSIFIED

|                                                                                                                                                                                                                                                                       |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| AD NUMBER                                                                                                                                                                                                                                                             |
| <b>AD800533</b>                                                                                                                                                                                                                                                       |
| LIMITATION CHANGES                                                                                                                                                                                                                                                    |
| TO:<br><b>Approved for public release; distribution is unlimited. Document partially illegible.</b>                                                                                                                                                                   |
| FROM:<br><b>Distribution authorized to U.S. Gov't. agencies and their contractors;<br/>Administrative/Operational Use; JUN 1966. Other requests shall be referred to Air Force Material Laboratory, Wright-Patterson AFB, OH 45433. Document partially illegible.</b> |
| AUTHORITY                                                                                                                                                                                                                                                             |
| <b>afml ltr, 7 dec 1972</b>                                                                                                                                                                                                                                           |

THIS PAGE IS UNCLASSIFIED

AFML-TR-65-90  
Part II

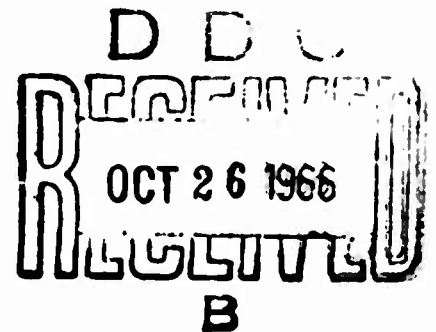
HIGH STRENGTH - HIGH MODULUS GLASS FIBERS

R. M. McMarlin, R. L. Tiede, F. M. Veazie  
OWENS-CORNING FIBERGLAS CORPORATION

TECHNICAL REPORT AFML-TR-65-90, Part II

July 1966

Qualified requesters may obtain copies of this report from the Defense Documentation Center (DDC) (formerly ASTIA) Cameron Station, Building 5, 5010 Duke Street, Alexandria 4, Virginia. Dissemination outside the Department of Defense or to recipients other than Government defense contractors is prohibited. DDC release to CFSTI not authorized.



Air Force Materials Laboratory  
Research and Technology Division  
Air Force Systems Command  
Wright-Patterson Air Force Base, Ohio

800533

2

**Best  
Available  
Copy**

## NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

Copies of this report should not be returned to the Research and Technology Division unless return is required by security considerations, contractual obligations, or notice on a specific document.

## **HIGH STRENGTH - HIGH MODULUS GLASS FIBERS**

**R. M. McMarlin, R. L. Tiede, P. M. Veazie**

Qualified requesters may obtain copies of this report from the Defense Documentation Center (DDC) (formerly ASTIA) Cameron Station, Building 5, 5010 Duke Street, Alexandria 4, Virginia. Dissemination outside the Department of Defense or to recipients other than Government defense contractors is prohibited. DDC release to CFSTI not authorized.

## FOREWORD

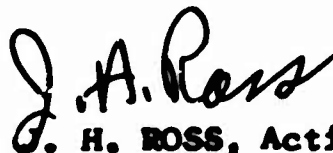
This report was prepared by the Glass Research Laboratory of Owens-Corning Fiberglas Corporation, Granville, Ohio under USAF Contract AF 33(615)-1370. This contract was initiated under Project No. 7320, "Fibrous Materials for Decelerators and Structures," Task No. 732001, "Nonmetallic and Composite Materials" and Project No. 7340, "Organic and Inorganic Fibers," Task No. 734003, "Structural Plastic and Composite Materials." This report covers the work accomplished during the period March 1965 through February 1966.

The contract was initiated by the Nonmetallic Materials Division of the Air Force Materials Laboratory with Walter H. Gloor (MANF) of the Fibrous Materials Branch as Project Engineer.

The work was performed under the technical direction of Mr. F. Munro Veazie, as Program Manager, with Mr. Robert McMarlin and Mr. Ralph Tiede as Project Managers. They were assisted by Messrs. Austin Walpole, Glen Lucase, John Brookbank and Don Coburn.

Manuscript released by authors for publication as an RTD Technical Report July 5, 1966.

This technical report has been reviewed and is approved.



J. H. ROSS, Acting Chief  
Fibrous Materials Branch  
Nonmetallic Materials Division

## ABSTRACT

The purpose of this investigation has been the study of glass compositions capable of producing glass fibers having high tensile strength - high modulus of elasticity properties which are also low in density. Of the many compositions studied, those which have the best promise of meeting the required properties are located in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$  system. In particular, compositions have been developed in this system with tensile strengths of 680-715,000 psi, moduli of  $14\text{-}15 \times 10^6$  psi, and bulk densities of 2.41-2.50. These properties produce fibers with specific strengths and specific moduli in excess of any currently available glass fiber. Successful multifilament manufacture of glasses of this nature was demonstrated and unidirectional filament wound composites were fabricated and tested. In a basic study aimed at relating glass properties, composition, and structure, three and four component systems were studied consisting of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  plus one of the following:  $\text{K}_2\text{O}$ ,  $\text{Li}_2\text{O}$ ,  $\text{K}_2\text{O} + \text{Li}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{ZnO}$  and  $\text{BeO}$ . For glasses containing  $\text{K}_2\text{O}$ ,  $\text{Li}_2\text{O}$ ,  $\text{K}_2\text{O} + \text{Li}_2\text{O}$ , or  $\text{CaO}$ , curves of modulus vs. the ratio of aluminum to added ion showed breaks at the point where one oxygen ion had been added per two aluminum ions. This was related to a proposed glass structure involving a change of coordination number of aluminum from four to six, with a corresponding change in the function of this ion in the structure.  $\text{BeO}$ ,  $\text{MgO}$  and  $\text{ZnO}$  were similar to each other in behavior, but differed from the other oxides. They showed breaks in composition vs. modulus curves at different points than the other oxides, with the inflection point changing with  $\text{SiO}_2$  content. These oxides increased modulus in the following order ( $\text{BeO}$  highest):  $\text{BeO}$ ,  $\text{MgO}$ ,  $\text{ZnO}$ ,  $\text{CaO}$ ,  $\text{Li}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ .

## TABLE OF CONTENTS

|                                                                                         | <u>Page</u> |
|-----------------------------------------------------------------------------------------|-------------|
| <b>Section I</b>                                                                        |             |
| Introduction . . . . .                                                                  | 1           |
| <b>Section II</b> . . . . .                                                             | 1           |
| <b>Study of High Tensile Strength-High Modulus Glass Fiber</b>                          |             |
| <b>Compositions</b> . . . . .                                                           | 1           |
| 1. Glass Preparation, Fiberization, and Testing. . . . .                                | 1           |
| 2. Tensile Strength and Modulus of Elasticity Results. . .                              | 2           |
| a. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$ System . . . . .                     | 2           |
| b. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$ System . . . . .                     | 10          |
| c. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ System . . . . .                     | 25          |
| d. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Li}_2\text{O}$ System . . . . .            | 36          |
| e. High Modulus Glasses . . . . .                                                       | 39          |
| <b>Section III</b> . . . . .                                                            | 42          |
| <b>Relationship Between Composition and Properties of</b>                               |             |
| <b>Selected Glasses.</b> . . . .                                                        | 42          |
| 1. Background Relating to Study. . . . .                                                | 42          |
| 2. Experimental Procedure. . . . .                                                      | 43          |
| a. Formulation of Compositions Studied . . . . .                                        | 43          |
| b. Preparation of Melts. . . . .                                                        | 43          |
| c. Preparation of Fibers . . . . .                                                      | 43          |
| d. Tensile Strength Measurement. . . . .                                                | 44          |
| e. Modulus of Elasticity Measurement . . . . .                                          | 44          |
| f. Density Measurement . . . . .                                                        | 45          |
| 3. Results . . . . .                                                                    | 45          |
| a. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O}$ System . . . . .             | 45          |
| b. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Li}_2\text{O}$ System . . . . .            | 49          |
| c. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O-Li}_2\text{O}$ System. . . . . | 54          |
| d. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ System . . . . .                     | 63          |
| e. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-CaO}$ System . . . . .                     | 65          |
| f. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$ System . . . . .                     | 69          |
| 4. Discussion of Results . . . . .                                                      | 72          |
| <b>References.</b> . . . . .                                                            | 75          |

# LIST OF ILLUSTRATIONS

| <u>Figure</u> |                                                                                                                                          | <u>Page</u> |
|---------------|------------------------------------------------------------------------------------------------------------------------------------------|-------------|
| 1             | Tensile Strength Apparatus . . . . .                                                                                                     | 3           |
| 2             | Modulus of Elasticity Apparatus . . . . .                                                                                                | 4           |
| 3             | SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> -ZnO System . . . . .                                                                   | 8           |
| 4             | Modulus Vs. 2 Al/Zn Ratio. . . . .                                                                                                       | 9           |
| 5             | SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> -BeO System . . . . .                                                                   | 17          |
| 6             | SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> -MgO System . . . . .                                                                   | 31          |
| 7             | Modulus Vs. 2 Al/Mg Ratio. . . . .                                                                                                       | 33          |
| 8             | SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> -Li <sub>2</sub> O System . . . . .                                                     | 38          |
| 9             | Modulus Vs. Al/K Ratio . . . . .                                                                                                         | 47          |
| 10            | Modulus Vs. $\frac{Si + Al}{O}$ Ratio For Glasses Containing K <sub>2</sub> O. . . . .                                                   | 48          |
| 11            | Log Viscosity Vs. Temperature For Glasses in the<br>SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> -Li <sub>2</sub> O System . . . . . | 51          |
| 12            | Modulus Vs. Al/Li Ratio . . . . .                                                                                                        | 53          |
| 13            | Modulus Vs. $\frac{Si + Al}{O}$ Ratio For Glasses Containing Li <sub>2</sub> O . . . . .                                                 | 55          |
| 14            | Silica-Alumina-Lithia Glasses<br>Density Vs. Composition. . . . .                                                                        | 56          |
| 15            | Silica-Alumina-Potash-Lithia Glasses<br>Modulus Vs. Al/R Ratio at 75% SiO <sub>2</sub> . . . . .                                         | 60          |
| 16            | Silica-Alumina-Potash-Lithia Glasses<br>Modulus Vs. Al/R Ratio at 66% SiO <sub>2</sub> . . . . .                                         | 61          |
| 17            | Silica-Alumina-Potash-Lithia Glasses<br>Modulus Vs. Al/R Ratio at 57% SiO <sub>2</sub> . . . . .                                         | 62          |
| 18            | Density Vs. 2 Al/Mg Ratio . . . . .                                                                                                      | 66          |
| 19            | Modulus Vs. 2 Al/Ca Ratio For Silica-Alumina-Calcia<br>Glasses . . . . .                                                                 | 70          |

## LIST OF TABLES

| <u>Table</u>                                                                                                            | <u>Page</u> |
|-------------------------------------------------------------------------------------------------------------------------|-------------|
| I $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$ System and Modifications . . . . .                                    | 6-7         |
| II $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$ System and Modifications . . . . .                                   | 11-16       |
| III   The Effect of Thermal Treatment on the Tensile<br>Strength and Modulus of X-2124 . . . . .                        | 20          |
| IV   Property Comparisons. . . . .                                                                                      | 21          |
| V    Laminate Properties for X-2124 and AF-994 Glasses . . . . .                                                        | 24          |
| VI $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ System and Modifications . . . . .                                   | 26-31       |
| VII   Effect of Forming Temperature and Orifice Size on<br>AF-994 Tensile Strength and Modulus of Elasticity . . . . .  | 35          |
| VIII $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Li}_2\text{O}$ System. . . . .                                           | 37          |
| IX   Compositions Prepared for High Modulus Studies . . . . .                                                           | 40-4        |
| X    Compositions and Properties of Silica-Alumina-Potash<br>Glasses . . . . .                                          | 46          |
| XI   Compositions and Properties of Silica-Alumina-Lithia<br>Glasses . . . . .                                          | 50          |
| XII   Composition and Properties of Silica-Alumina-Potash-<br>Lithia Glasses, Series I . . . . .                        | 57          |
| XIII   Composition and Properties of Silica-Alumina-Potash-<br>Lithia Glasses, Series 2 . . . . .                       | 58          |
| XIV   Ionic Radii . . . . .                                                                                             | 63          |
| XV   Compositions and Properties of $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ Glasses . . . . .                   | 64          |
| XVI   Composition and Properties of Glasses in the $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-}$<br>CaO System . . . . . | 67-6        |
| XVII   Compositions and Properties of $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$ Glasses. . . . .                  | 71          |

# HIGH STRENGTH - HIGH MODULUS GLASS FIBERS

## SECTION I

### INTRODUCTION

This present study is a continuation of the work reported in Technical Report AFML-TR-65-90, Part I, High Strength-High Modulus Glass Fibers<sup>(1)</sup>. Its purpose has been the preparation and study of glass compositions capable of producing glass fibers with high tensile strengths and high moduli of elasticity. An additional study was also continued which has sought to establish a relationship between tensile strength, modulus of elasticity, and the composition and probable constitution of selected glasses. Results of this additional study are presented in the section on "Relationship Between Composition and Properties of Selected Glasses."

In the study of high strength- high modulus glass fibers, emphasis was placed on glass compositions in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$ ,  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ ,  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$ , and  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Li}_2\text{O}$  systems. Subsequent studies were made by adding various oxides to these base systems.

In the study of the relationship between composition and properties of glass, the systems studied contained  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  plus one of the following:  $\text{K}_2\text{O}$ ,  $\text{Li}_2\text{O}$ ,  $\text{K}_2\text{O} + \text{Li}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{ZnO}$  and  $\text{BeO}$ . This represents a continuation of the initial work in which the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O}$  system was studied.

## SECTION II

### Study of High Tensile Strength-High Modulus Glass Fiber Compositions

#### 1. Glass Preparation, Fiberization, and Testing

Each glass composition was prepared by dry mixing the appropriate oxides, which were of at least U.S.P. grade, in a double-cone agitator wheel blender for approximately ten minutes. The dry batch was melted in precious metal crucibles. The melting temperatures utilized ranged from  $2900^\circ\text{F}$  to  $3200^\circ\text{F}$ ; each glass was held at temperature for periods ranging from 2 to 24 hours. All glasses were cooled by plunging the hot crucible directly from the furnace into a water bath. Fibers were obtained from the glass compositions by remelting the prepared cullet in a one-hole precious metal bushing capable of reaching temperatures in excess of  $3200^\circ\text{F}$ .

To test the fibers, virgin filaments were captured by hand in the usual manner and, to determine the tensile strength, were wound on a stainless steel "fork" or holder. Each fork normally contained 6 to 8 fiber lengths for study after this operation. The apparatus for measuring tensile strength is designed to test one

fiber at a time, recording essentially the load elongation diagram of each fiber. The fibers are loaded by a screw-driven cross-head at a constant rate of strain of 0.25 in/in./minute over a gage length of two inches. The load cell consists of a cantilever beam with a linear variable differential transformer (LVDT) as the displacement sensing element. The load cell is calibrated by dead-weight loading. The LVDT is powered by a strain gage amplifier. The signals from the LVDT are first rectified through matched pairs of germanium diodes, then passed through gain and zero potentiometers and recorded on a single channel Esterline-Angus recorder. A photograph of the apparatus is shown in Figure 1.

The fiber diameter of each fiber length (6-8) was determined from a short length of fiber remaining on the fork after a sample was mounted on the tensile strength apparatus. These short fiber lengths were mounted on a microscope slide in a liquid of appropriate index of refraction. The fiber diameters were measured under a 430 power microscope using a Vickers Image-Splitting Eyepiece.\*

The measurement of tensile strength was considered, in this study, to be of primary importance. Care was taken, therefore, to obtain values which were representative of an optimized fiberization operation. The modulus of elasticity measurements were considered of secondary importance, except for glasses which showed promise of also having good tensile strength and density values.

The modulus of elasticity of a particular glass composition was determined by direct measurement of the elongation of an unannealed fiber under load. A photograph of the apparatus is shown in Figure 2.

Two fibers were utilized for the measurement. The sample diameter normally ranged from 0.00025- 0.00040 inches. To prevent fiber breakage under elongation only 1-5 gram weights could be used. All these factors (low sample numbers, small sample diameters, and low load application) produced modulus values which are less accurate than values described in Section III of this report titled "Relationship Between Composition and Properties of Selected Glasses." Indications are that the values are too low rather than too high under the test conditions described.

The bulk density of a glass sample was determined by the Archimedes method, using a piece of culet weighing 5-7 grams which was essentially seed-free.

## 2. Tensile Strength and Modulus of Elasticity Results

### a. SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-ZnO System

Of those glass systems studied under the first year's program,

---

\*Supplied by Cooke, Troughton and Simms, Inc., Massachusetts

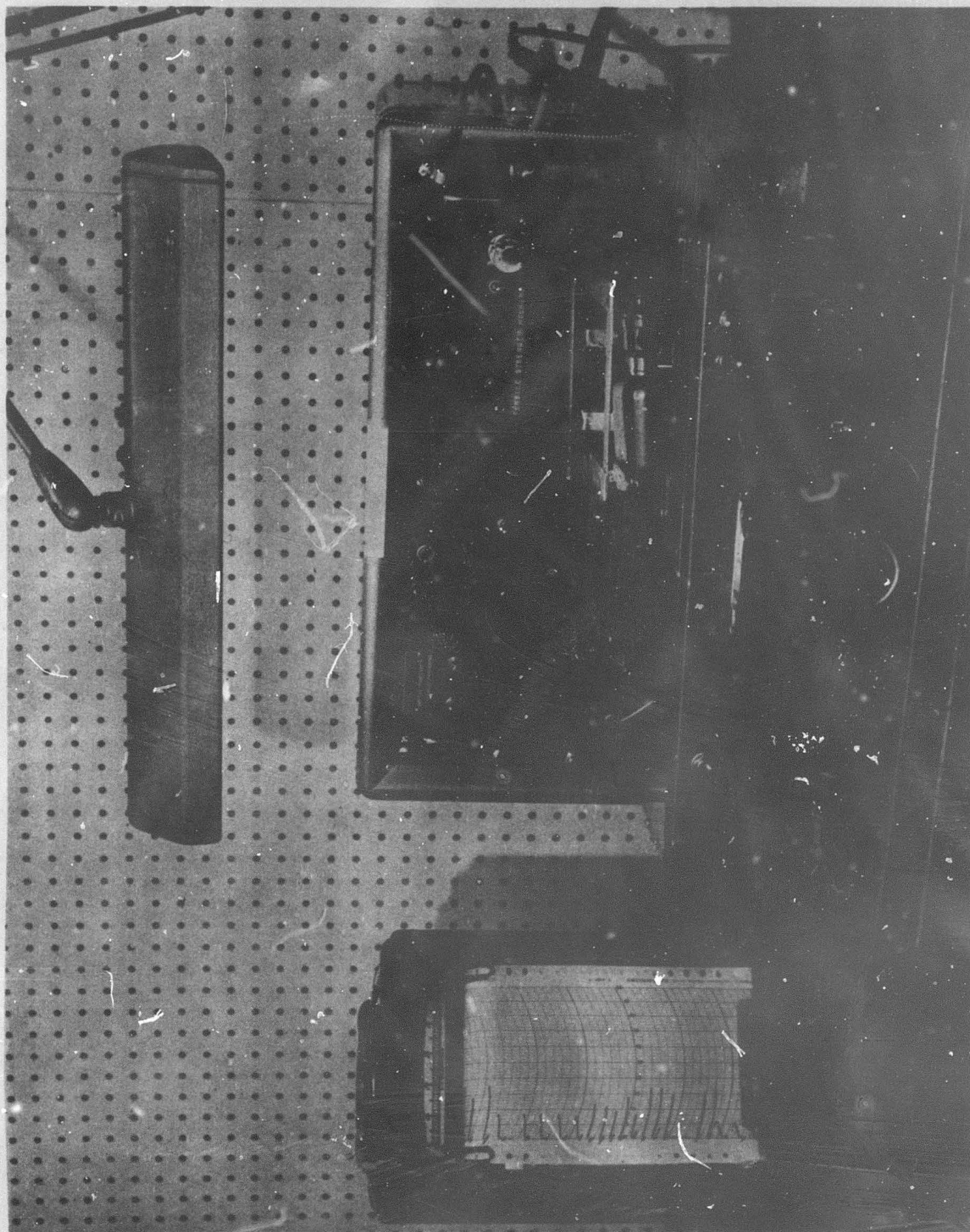


Figure 1 Tensile Strength Apparatus



Figure 2 Modulus of Elasticity Apparatus

one of the most promising was the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$  system. The system contains glass X-1792-2, which was previously reported to have a tensile strength in excess of 800,000 psi. More extensive studies of  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$  glasses were therefore carried out. Those compositions studied are shown in Table I. Various properties for each composition are also shown in Table I. Also see Figure 3, a diagram of the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$  system showing the location of all three component glasses studied in the system.

Previous work with this system indicated that the best tensile strength properties could be found with glasses having  $\text{Al}_2\text{O}_3$  to ZnO mole ratios of 1.0. Eight compositions with variable  $\text{SiO}_2$  contents were therefore selected at this mole ratio. Four others were selected at a mole ratio of 0.5. Two were selected at mole ratios to either side of 1.0. A small amount of MgO was also added to one final composition.

The eight glasses represented by X-2034 through X-2041 ( $\text{Al}_2\text{O}_3$  to ZnO mole ratio equals 1.0) demonstrate the effect of variations in  $\text{SiO}_2$  content on the tensile strength of the final glass fiber. As indicated by the results in Table I, the tensile strength increases from 60-68 mole percent  $\text{SiO}_2$  and then decreases slowly from 68-80 mole percent  $\text{SiO}_2$ . In the same series the modulus has decreased continuously from 60-80 mole percent  $\text{SiO}_2$ . The bulk density has also decreased continuously over the same range of values. The tensile strength, therefore, is a maximum at a point approximately midway between the minimum and maximum modulus and midway between the maximum and minimum bulk density. This has proved to be the case in all of the other three-component systems investigated. It appears then that no one glass in a system can possess the highest tensile strength, the highest modulus, and the lowest density of all glasses in the system.

The four glasses represented by X-2043 through X-2046 ( $\text{Al}_2\text{O}_3$  to ZnO mole ratio of 0.5) demonstrate the effect on tensile strength of a reduction in the ratio of  $\text{Al}_2\text{O}_3$  to ZnO. The increase in the ZnO content reduces the strength, at these  $\text{SiO}_2$  levels, and increases the modulus and density. Glasses with an  $\text{Al}_2\text{O}_3$  to ZnO mole ratio slightly below 1.0 (see X-2042) show similar reactions with respect to the tensile strength and density.

Glasses with  $\text{Al}_2\text{O}_3$  to ZnO mole ratios slightly greater than 1.0 (X-2060) produce no great modulus improvement and no strength improvement.  $\text{Al}_2\text{O}_3$  to ZnO mole ratios greater than 1.13 (X-2060) were not studied because of the expected increases in forming temperature and decreases in tensile strength. The relationship

TABLE I

SiO<sub>2</sub> - Al<sub>2</sub>O<sub>3</sub> - ZnO System and Modifications

| Glass No. | Oxides (a. weight %, b. mole %) |                                |     |       | Average Tensile Strength $\times 10^3$ psi | Modulus of Elasticity $\times 10^6$ psi | Average Diameter $\times 10^{-5}$ in. | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------|---------------------------------|--------------------------------|-----|-------|--------------------------------------------|-----------------------------------------|---------------------------------------|------------------|---------------|-------------------|
|           | SiO <sub>2</sub>                | Al <sub>2</sub> O <sub>3</sub> | MgO | ZnO   | BeO                                        | Li <sub>2</sub> O                       |                                       |                  |               |                   |
| X-2034    | a. 44.5                         | 30.9                           |     | 24.6  |                                            |                                         |                                       |                  |               |                   |
|           | b. 55.0                         | 22.5                           |     | 22.5  |                                            |                                         |                                       |                  |               |                   |
| X-2035    | 49.57                           | 28.05                          |     | 22.38 |                                            |                                         |                                       |                  |               |                   |
|           | 60.0                            | 20.0                           |     | 20.0  |                                            |                                         | 32                                    | 2880             | 17            | 2.8               |
| X-2036    | 54.9                            | 25.1                           |     | 20.0  |                                            |                                         |                                       |                  |               |                   |
|           | 65.0                            | 17.5                           |     | 17.5  |                                            |                                         | 37                                    | 2850             | 14            | 2.8               |
| X-2037    | 58.2                            | 23.2                           |     | 18.6  |                                            |                                         |                                       |                  |               |                   |
|           | 68.0                            | 16.0                           |     | 16.0  |                                            |                                         | 30                                    | 2955             | 9             | 2.7               |
| X-2038    | 60.46                           | 21.99                          |     | 17.55 |                                            |                                         |                                       |                  |               |                   |
|           | 70.0                            | 15.0                           |     | 15.0  |                                            |                                         | 22                                    | 2945             | 17            | 2.7               |
| X-2039    | 62.8                            | 20.7                           |     | 16.5  |                                            |                                         |                                       |                  |               |                   |
|           | 72.0                            | 14.0                           |     | 14.0  |                                            |                                         | 33                                    | 2995             | 15            | 2.6               |
| X-2040    | 66.29                           | 18.75                          |     | 14.96 |                                            |                                         |                                       |                  |               |                   |
|           | 75.0                            | 12.5                           |     | 12.5  |                                            |                                         | 34                                    | 3025             | 16            | 2.7               |
| X-2041    | 72.39                           | 15.36                          |     | 12.26 |                                            |                                         |                                       |                  |               |                   |
|           | 80.0                            | 10.0                           |     | 10.0  |                                            |                                         | 28                                    | 3075             | 16            | 2.5               |

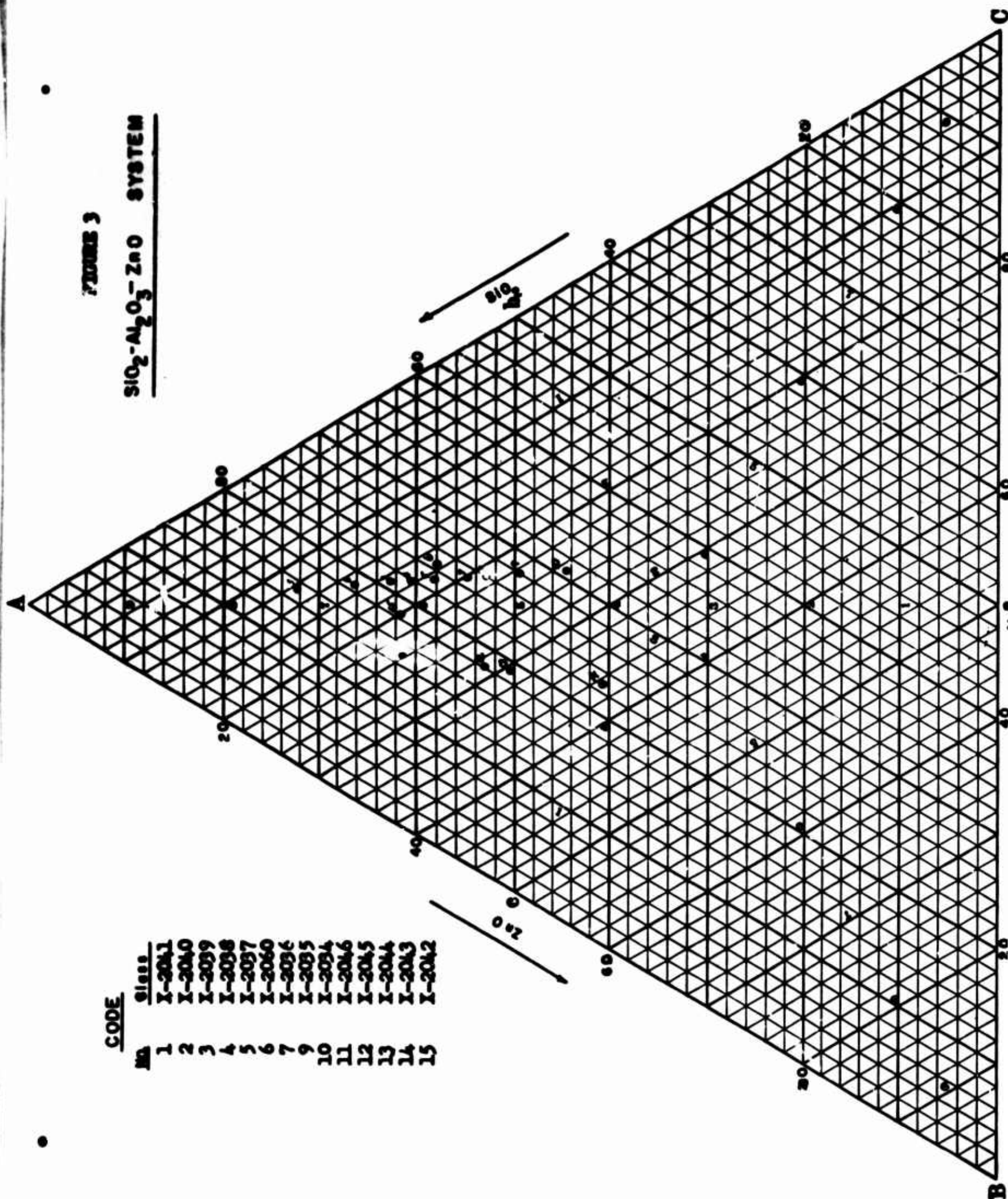
Could not be fiberized

TABLE I (Continued)

| Glass No.                         | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO         | ZnO           | BeO | Li <sub>2</sub> O | Average Tensile Strength x 10 <sup>3</sup> psi | Modulus of Elasticity x 10 <sup>6</sup> psi | Average Diameter x 10 <sup>-5</sup> in. | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------------------------------|------------------|--------------------------------|-------------|---------------|-----|-------------------|------------------------------------------------|---------------------------------------------|-----------------------------------------|------------------|---------------|-------------------|
| X-2042                            | 60.8<br>70.0     | 19.2<br>13.0                   |             | 20.0<br>17.0  |     |                   | 394                                            | 11.2                                        | 37                                      | 2850             | 16            | 2.78              |
| X-2043                            | 40.5<br>50.0     | 22.9<br>16.7                   |             | 36.6<br>33.3  |     |                   | Could not be fiberized                         |                                             |                                         |                  |               |                   |
| X-2044                            | 50.5<br>60.0     | 19.1<br>13.3                   |             | 30.4<br>26.7  |     |                   | 557                                            | 12.2                                        | 29                                      | 2715             | 18            | 2.9               |
| X-2045                            | 53.2<br>62.5     | 18.0<br>12.5                   |             | 28.8<br>25.0  |     |                   | 314                                            | 12.8                                        | 30                                      | 2700             | 16            | 2.9               |
| X-2046                            | 61.37<br>70.0    | 14.88<br>10.0                  |             | 23.75<br>20.0 |     |                   | 468                                            | 11.3                                        | 44                                      | 2900             | 14            | 2.8               |
| X-2060                            | 58.04<br>68.0    | 24.62<br>17.0                  |             | 17.34<br>15.0 |     |                   | 488                                            | 11.9                                        | 36                                      | 2850             | 18            | 2.6               |
| X-2324                            | 62.7<br>68.68    | 20.8<br>13.42                  | 5.5<br>8.95 | 11.1<br>8.95  |     |                   | 690                                            | 12.4                                        | 34                                      | 2900             | 15            | 2.63              |
| X-1792-2<br>(previously reported) | 59.0<br>68.68    | 22.7<br>15.56                  |             | 18.3<br>15.76 |     |                   | 815                                            | 11.5                                        | 26                                      | 2915             | 11            | 2.7               |

FIGURE 3

SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-ZnO SYSTEM



CODE

| MA    | 1      | 2      | 3      | 4      | 5      | 6      | 7      | 9      | 10     | 11     | 12     | 13     | 14     | 15     |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Class | I-2041 | I-2040 | I-2039 | I-2038 | I-2037 | I-2040 | I-2036 | I-2035 | I-2034 | I-2046 | I-2045 | I-2044 | I-2043 | I-2042 |

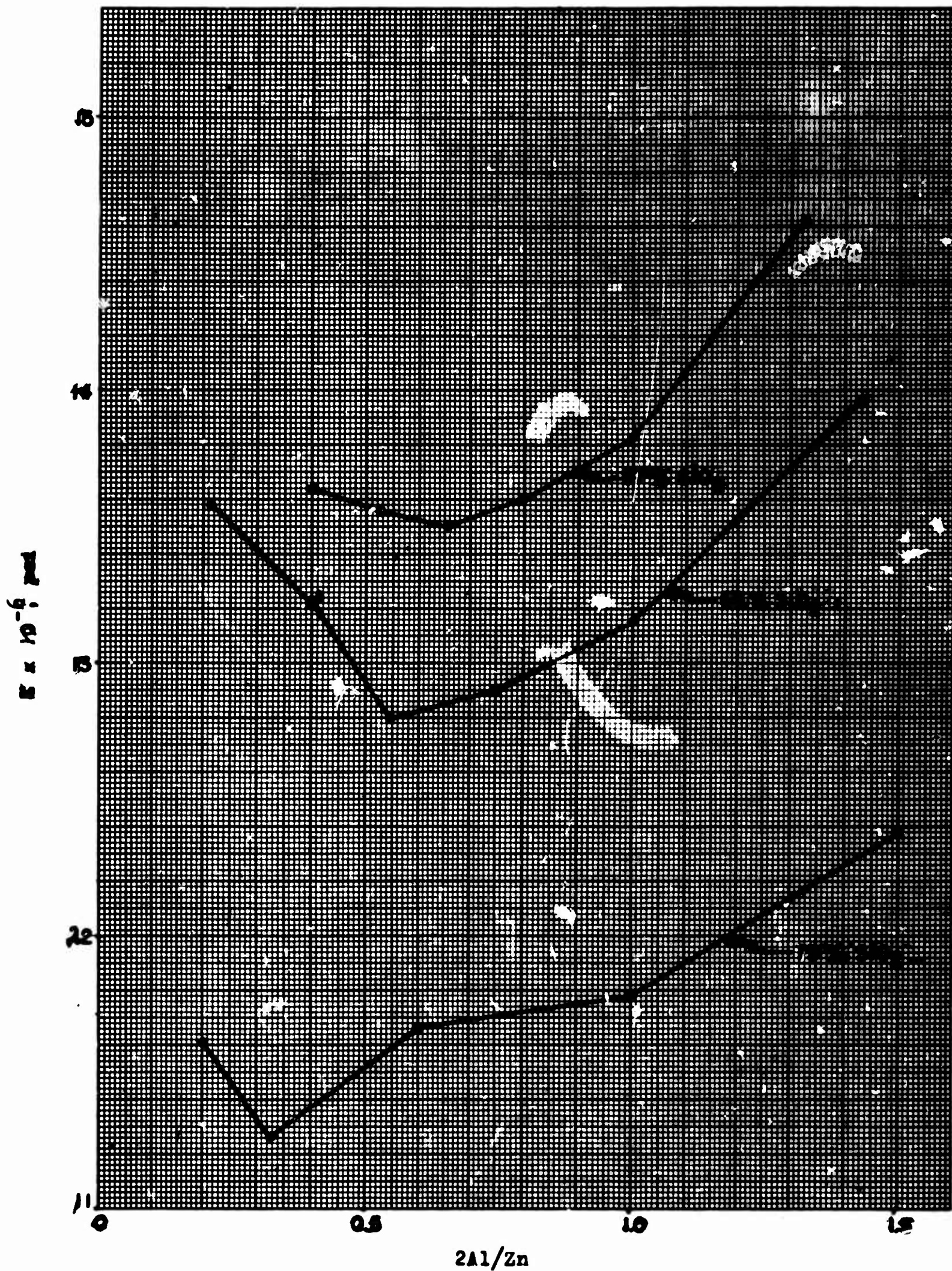


Figure 4 Modulus Vs. 2 Al/Zn Ratio

between modulus and  $\text{Al}_2\text{O}_3$  to  $\text{ZnO}$  mole ratio are more clearly shown in the study, "Relationship Between Composition and Properties of Selected Glasses" (See Figure 4). The primary problem with glasses in the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{ZnO}$  system, some of which have tensile strength properties equal to or greater than S-994, is the fact that the densities are greater than 2.5 g/cc. As shown by X-2324, a modification of X-1792-2 containing  $\text{MgO}$ , both the modulus and density values can be improved by the proper addition of an appropriate oxide. Strength is sacrificed, however, and the density still does not drop below 2.5 g/cc.

To determine some of the problems which might be expected with a glass of the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{ZnO}$  type, a multifilament trial of X-1792-2 was made. A standard 204-hole production-type bushing was utilized and an initial run of 20 hours was made. Bushing capacity and design were such that, after 10 hours, devitrification had begun to interfere with the fiberization process. Nevertheless, three sample forks of tensile strength fibers were obtained five hours after the run started. The strength, an average of 21 separate breaks, was 635,000 psi. This was considered to be excellent in light of the fact that strength optimization in a multifilament operation requires at least 24 hours of continuous, uninterrupted operation.

No problems were seen that would prevent eventual continuous multifilament fiberization of X-1792-2. Proper selection of a standard 204-hole production bushing, keeping in mind the higher forming temperature, higher density, and increased devitrification tendencies of this glass, would be the only major consideration required.

b.  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO System

Another promising glass system studied during the course of the year's program was the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO system. More extensive studies of this system were therefore made. These initially dealt with glasses having  $\text{Al}_2\text{O}_3$  to BeO mole ratios of 1.0 or 0.5 and variable mole percentages of  $\text{SiO}_2$ . The composition and properties of these glasses are shown in Table II titled " $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO System and Modifications" (see X-2047 to X-2055).

Figure 5 shows the location of these and the remaining three-component glasses studied in the system.

At an  $\text{Al}_2\text{O}_3$  to BeO mole ratio of 1.0, as indicated by the results for X-2047 through X-2051, the fiber tensile strength increases from 60 to 70 mole percent  $\text{SiO}_2$  and then decreases from 70 to 80 mole percent  $\text{SiO}_2$ . Over the same range of values the modulus and the bulk density decrease continuously. Maximum tensile strength and modulus with minimum density are therefore not found in any one glass.

TABLE II

SiO<sub>2</sub> - Al<sub>2</sub>O<sub>3</sub> - BeO System and Modifications

| Glass No. | Oxides (a. weight %, b. mole %) |                                |       | Average Modulus                        |                                         | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------|---------------------------------|--------------------------------|-------|----------------------------------------|-----------------------------------------|------------------|---------------|-------------------|
|           | SiO <sub>2</sub>                | Al <sub>2</sub> O <sub>3</sub> | MgO   | Tensile Strength x 10 <sup>3</sup> psi | Elasticity x 10 <sup>6</sup> psi        |                  |               |                   |
|           |                                 |                                |       |                                        | Average Diameter x 10 <sup>-5</sup> in. |                  |               |                   |
| X-2047    | a.                              | 58.67                          | 33.19 |                                        |                                         |                  |               |                   |
|           | b.                              | 60.0                           | 20.0  |                                        |                                         |                  |               |                   |
|           |                                 |                                |       |                                        |                                         |                  |               |                   |
| X-2048    |                                 | 63.74                          | 29.12 |                                        |                                         |                  |               |                   |
|           |                                 | 65.0                           | 17.5  |                                        |                                         |                  |               |                   |
| X-2049    |                                 | 69.1                           | 25.1  |                                        |                                         |                  |               |                   |
|           |                                 | 70.0                           | 15.0  |                                        |                                         |                  |               |                   |
| X-2050    |                                 | 74.0                           | 20.9  |                                        |                                         |                  |               |                   |
|           |                                 | 75.0                           | 12.5  |                                        |                                         |                  |               |                   |
| X-2051    |                                 | 79.1                           | 16.8  |                                        |                                         |                  |               |                   |
|           |                                 | 80.0                           | 10.0  |                                        |                                         |                  |               |                   |
| X-2052    |                                 | 64.02                          | 24.14 |                                        |                                         |                  |               |                   |
|           |                                 | 60.0                           | 13.3  |                                        |                                         |                  |               |                   |
| X-2053    |                                 | 68.78                          | 20.96 |                                        |                                         |                  |               |                   |
|           |                                 | 65.0                           | 11.7  |                                        |                                         |                  |               |                   |
| X-2054    |                                 | 73.46                          | 17.81 |                                        |                                         |                  |               |                   |
|           |                                 | 70.0                           | 10.0  |                                        |                                         |                  |               |                   |
| X-2055    |                                 | 78.1                           | 14.7  |                                        |                                         |                  |               |                   |
|           |                                 | 75.0                           | 8.3   |                                        |                                         |                  |               |                   |

Could not be fiberized

TABLE II (Continued)

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO          | BeO          | Li <sub>2</sub> O | Average Tensile Strength $\times 10^3$ psi | Modulus of Elasticity $\times 10^6$ psi | Average Diameter $\times 10^{-5}$ in. | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------|------------------|--------------------------------|--------------|--------------|-------------------|--------------------------------------------|-----------------------------------------|---------------------------------------|------------------|---------------|-------------------|
| X-2056    | 47.2<br>50.0     | 40.0<br>25.0                   | 7.9<br>12.5  | 4.9<br>12.5  |                   | Could not be fiberized                     |                                         |                                       |                  |               |                   |
| X-2057    | 57.25<br>60.0    | 32.39<br>20.0                  | 6.40<br>10.0 | 3.97<br>10.0 |                   | Could not be fiberized                     |                                         |                                       |                  |               |                   |
| X-2058    | 67.56<br>70.0    | 24.57<br>15.0                  | 4.85<br>7.5  | 3.01<br>7.5  |                   | 614                                        | 11.4                                    | 33                                    | 2915             | 15            | 2.47              |
| X-2059    | 78.1<br>80.0     | 16.6<br>10.0                   | 3.3<br>5.0   | 2.0<br>5.0   |                   | 600                                        | 11.0                                    | 25                                    | 3050             | 15            | 2.3               |
| X-2061    | 67.2<br>70.0     | 24.4<br>15.0                   | 6.4<br>10.0  | 2.0<br>5.0   |                   | 739                                        | 12.9                                    | 20                                    | 2915             | 16            | 2.5               |
| X-2062    | 66.7<br>70.0     | 24.3<br>15.0                   | 8.0<br>12.5  | 1.0<br>2.5   |                   | 510                                        | 12.4                                    | 35                                    | 2915             | 16            | 2.5               |
| X-2063    | 68.4<br>70.0     | 24.9<br>15.0                   |              | 3.1<br>7.5   | 3.6<br>7.5        | 757                                        | 11.7                                    | 14                                    | 2915             | 15            | 2.41              |
| X-2064    | 67.4<br>70.0     | 24.5<br>15.0                   | 4.8<br>7.5   | 1.5<br>3.75  | 1.8<br>3.75       | 776                                        | 12.2                                    | 19                                    | 3060             | 17            | 2.38              |
| X-2065    | 67.99<br>70.0    | 24.73<br>15.0                  | 2.44<br>3.75 | 3.03<br>7.5  | 1.81<br>3.75      | 653                                        | 11.7                                    | 30                                    | 2975             | 18            | 2.41              |
| X-2124    | 71.1<br>70.0     | 21.5<br>12.5                   |              | 7.4<br>17.5  |                   | 681                                        | 14.1                                    | 31                                    | 2850-<br>2915    | 15            | 2.41              |

TABLE II (Continued)

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO         | ZnO        | BeO         | Li <sub>2</sub> O | Average Tensile Strength x 10 <sup>3</sup> psi | Modulus of Elasticity x 10 <sup>6</sup> psi | Average Diameter x 10 <sup>-5</sup> in. | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------|------------------|--------------------------------|-------------|------------|-------------|-------------------|------------------------------------------------|---------------------------------------------|-----------------------------------------|------------------|---------------|-------------------|
| X-2125    | 61.9<br>65.0     | 28.3<br>17.5                   | 7.5<br>11.7 |            | 2.3<br>5.8  |                   | 841                                            | 13.3                                        | 20                                      | 2850             | 15            | 2.5               |
| X-2126    | 67.1<br>70.0     | 27.7<br>17.0                   |             |            | 5.2<br>13.0 |                   | 778                                            | 14.1                                        | 32                                      | 3040             | 15            | 2.45              |
| X-2127    | 61.9<br>65.0     | 32.1<br>19.8                   |             |            | 6.0<br>15.2 |                   | 655                                            | 13.6                                        | 37                                      | 3100             | 15            | 2.52              |
| X-2128    | 62.4<br>64.0     | 29.8<br>18.0                   |             |            | 4.9<br>12.0 | 2.9<br>6.0        | 643                                            | 13.0                                        | 27                                      | 2915             | 16            | 2.45              |
| X-2129    | 62.2<br>65.0     | 28.4<br>17.5                   | 5.6<br>8.75 |            | 1.7<br>4.38 | 2.1<br>4.38       | 764                                            | 13.3                                        | 24                                      | 2850             | 15            | 2.47              |
| X-2132    | 57.0<br>60.0     | 32.3<br>20.0                   | 6.4<br>10.0 |            | 2.0<br>5.0  | 2.4<br>5.0        | 590                                            | 13.4                                        | 38                                      | 2850             | 15            | 2.55              |
| X-2167    | 66.2<br>65.0     | 25.2<br>14.6                   |             |            | 8.6<br>20.4 |                   | 537                                            | 14.2                                        | 27                                      | 2900             | 15            | 2.40              |
| X-2168    | 59.4<br>60.0     | 29.4<br>17.5                   | 5.0<br>7.5  |            | 6.2<br>15.0 |                   | 421                                            | 15.7                                        | 36                                      | 2850             | 10            | 2.55              |
| X-2170    | 61.6<br>65.0     | 28.2<br>17.5                   | 1.8<br>2.9  | 3.7<br>2.9 | 4.6<br>11.7 |                   | 712                                            | 12.9                                        | 26                                      | 2955             | 15            | 2.55              |
| X-2171    | 60.6<br>65.0     | 27.6<br>17.5                   |             | 7.3<br>5.8 | 4.5<br>11.7 |                   | 694                                            | 14.3                                        | 34                                      | 2955             | 17            | 2.6               |

TABLE II (Continued)

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO | ZnO | BeO         | Li <sub>2</sub> O | Average<br>Tensile<br>Strength<br>$\times 10^3$ psi | Modulus<br>of<br>Elasticity<br>$\times 10^6$ psi | Average<br>Diameter<br>$\times 10^{-5}$ in. | Forming<br>Temp.<br>°F | No. of<br>Fibers | Bulk<br>Density<br>g/cc |
|-----------|------------------|--------------------------------|-----|-----|-------------|-------------------|-----------------------------------------------------|--------------------------------------------------|---------------------------------------------|------------------------|------------------|-------------------------|
| X-2252    | 73.0<br>71.8     | 20.0<br>11.6                   |     |     | 7.0<br>16.6 |                   | 500                                                 | 13.3                                             | 34                                          | 2975                   | 15               | 2.4                     |
| X-2253    | 71.0<br>68.6     | 20.5<br>11.7                   |     |     | 8.5<br>19.7 |                   | 702                                                 | 13.8                                             | 29                                          | 2965                   | 15               | 2.4                     |
| X-2254    | 71.0<br>71.1     | 22.5<br>13.3                   |     |     | 6.5<br>15.6 |                   | 452                                                 | 11.5                                             | 28                                          | 2955                   | 5                | 2.4                     |
| X-2255    | 69.0<br>67.8     | 23.0<br>13.3                   |     |     | 8.0<br>18.9 |                   | 473                                                 | 12.2                                             | 39                                          | 2975                   | 17               | 2.5                     |
| X-2256    | 68.0<br>65.3     | 22.5<br>12.7                   |     |     | 9.5<br>21.9 |                   | 521                                                 | 12.3                                             | 35                                          | 3040                   | 15               | 2.6                     |
| X-2257    | 68.0<br>67.7     | 24.5<br>14.4                   |     |     | 7.5<br>17.9 |                   | 651                                                 | 13.5                                             | 34                                          | 2965                   | 15               | 2.3                     |
| X-2258    | 67.0<br>65.8     | 24.5<br>14.2                   |     |     | 8.5<br>20.1 |                   | 422                                                 | 13.7                                             | 45                                          | 2975                   | 15               | 2.4                     |
| X-2259    | 65.0<br>63.8     | 26.0<br>15.0                   |     |     | 9.0<br>21.2 |                   | 439                                                 | 12.9                                             | 48                                          | 2850                   | 15               | 2.5                     |
| X-2260    | 62.5<br>61.4     | 28.0<br>16.2                   |     |     | 9.5<br>22.4 |                   | 633                                                 | 13.9                                             | 21                                          | 2880                   | 17               | 2.5                     |
| X-2261    | 74.0<br>76.6     | 22.0<br>13.4                   |     |     | 4.0<br>10.0 |                   | 754                                                 | 12.8                                             | 19                                          | 3095                   | 15               | 2.4                     |

TABLE II (Continued)

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO  | ZnO  | BeO   | ZrO <sub>2</sub> | Average<br>Tensile<br>Strength<br>x 10 <sup>3</sup> psi | Modulus<br>of<br>Elasticity<br>x 10 <sup>6</sup> psi | Average<br>Dia. meter<br>x 10 <sup>-5</sup> in. | Forming<br>Temp.<br>°F | No. of<br>Fibers | Bulk<br>Density<br>g/cc |
|-----------|------------------|--------------------------------|------|------|-------|------------------|---------------------------------------------------------|------------------------------------------------------|-------------------------------------------------|------------------------|------------------|-------------------------|
| X-2275    | 72.0             | 20.8                           |      |      | 7.2   |                  | 416                                                     | 12.6                                                 | 40                                              | 3040                   | 16               | 2.4                     |
|           | 70.9             | 12.1                           |      |      | 17.0  |                  |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2276    | 70.0             | 22.4                           |      |      | 7.6   |                  | 542                                                     | 13.5                                                 | 36                                              | 2915                   | 15               | 2.4                     |
|           | 68.9             | 13.0                           |      |      | 18.1  |                  |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2277    | 71.0             | 21.0                           |      |      | 8.0   |                  | 511                                                     | 12.6                                                 | 46                                              | 3040                   | 14               | 2.4                     |
|           | 69.2             | 12.1                           |      |      | 18.7  |                  |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2278    | 71.0             | 22.0                           |      |      | 7.0   |                  | 481                                                     | 13.3                                                 | 39                                              | 2915                   | 15               | 2.45                    |
|           | 70.4             | 12.9                           |      |      | 16.7  |                  |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2279    | 69.62            | 21.10                          |      |      | 7.24  | 2.04             | 477                                                     | 13.9                                                 | 37                                              | 2915                   | 15               | 2.4                     |
|           | 69.31            | 12.38                          |      |      | 17.33 | 0.99             |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2280    | 68.2             | 30.7                           |      |      | 7.1   | 4.0              | 544                                                     | 13.1                                                 | 35                                              | 2915                   | 15               | 2.5                     |
|           | 68.63            | 12.25                          |      |      | 17.16 | 1.96             |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2281    | 66.9             | 20.3                           |      |      | 6.9   | 5.9              | 670                                                     | 14.4                                                 | 41                                              | 2950                   | 15               | 2.5                     |
|           | 68.0             | 12.1                           |      |      | 17.0  | 2.9              |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2282    | 68.3             | 20.7                           |      | 3.9  | 7.1   |                  | 476                                                     | 13.6                                                 | 35                                              | 2915                   | 15               | 2.5                     |
|           | 68.0             | 12.1                           |      | 2.9  | 17.0  |                  |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2283    | 66.5             | 20.2                           |      | 6.4  | 6.9   |                  | 422                                                     | 12.8                                                 | 31                                              | 2915                   | 15               | 2.5                     |
|           | 66.69            | 11.93                          |      | 4.74 | 16.64 |                  |                                                         |                                                      |                                                 |                        |                  |                         |
| X-2284    | 69.3             | 21.0                           | 1.3  |      | 8.4   |                  | 518                                                     | 13.8                                                 | 32                                              | 2845                   | 15               | 2.4                     |
|           | 68.63            | 12.25                          | 1.96 |      | 17.16 |                  |                                                         |                                                      |                                                 |                        |                  |                         |

**TABLE II (Continued)**

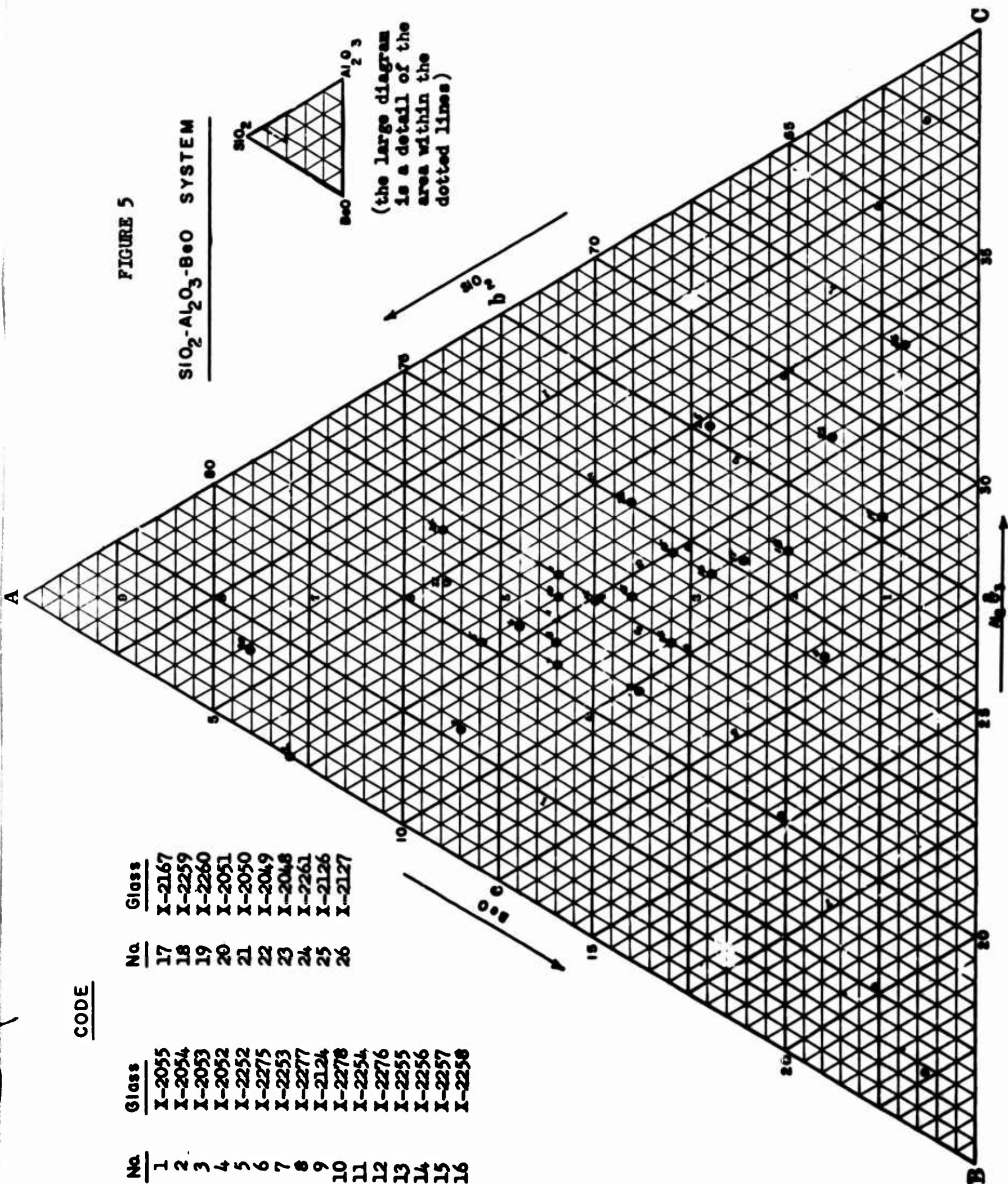
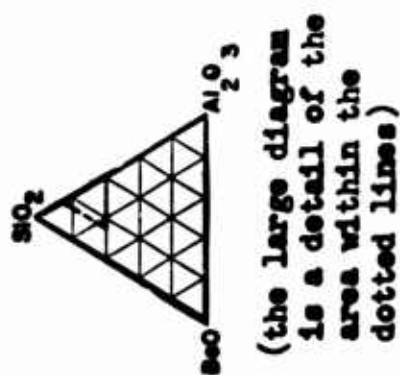
| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO  | BeO   | CeO <sub>2</sub> | ZrO <sub>2</sub> | Average<br>Tensile<br>Strength<br>x 10 <sup>3</sup> psi | Modulus<br>of<br>Elasticity<br>x 10 <sup>6</sup> psi | Average<br>Diameter<br>x 10 <sup>-5</sup> in. | Forming<br>Temp.<br>°F | No. of<br>Fibers | Bulk<br>Density<br>g/cc |
|-----------|------------------|--------------------------------|------|-------|------------------|------------------|---------------------------------------------------------|------------------------------------------------------|-----------------------------------------------|------------------------|------------------|-------------------------|
| X-2285    | 69.1             | 21.0                           | 2.7  | 7.2   |                  |                  | 715                                                     | 14.95                                                | 25                                            | 2915                   | 15               | 2.5                     |
|           | 67.31            | 12.02                          | 3.85 | 16.83 |                  |                  |                                                         |                                                      |                                               |                        |                  |                         |
| X-2320    | 69.71            | 21.08                          |      | 7.25  | 1.96             |                  | 545                                                     | 13.6                                                 | 27                                            | 2915                   | 14               | 2.5                     |
|           | 69.5             | 12.4                           |      | 17.4  | 0.7              |                  |                                                         |                                                      |                                               |                        |                  |                         |
| X-2321    | 68.4             | 20.7                           |      | 7.1   | 3.8              |                  | 565                                                     | 14.3                                                 | 27                                            | 2915                   | 15               | 2.5                     |
|           | 69.2             | 12.3                           |      | 17.2  | 1.3              |                  |                                                         |                                                      |                                               |                        |                  |                         |

CODE

| No. | Glass  |
|-----|--------|
| 1   | X-2055 |
| 2   | X-2054 |
| 3   | X-2053 |
| 4   | X-2052 |
| 5   | X-2252 |
| 6   | X-2275 |
| 7   | X-2253 |
| 8   | X-2277 |
| 9   | X-2124 |
| 10  | X-2278 |
| 11  | X-2254 |
| 12  | X-2276 |
| 13  | X-2255 |
| 14  | X-2256 |
| 15  | X-2257 |
| 16  | X-2258 |

| No. | Glass  |
|-----|--------|
| 17  | X-2167 |
| 18  | X-2259 |
| 19  | X-2260 |
| 20  | X-2051 |
| 21  | X-2050 |
| 22  | X-2049 |
| 23  | X-2048 |
| 24  | X-2261 |
| 25  | X-2126 |
| 26  | X-2127 |

FIGURE 5  
 $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$  SYSTEM



X-2052 through X-2055, with  $\text{Al}_2\text{O}_3$  to  $\text{BeO}$  mole ratios of 0.5, show a similar peak in strength at 70<sup>3</sup> mole percent  $\text{SiO}_2$ . In general, both the modulus and density decrease over the range of 60 to 75 mole percent  $\text{SiO}_2$ . The tensile strength and modulus properties of this series are lower than those for the X-2047 to X-2051 series, however.

A major problem encountered with the glasses previously discussed is their high forming temperatures. As seen in Table II, these are greater than 3000°F for all but two glasses.  $\text{MgO}$  and/or  $\text{Li}_2\text{O}$  were therefore added in various combinations both to lower forming temperature and to determine their effects on fiber strength, modulus and density. In Table II these glasses are designated X-2056 through X-2059 and X-2061 through X-2065.

The  $\text{Al}_2\text{O}_3$  to  $\text{BeO} + \text{R}_x\text{O}$  mole ratio for each glass was maintained at 1.0. X-2056 through X-2059, with  $\text{MgO}$  substituted for half the  $\text{BeO}$ , show a reduction in strength and modulus. Below 70 mole percent  $\text{SiO}_2$  the glass could not be fiberized. Additions of unequal proportions of  $\text{R}_x\text{O}$  and  $\text{BeO}$  were more successful, as shown by several glasses in the X-2061 to X-2065 series. Note that X-2061, X-2063, and X-2064 have strengths well in excess of 700,000 psi. X-2061 and X-2063 have more acceptable forming temperatures. As for the modulus of elasticity values for these three glasses, they should be related to the density of each glass. Since all three have lower densities than AF-994 for example, their specific moduli are greater than AF-994.

These modifications of the basic three-component system,  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{BeO}$ , were further modified to try to increase modulus of elasticity. X-2125 and X-2168 were melted and tested on the basis of results previously obtained for X-2058, X-2061 and X-2062. X-2125 and X-2168 have less  $\text{SiO}_2$  and more  $\text{Al}_2\text{O}_3$  on a mole basis; as expected, their moduli are higher than the compositions on which they are based. The high tensile strength of X-2125 is attributed to the high  $\text{Al}_2\text{O}_3$  content and optimum  $\text{Al}_2\text{O}_3$  to  $\text{RO}$  mole ratio. The modulus of X-2168 is attributed to its high  $\text{BeO}$  content.

X-2170 and X-2171 are modifications of X-2125 containing  $\text{ZnO}$ . In the case of X-2171 the  $\text{ZnO}$  addition apparently improved the modulus materially. However, it also produced an immediate increase in the bulk density.

X-2128, a modification based on X-2063, has a lower  $\text{SiO}_2$  content and a change in the ratio of  $\text{BeO}$  to  $\text{Li}_2\text{O}$ . As expected, when the  $\text{SiO}_2$  content is lowered, the modulus of X-2128 improves above that of X-2063, but its tensile strength drops. The density has also increased slightly.

Finally, X-2129 and X-2132 were based on X-2064 and have a lower  $\text{SiO}_2$  content with changes in the various ratios between

$\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{Li}_2\text{O}$ , and  $\text{BeO}$ . As expected, the lower  $\text{SiO}_2$  content improved the modulus and increased the density. In the case of X-2129, the final tensile strength was not impaired by these changes when compared to X-2064.

In general, these modification studies have shown that  $\text{MgO}$  and/or  $\text{Li}_2\text{O}$  may be added to glasses in the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{BeO}$  system in various combinations and they will improve fiberization properties and lower density. As the density is lowered within a given composition, however, the modulus also decreases. All of these effects seem to be tied to the  $\text{SiO}_2$  content. The higher  $\text{SiO}_2$  content glasses have improved strength, lower density, and lower modulus values. Lower  $\text{SiO}_2$  contents produce higher moduli, higher densities, and lower strengths.

Such modification operations, however, have not substantially improved overall glass properties in this system. More intensive studies of the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{BeO}$  system were therefore made in areas other than those associated with the  $\text{Al}_2\text{O}_3$  to  $\text{BeO}$  mole ratio of 1.0 and 0.5. Initial objectives were to locate compositions with higher tensile strengths and/or lower forming temperatures.

These objectives led to fiberization of glasses designated X-2124, X-2167, X-2126, and X-2127. Note that the  $\text{SiO}_2$  level for all four glasses falls between 65 and 70 mole percent, the area associated with fibers of the highest tensile strength. The first two compositions have an  $\text{Al}_2\text{O}_3$  to  $\text{BeO}$  mole ratio of 0.71, while with the second two,  $\text{Al}_2\text{O}_3/\text{BeO} = 1.31$ .

Though the tensile strength of X-2126 is the best in this series, its forming temperature is above  $3000^\circ\text{F}$ . It appears that this is the case for all glasses with an  $\text{Al}_2\text{O}_3$  to  $\text{BeO}$  mole ratio greater than 1.0. This is borne out by the other results (see the properties of X-2261,  $\text{Al}_2\text{O}_3/\text{BeO} = 1.34$ ). The high forming temperatures are attributed to the increased  $\text{Al}_2\text{O}_3$  content of these glasses.

X-2124 and X-2167 have much more desirable forming temperatures. Overall, X-2124 has the best properties, combining a high tensile strength, good modulus, and very low density. Virgin fiber samples of X-2124 were submitted to the Air Force Materials Laboratory for their evaluation. Their results are compared below to those originally reported by OCF.

#### AFML Results

T.S.  $= 643 \times 10^3$  psi  
E  $= 14.7 \times 10^6$  psi  
(sonic method)

Fiber Diameter  $= 33.4 \times 10^{-5}$  in.

#### OCF Results

T.S.  $= 681 \times 10^3$  psi  
E  $= 14.1 \times 10^6$  psi  
(elongation method)

Fiber Diameter for E measurement  $= 56 \times 10^{-5}$  in.

These very interesting results led to extensive studies of the region in Figure 5 bounded by an imaginary line drawn through the points labeled 1,2,3,4 and an imaginary line drawn through 20, 21, 22, 23. This region is characterized by  $\text{Al}_2\text{O}_3$  to  $\text{BeO}$  mole ratios ranging from 0.58 to 0.85 and  $\text{SiO}_2$  mole percentages of 61.4 to 71.8. The glasses in this region were designated X-2252 through X-2260 and X-2275 through X-2278. Their compositions and properties may be found in Table II and their positions in relation to the X-2124 composition may be seen in Figure 5.

It was expected that the properties of these glasses would in several cases be more desirable than those associated with X-2124. However, both the tensile strength and modulus values for all the glasses were lower than expected. From data developed later in melting and forming temperature studies of X-2124, it was concluded that the two property values were low because the glasses were not melted under optimum conditions. All of the modified compositions were melted from batch at temperatures of  $2900^\circ\text{F}$  to  $3200^\circ\text{F}$  for periods of up to four hours.

The data mentioned earlier concerning melting and forming temperature studies of X-2124 is shown in Table III. It is evident that if X-2124 is melted under less than optimum conditions (assumed to be 24 hours at  $2900^\circ\text{F}$ ) its tensile strength and modulus properties will be similar to those of X-2252 through X-2260 and X-2275 through X-2278.

TABLE III  
THE EFFECT OF THERMAL TREATMENT ON THE  
TENSILE STRENGTH AND MODULUS OF X-2124

| BATCH MELTING<br>TIME and TEMP. | FORMING TEMP.<br>$^\circ\text{F}$ | FORMING TIME<br>HR. | T.S. $\times 10^3$ psi | E $\times 10^6$ psi |
|---------------------------------|-----------------------------------|---------------------|------------------------|---------------------|
| 4 Hr. $2900^\circ\text{F}$      | 2915                              | 1/2                 | 471                    | -                   |
| 4        2900                   | 2965                              | 2                   | 625                    | 12.7                |
| 4        3200                   | 2850                              | 2                   | 440                    | 12.5                |
| 4        3200                   | 2915                              | 1                   | 500                    | 11.8                |
| 4*       3200                   | 2915                              | 1                   | 500                    | 14.3                |
| 4        3200                   | 2975                              | 3                   | 540                    | -                   |
| 8 Hr. $2900^\circ\text{F}$      | 2890                              | 1                   | 453                    | -                   |
| 8        2900                   | 2915                              | 1/2                 | 532                    | 13.4                |
| 8        3200                   | 2915                              | 7                   | 535                    | -                   |
| 8        3200                   | 2965                              | 2                   | 525                    | 14.5                |
| 24 Hr. $2900^\circ\text{F}$     | 2900                              | 5                   | 700                    | 12.4                |
| 24       2900                   | 2915                              | 3                   | 681                    | 14.1                |
| 24*      2900                   | 2915                              | 3                   | 670                    | 14.4                |
| 24       2900                   | 2950                              | 1                   | 696                    | -                   |

\* Properties rechecked using a new batch of glass

It is apparent that the melting conditions used for  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO type glasses influence properties to a great extent, probably much more than small composition modifications or additions.

Before the X-2124 melting history study was completed, several oxide additions were made to this composition to determine their effect on strength, modulus and density. The resultant glasses are represented by X-2279 through X-2285 and X-2320 through X-2321. X-2279 through X-2283 contain  $\text{ZrO}_2$  or  $\text{ZrO}$  and were melted for four hours at 3200 F, melting conditions which are not optimum for glasses of the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO type. Therefore the tensile strengths are undoubtedly lower than could be obtained. The moduli of these glasses do not appear to have decreased as much as those for X-2124 melted under similar conditions. Their densities have increased, however, and would be expected to go higher with increasing oxide additions, thus preventing improved specific modulus values. Subsequent melts of these glasses at 2900 F for 24 hours definitely improved their properties. For instance, X-2281 melted under optimum conditions had an average tensile strength of 670,000 psi and an average modulus of  $14.4 \times 10^6$ . Earlier less optimum melts gave tensile strength properties of 582,000 psi and moduli of  $13.6 \times 10^6$  psi. Glasses X-2253, X-2257, and X-2260 showed similar improvements when melted under optimum conditions with the best properties being reported in Table II.

Glasses X-2284 and X-2285 contain MgO while glasses X-2320 and X-2321 contain  $\text{CeO}_2$ . These glasses were melted at near optimum conditions, 2900 F for 23 hours. At least one of these, X-2285, had excellent properties. Though its bulk density has increased to 2.5, its modulus has also increased. The  $\text{CeO}_2$  additions do not improve modulus and apparently lower the tensile strength. As expected,  $\text{CeO}_2$  also increases bulk density.

The properties of the best  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO glass, X-2124, and the best modified compositions, X-2285, are compared with the properties of AF-994 and M-glass (YM-31A) in Table IV.

TABLE IV  
PROPERTY COMPARISONS

| Glass  | T.S. $\times 10^3$ psi | E $\times 10^6$ psi | Density, g/cc | Specific Modulus |
|--------|------------------------|---------------------|---------------|------------------|
| AF-994 | 700                    | 12.4                | 2.49 (fiber)  | 4.98             |
| YM-31A | 500                    | 16.4                | 2.89 (fiber)  | 5.67             |
| X-2124 | 681                    | 14.1                | 2.41 (bulk)   | 5.85             |
| X-2285 | 715                    | 14.95               | 2.50 (bulk)   | 5.98             |

In order to obtain additional data on the properties of X-2124,

this glass was fiberized in a standard 52-hole laboratory bushing. AF-994 glass fibers were also produced from the same bushing in order to compare the properties of the two glasses when prepared under the same conditions.

X-2124 batch, prepared in the usual manner, was melted in platinum crucibles for 24 hours at 2900°F. The cullet produced from this operation was then fed into the 52-hole bushing. Forming temperatures of 2900°F to 2950°F were utilized to fiberize the glass. In this manner, approximately twenty-five (25) 1/4 lb. packages of forming strand were prepared. Owens-Corning size, P682, was applied to the glass strand during the forming operation.

Standard AF-994 batch was melted in platinum crucibles for 24 hours at 2900°F. The cullet produced from this operation was then fed into the 52-hole bushing. Forming temperatures of 2800°F to 2850°F were utilized to fiberize the glass. In this manner, approximately twenty-five (25) 1/4 lb. packages of forming strand were prepared. Owens-Corning size, P682, was applied to the glass strand during the forming operation.

No major problems were encountered in the forming operation. The glass fiberized well. Proper manipulation of forming temperature, glass head, and pulling speed resulted in an excellent operation. The only problems foreseen in a scale-up of the operation to a full-size 204-hole production unit involved the establishment of the proper process conditions so that the optimum properties are obtained in the glass fibers. As seen from the data below, neither the AF-994 glass nor the X-2124 glass fiberized in the 52-hole bushing had the virgin fiber properties that have been consistently achieved in a one-hole bushing for the X-2124 glass and in commercial production for the AF-994 glass.

| <u>Glass</u> | <u>Tensile<br/>Strength<br/>(psi)</u> | <u>Young's<br/>Modulus<br/>(psi)</u> |
|--------------|---------------------------------------|--------------------------------------|
| AF-994       | 478,000                               | $12.2 \times 10^6$                   |
| X-2124       | 443,000                               | $12.1 \times 10^6$                   |

Using the 1/4 lb. packages of 52-filament strand of both AF-994 and X-2124, the equivalent of single-end roving (204-filaments) was produced by the conventional roving process. Minor adjustments were necessary in taking the strand off the forming package because of its fewer number of filaments and non-standard package build.

Five single-end roving packages were used as the input material to filament wind flat laminates approximately 12" x 6" x 1/8". The resin system used was Epon 828, 10 phr phenyl glycidyl ether,

14 phr metaphenylenediamine (C1). Test specimens parallel to the fibers were cut from these laminates to determine comparative composite properties. Federal Test Method 406 was used in each instance:

|                              |             |
|------------------------------|-------------|
| Tensile Strength and Modulus | Method 1011 |
| Compressive Strength         | Method 1021 |
| Flexural Strength            | Method 1031 |

In the case of the tensile specimens, the test area was reduced to 1/4" width so as to minimize gripping problems. In the case of the flexural specimens, the length was reduced to 3-1/2" (from 4") to maximize the number of specimens attainable from each laminate.

The physical properties of each laminate are as follows:

|                  | <u>AF-994</u> | <u>X-2124</u> |
|------------------|---------------|---------------|
| Resin Content    | 18.0%         | 33.4%         |
| Specific Gravity | 2.03          | 1.72          |
| Barcol Hardness  | 75            | 67            |
| Thickness        | .123"         | .124"         |

The mechanical properties of each laminate are given in Table V. For purposes of data comparison, the properties of the AF-994 laminate have been reduced by the law of mixtures to calculated values for a resin content of 50.5% by volume equal to that of the X-2124 laminate.

The data of Table V indicates that at equal glass content, the X-2124 glass gives greater laminate strength and modulus than the AF-994 glass. In view of the fact that these data are not consistent with the virgin filament strengths reported above and are the result of a very limited number of tests, it is recommended that these data not be considered conclusive with respect to the comparative properties of AF-994 and X-2124 glass. A more extensive testing program should be undertaken to conclusively compare these two glasses. This is of major significance in view of the fact that AF-994 glass produced by a standard commercial process has a virgin filament strength of 665,000 psi and a modulus of  $12.4 \times 10^6$  psi and would give laminates of greater stiffness and strength than reported in Table V.

Several conclusions have been drawn from this study of glass compositions in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$  system.

1. Glasses with the best properties are located at  $\text{SiO}_2$  levels of 65 to 70 mole percent with  $\text{Al}_2\text{O}_3$  to BeO mole ratios of approximately 0.71.
2. Properties of glasses in this region can be improved to

TABLE V

LANINATE PROPERTIES FOR X-2124 AND AF-994 GLASSES  
(NORMALIZED AT A RESIN CONTENT OF 50.5% BY VOLUME)

| <u>Property</u>                             | <u>X-2124 Glass</u> | <u>AF-994 Glass</u> |
|---------------------------------------------|---------------------|---------------------|
| Tensile Strength (psi)                      | 188,000 (5)         | 159,000 (3)         |
| Tensile Modulus (psi $\times 10^{-6}$ )     | 6.66 (3)            | 5.82 (2)            |
| Compressive Strength (psi)                  | 50,200 (3)          | 40,300 (4)          |
| Compressive Modulus (psi $\times 10^{-6}$ ) | 6.72 (3)            | 6.29 (2)            |
| Flexural Strength (psi)                     | 180,000 (5)         | 116,000 (5)         |
| Flexural Modulus (psi $\times 10^{-6}$ )    | 5.70 (5)            | 5.92 (5)            |

( ) Refers to number of specimens tested

some extent by the proper addition of a fourth oxide, the best being MgO. This will result, however, in a density increase.

3. In general, glasses based on the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-BeO}$  system must be melted from batch at approximately  $2900^\circ\text{F}$  for 24 hours to obtain optimum strength and modulus properties.

#### c. $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ System

This system has continually been of importance because of its potential properties, as shown most notably by AF-994. Work on the system during the previous year has been characterized by the study of properties as influenced by oxide additions, by major changes in the  $\text{SiO}_2$  content coupled with large variations in  $\text{Al}_2\text{O}_3$  to MgO mole ratios, and by changes in melting and forming conditions.

The list of compositions studied and their properties are shown in Table VI. Figure 6 is a portion of the diagram of the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  system showing the location of all three-component<sup>2</sup> glasses studied in the system.

Initial studies dealt with the addition of various oxides to one or more  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  glasses for the purpose of studying property variations. Based on the results obtained with X-1857, a glass containing small amounts of  $\text{ZrO}_2$ , X-2118, X-2119, and X-2120 were melted and fiberized. X-2118 itself was further modified with CaO,  $\text{TiO}_2$ , and/or  $\text{Li}_2\text{O}$  to yield X-2121, X-2122, and X-2123.

Based on the modulus of X-1857, further  $\text{ZrO}_2$  additions had little or no effect on modulus of elasticity. It appears, however, that in some cases quite a large effect is exerted on tensile strength (see X-2118 and X-2119).  $\text{ZrO}_2$  does increase overall bulk density when used in a glass as shown by the density of X-2119.

As for the additional use of CaO,  $\text{Li}_2\text{O}$ , and/or  $\text{TiO}_2$  in any of these glasses, no beneficial effects would be expected. This is based on the results shown for X-2121, X-2122, and X-2123. Tensile strengths are definitely lowered and moduli are not beneficially affected.

X-2152 through X-2166 represent modifications of the general compositional area in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  system previously found to have maximum tensile strength and modulus properties, densities less than 2.5 g/cc, and the best fiberization properties.  $\text{Li}_2\text{O}$  in particular has been utilized to check its effect on properties, especially density. X-2152 through X-2155 represent a series of such compositions with decreasing mole percentages of  $\text{SiO}_2$ . At the same time, the MgO to  $\text{Al}_2\text{O}_3$  content

TABLE VI

SiO<sub>2</sub> - Al<sub>2</sub>O<sub>3</sub> - MgO System and Modifications

| Glass No. | Oxides (a. weight %, b. mole %) |                                |      |      |                   | Average Tensile Strength x 10 <sup>3</sup> psi |  | Modulus of Elasticity x 10 <sup>6</sup> psi |  | Average Diameter x 10 <sup>-5</sup> in. |      | Forming Temp. °F |      | N <sub>2</sub> of Fibers |  | Bulk Density g/cc |  |
|-----------|---------------------------------|--------------------------------|------|------|-------------------|------------------------------------------------|--|---------------------------------------------|--|-----------------------------------------|------|------------------|------|--------------------------|--|-------------------|--|
|           | SiO <sub>2</sub>                | Al <sub>2</sub> O <sub>3</sub> | MgO  | CaO  | Li <sub>2</sub> O | ZrO <sub>2</sub>                               |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-1857    | a. 62.0                         | 25.0                           | 10.0 |      |                   | 3.0                                            |  |                                             |  | 511                                     | 13.5 | 31               | 2770 | 17                       |  | -                 |  |
|           | b. 66.6                         | 15.8                           | 16.0 |      |                   | 1.5                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2118    | 60.0                            | 25.0                           | 10.0 |      |                   | 5.0                                            |  |                                             |  | 745                                     | 13.2 | 19               | 2795 | 16                       |  | -                 |  |
|           | 65.2                            | 16.0                           | 16.2 |      |                   | 2.6                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2119    | 58.0                            | 25.0                           | 10.0 |      |                   | 7.0                                            |  |                                             |  | 777                                     | 13.0 | 20               | 2795 | 15                       |  | 2.62              |  |
|           | 63.7                            | 16.2                           | 16.4 |      |                   | 3.7                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2120    | 57.0                            | 30.0                           | 10.0 |      |                   | 3.0                                            |  |                                             |  | 616                                     | 13.1 | 22               | 2850 | 15                       |  | -                 |  |
|           | 62.6                            | 19.4                           | 16.4 | 3    |                   | 1.6                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2121    | 60.0                            | 15.0                           | 10.0 | 10.0 |                   | 5.0                                            |  |                                             |  | 436                                     | 12.8 | 40               | 2680 | 18                       |  | -                 |  |
|           | 61.9                            | 9.1                            | 15.4 | 11.1 |                   | 2.5                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2122*   | 58.0                            | 24.1                           | 9.6  |      |                   | 4.7                                            |  |                                             |  | 475                                     | 12.1 | 47               | 2735 | 16                       |  | -                 |  |
|           | 63.3                            | 15.5                           | 15.7 |      |                   | 2.5                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2123    | 56.3                            | 23.4                           | 9.4  | 4.0  | 2.1               | 4.9                                            |  |                                             |  | 426                                     | 13.0 | 35               | 2620 | 15                       |  | -                 |  |
|           | 59.3                            | 14.5                           | 14.7 | 4.5  | 4.5               | 2.5                                            |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2152    | 68.4                            | 24.9                           | 4.9  |      | 1.8               |                                                |  |                                             |  | 712                                     | 12.8 | -                | -    | -                        |  | 2.41              |  |
|           | 72.7                            | 15.6                           | 7.8  |      | 3.9               |                                                |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |
| X-2       | 66.9                            | 24.3                           | 6.4  |      | 2.4               |                                                |  |                                             |  | 671                                     | 13.0 | 35               | 2850 | 15                       |  | 2.47              |  |
|           | 70.0                            | 15.0                           | 10.0 |      | 5.0               |                                                |  |                                             |  |                                         |      |                  |      |                          |  |                   |  |

\*Balance is TiO<sub>2</sub>

TABLE VI (Continued)

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO  | Li <sub>2</sub> O | ZrO <sub>2</sub> | Ta <sub>2</sub> O <sub>5</sub> | Average Tensile Strength x 10 <sup>3</sup> psi | Modulus of Elasticity x 10 <sup>6</sup> psi | Average Diameter x 10 <sup>-5</sup> in. | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------|------------------|--------------------------------|------|-------------------|------------------|--------------------------------|------------------------------------------------|---------------------------------------------|-----------------------------------------|------------------|---------------|-------------------|
| X-2154    | 67.5             | 24.5                           | 4.8  | 3.2               |                  |                                | 671                                            | 13.0                                        | 27.0                                    | 2850             | 15            | 2.47              |
|           | 70.0             | 15.0                           | 5.0  | 10.0              |                  |                                |                                                |                                             |                                         |                  |               |                   |
| X-2155    | 61.7             | 28.2                           | 7.4  | 2.7               |                  |                                | 687                                            | 13.0                                        | 30.0                                    | 2800             | 15            | 2.54              |
|           | 65.0             | 17.5                           | 11.7 | 5.8               |                  |                                |                                                |                                             |                                         |                  |               |                   |
| X-2156    | 65.3             | 24.1                           | 6.5  | 2.3               | 1.9              |                                | 678                                            | 12.5                                        | 37.0                                    | 2850             | 15            | 2.5               |
|           | 69.0             | 15.0                           | 10.0 | 5.0               | 1.0              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2157    | 64.3             | 23.3                           | 3.2  | 4.6               | 4.6              |                                | 645                                            | 11.9                                        | 27.0                                    | 2850             | 15            | 2.49              |
|           | 68.3             | 14.6                           | 4.9  | 9.8               | 2.4              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2158    | 61.4             | 22.3                           | 3.0  | 4.3               | 9.0              |                                | 430                                            | 11.2                                        | 33.0                                    | 2915             | 15            | 2.49              |
|           | 66.7             | 14.3                           | 4.8  | 9.5               | 4.8              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2159    | 61.3             | 29.8                           | 2.9  | 4.3               | 1.7              |                                | 660                                            | 11.8                                        | 30.0                                    | 2915             | 15            | 2.46              |
|           | 66.0             | 18.9                           | 4.7  | 9.4               | 0.9              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2160    | 57.0             | 34.6                           | 2.7  | 4.0               | 1.7              |                                | 677                                            | 11.6                                        | 24.0                                    | 2915             | 15            | 2.5               |
|           | 63.0             | 22.5                           | 4.5  | 9.0               | 0.9              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2161    | 65.8             | 24.3                           | 3.2  | 4.7               | 2.0              |                                | 741                                            | 11.7                                        | 22.0                                    | 2850             | 15            | 2.44              |
|           | 69.0             | 15.0                           | 5.0  | 10.0              | 1.0              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2162    | 65.9             | 24.3                           | 2.6  | 5.2               | 2.0              |                                | 710                                            | 11.5                                        | 23.0                                    | 2915             | 15            | 2.48              |
|           | 69.0             | 15.0                           | 4.0  | 11.0              | 1.0              |                                |                                                |                                             |                                         |                  |               |                   |
| X-2163    | 66.7             | 24.4                           | 1.9  | 5.7               | 2.0              |                                | 671                                            | 11.0                                        | 28.0                                    | 2915             | 14            | 2.39              |
|           | 69.0             | 15.0                           | 3.0  | 12.0              | 1.0              |                                |                                                |                                             |                                         |                  |               |                   |

**TABLE VI (Continued)**

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO  | Li <sub>2</sub> O | ZrO <sub>2</sub> | Ta <sub>2</sub> O <sub>5</sub> | Average Modulus                                |                                  | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |      |
|-----------|------------------|--------------------------------|------|-------------------|------------------|--------------------------------|------------------------------------------------|----------------------------------|------------------|---------------|-------------------|------|
|           |                  |                                |      |                   |                  |                                | Average Tensile Strength x 10 <sup>3</sup> psi | Elasticity x 10 <sup>6</sup> psi |                  |               |                   |      |
| X-2164    | 60.1             | 27.9                           | 7.4  | 2.7               | 1.9              |                                | 530                                            | 11.8                             | 36.0             | 2800          | 15                | 2.5  |
|           | 64.0             | 17.5                           | 11.7 | 5.8               | 1.0              |                                |                                                |                                  |                  |               |                   |      |
| X-2165    | 65.6             | 24.2                           | 4.5  | 3.8               | 1.9              |                                | 720                                            | 11.5                             | 23.0             | 2795          | 14                | 2.51 |
|           | 69.0             | 15.0                           | 7.0  | 8.0               | 1.0              |                                |                                                |                                  |                  |               |                   |      |
| X-2166    | 65.0             | 23.8                           | 3.2  | 4.6               |                  | 3.4                            | 688                                            | 11.5                             | 26.0             | 2850          | 15                | 2.45 |
|           | 69.5             | 15.0                           | 5.0  | 10.0              |                  | 0.5                            |                                                |                                  |                  |               |                   |      |
| X-2262    | 60.0             | 35.0                           | 5.0  |                   |                  |                                | 561                                            | 12.5                             | 48               | 3190          | 15                | 2.5  |
|           | 68.1             | 23.4                           | 8.5  |                   |                  |                                |                                                |                                  |                  |               |                   |      |
| X-2263    | 55.0             | 35.0                           | 10.0 |                   |                  |                                | 644                                            | 13.6                             | 35               | 2955          | 15                | 2.6  |
|           | 60.8             | 22.8                           | 16.5 |                   |                  |                                |                                                |                                  |                  |               |                   |      |
| X-2264    | 55.0             | 30.0                           | 15.0 |                   |                  |                                | 704                                            | 12.9                             | 22               | 2735          | 18                | 2.6  |
|           | 57.9             | 18.6                           | 23.5 |                   |                  |                                |                                                |                                  |                  |               |                   |      |
| X-2265    | 55.0             | 25.0                           | 20.0 |                   |                  |                                | 389                                            | -                                | 39               | 2575          | 15                | 2.6  |
|           | 55.2             | 14.8                           | 30.0 |                   |                  |                                |                                                |                                  |                  |               |                   |      |
| X-2266    | 65.0             | 15.0                           | 20.0 |                   |                  |                                | 486                                            | 11.8                             | 31               | 2735          | 16                | 2.6  |
|           | 62.7             | 8.5                            | 28.8 |                   |                  |                                |                                                |                                  |                  |               |                   |      |
| X-2267    | 51.3             | 34.9                           | 13.8 |                   |                  |                                | 578                                            | 13.1                             | 40               | 2760          | 15                | 2.6  |
|           | 55.5             | 22.2                           | 22.3 |                   |                  |                                |                                                |                                  |                  |               |                   |      |
| X-2268    | 50.0             | 40.0                           | 10.0 |                   |                  |                                | Could not be fiberized                         |                                  |                  |               |                   |      |
|           | 56.5             | 26.6                           | 16.9 |                   |                  |                                |                                                |                                  |                  |               |                   |      |

TABLE VI (Continued)

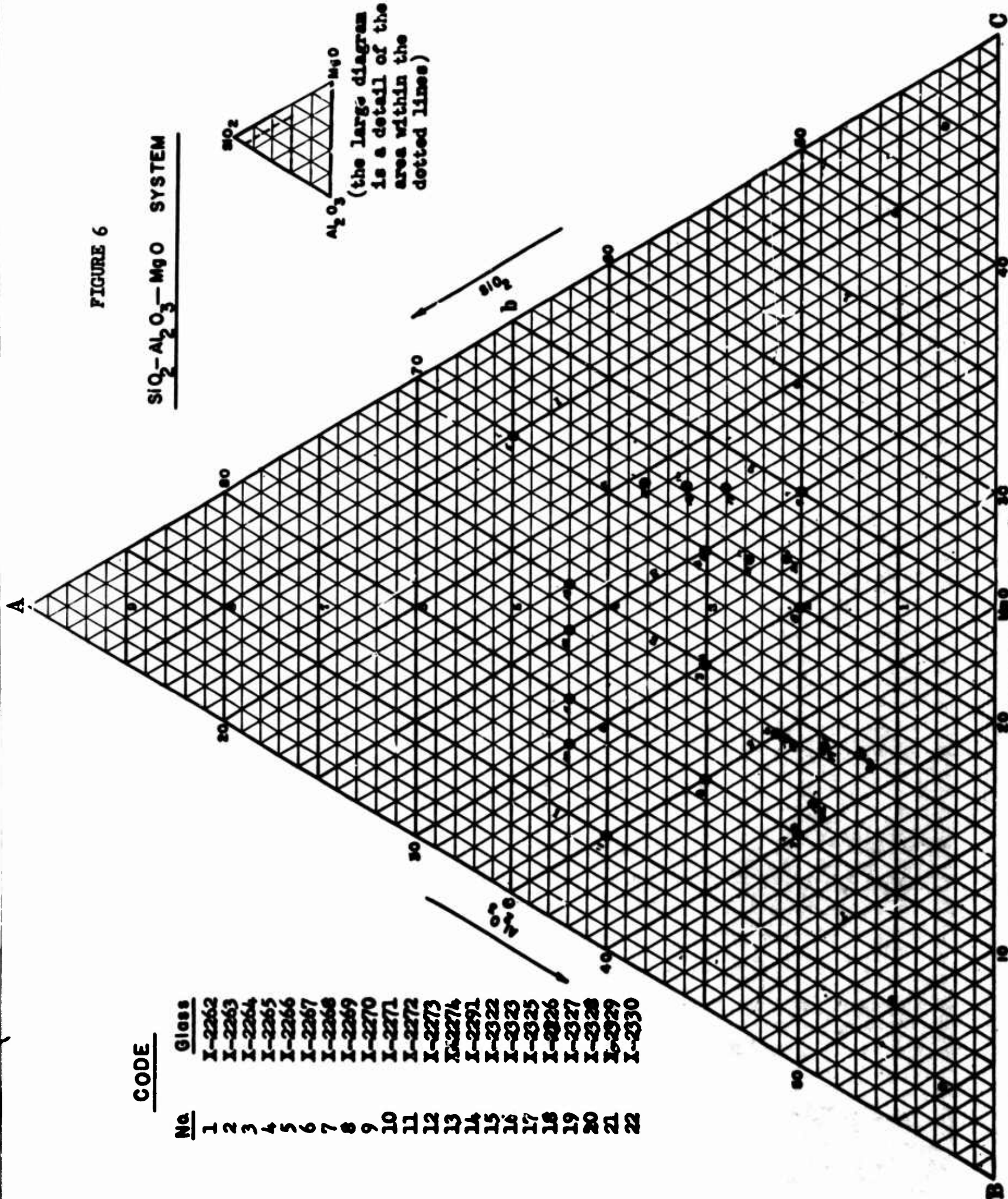
| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO            | Li <sub>2</sub> O | ZrO <sub>2</sub> | Ta <sub>2</sub> O <sub>5</sub> | Average Tensile Strength x 10 <sup>3</sup> psi | Modulus of Elasticity x 10 <sup>6</sup> psi | Average Diameter x 10 <sup>-5</sup> in. | Forming Temp. °F | No. of Fibers | Bulk Density g/cc |
|-----------|------------------|--------------------------------|----------------|-------------------|------------------|--------------------------------|------------------------------------------------|---------------------------------------------|-----------------------------------------|------------------|---------------|-------------------|
| X-2269    | 50.0<br>51.3     | 30.0<br>18.1                   | 20.0<br>30.6   |                   |                  |                                | 497                                            | 13.4                                        | 48                                      | 2560             | 13            | 2.7               |
| X-2270    | 50.0<br>49.0     | 25.0<br>14.4                   | 25.0<br>36.6   |                   |                  |                                | 377                                            | 13.3                                        | 51                                      | 2445             | 15            | 2.7               |
| X-2271    | 62.0<br>67.7     | 30.0<br>19.3                   | 8.0<br>13.0    |                   |                  |                                | 603                                            | 13.5                                        | 46                                      | 2915             | 15            | 2.6               |
| X-2272    | 62.0<br>66.4     | 28.0<br>17.7                   | 10.0<br>16.0   |                   |                  |                                | 845                                            | 12.2                                        | 17                                      | 2850             | 15            | 2.5               |
| X-2273    | 62.0<br>64.5     | 25.0<br>15.3                   | 13.0<br>20.2   |                   |                  |                                | 703                                            | 11.9                                        | 19                                      | 2750             | 15            | 2.5               |
| X-2274    | 62.0<br>63.3     | 23.0<br>13.8                   | 15.0<br>22.8   |                   |                  |                                | 727                                            | 12.0                                        | 22                                      | 2700             | 15            | 2.6               |
| X-2291    | 58.06<br>56.99   | 20.74<br>11.99                 | 21.19<br>31.02 |                   |                  |                                | 729                                            | 13.1                                        | 23                                      | 2600             | 17            | 2.6               |
| X-2322    | 52.68<br>53.00   | 26.43<br>15.67                 | 20.88<br>31.33 |                   |                  |                                | 543                                            | 13.4                                        | 40                                      | 2600             | 15            | 2.6               |
| X-2323    | 56.1<br>55.0     | 21.7<br>12.5                   | 22.2<br>32.5   |                   |                  |                                | 660                                            | 13.4                                        | 35                                      | 2515             | 15            | 2.68              |
| X-2325    | 54.05<br>53.00   | 22.83<br>13.19                 | 23.12<br>33.81 |                   |                  |                                | 466                                            | 13.2                                        | 38                                      | 2450             | 15            | 2.64              |

TABLE VI (Continued)

| Glass No. | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | MgO           | Li <sub>2</sub> O | ZrO <sub>2</sub> | Ta <sub>2</sub> O <sub>5</sub> | Average<br>Tensile<br>Strength<br>x 10 <sup>3</sup> psi | Modulus<br>of<br>Elasticity<br>x 10 <sup>6</sup> psi | Average<br>Diameter<br>x 10 <sup>-5</sup> in. | Forming<br>Temp.<br>°F | No. of<br>Fibers | Bulk<br>Density<br>g/cc |
|-----------|------------------|--------------------------------|---------------|-------------------|------------------|--------------------------------|---------------------------------------------------------|------------------------------------------------------|-----------------------------------------------|------------------------|------------------|-------------------------|
| X-2326    | 50.8<br>55.0     | 35.3<br>22.5                   | 13.9<br>22.5  |                   |                  |                                | 614                                                     | 13.6                                                 | 39                                            | 2750                   | 15               | 2.6                     |
| X-2327    | 48.8<br>53.0     | 36.7<br>23.5                   | 14.5<br>23.5  |                   |                  |                                | 698                                                     | 13.5                                                 | 27                                            | 2740                   | 15               | 2.65                    |
| X-2328    | 46.8<br>51.0     | 38.1<br>24.5                   | 15.1<br>24.5  |                   |                  |                                | 569                                                     | 13.6                                                 | 48                                            | 2835                   | 15               | 2.67                    |
| X-2329    | 50.7<br>51.0     | 27.5<br>16.33                  | 21.8<br>32.67 |                   |                  |                                | 671                                                     | 14.0                                                 | 30                                            | 2670                   | 15               | 2.63                    |
| X-2330    | 49.3<br>55.0     | 39.0<br>25.6                   | 11.7<br>19.4  |                   |                  |                                | 432                                                     | 13.5                                                 | 61                                            | 3015                   | 5                | 2.68                    |

FIGURE 6

$\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  SYSTEM



CODE

| No. | Glass  |
|-----|--------|
| 1   | I-2262 |
| 2   | I-2263 |
| 3   | I-2264 |
| 4   | I-2265 |
| 5   | I-2266 |
| 6   | I-2267 |
| 7   | I-2268 |
| 8   | I-2269 |
| 9   | I-2270 |
| 10  | I-2271 |
| 11  | I-2272 |
| 12  | I-2273 |
| 13  | I-2274 |
| 14  | I-2291 |
| 15  | I-2322 |
| 16  | I-2323 |
| 17  | I-2325 |
| 18  | I-2326 |
| 19  | I-2327 |
| 20  | I-2328 |
| 21  | I-2329 |
| 22  | I-2330 |

has been varied. As shown by the data, small amounts of  $\text{Li}_2\text{O}$  can produce densities as low as 2.41 g/cc. In this series of four glasses, a reduction in the  $\text{SiO}_2$  content causes an increase in the density and the modulus of elasticity. Therefore, the desirable qualities of both low density and high modulus cannot be obtained in this series of compositions. Nevertheless, glasses represented by X-2152, X-2154, and X-2155 are obtainable with specific moduli which are greater than that of AF-994.

X-2156 through X-2165 represent compositions containing  $\text{ZrO}_2$ , in addition to  $\text{Li}_2\text{O}$ . Again,  $\text{ZrO}_2$  was added because of its reported effect on modulus. X-2166 contains  $\text{Ta}_2\text{O}_5$  for the same reason. The results indicate, however, that neither addition improves the modulus. In fact, in all cases the modulus values are lower than those found for X-2152 through X-2155. The tensile strength figures for this series, for the most part, are relatively good, especially in the case of X-2161, X-2162 and X-2165.

It is concluded that several oxide additions can be made to the basic  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  system to obtain one specific result. However, attempts to lower density while improving modulus and tensile strength are unlikely to meet with success.

The glasses designated X-2262 through X-2270 were selected from compositional areas normally not associated with optimum forming characteristics. It was felt that studies of such areas might reveal improved fiber properties which had previously been overlooked. Note that most of the compositions contain less than 60 mole percent  $\text{SiO}_2$ . Those above 60 mole percent  $\text{SiO}_2$  have  $\text{Al}_2\text{O}_3$  to  $\text{MgO}$  mole ratios well below or well above 1.0. None of the glasses have noteworthy properties. Though one or two may have good strength or modulus properties, all have high densities. This is very evident for those glasses with less than 56 mole percent  $\text{SiO}_2$ . Such densities, of course, reduce the final specific strength and specific modulus of the glasses in question.

Glasses designated X-2271 through X-2274 show the influence of an increase in the  $\text{SiO}_2$  content. The strength has improved and the density has begun to drop.

The remaining compositions fiberized in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  system are shown in Table VI (X-2322 through X-2330). These were based on the results of work performed under the section of this report titled "Relationship Between Composition and Properties of Selected Glasses." Data for the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  system is summarized in Figure 7, Modulus vs.  $2\text{Al/MgO}$ , which indicates that modulus increases as the  $\text{SiO}_2$  content decreases and, is a maximum at either very high or very low  $\text{Al}_2\text{O}_3$  to  $\text{MgO}$  mole ratios.

Figure 7 was used, therefore, to design glasses having high

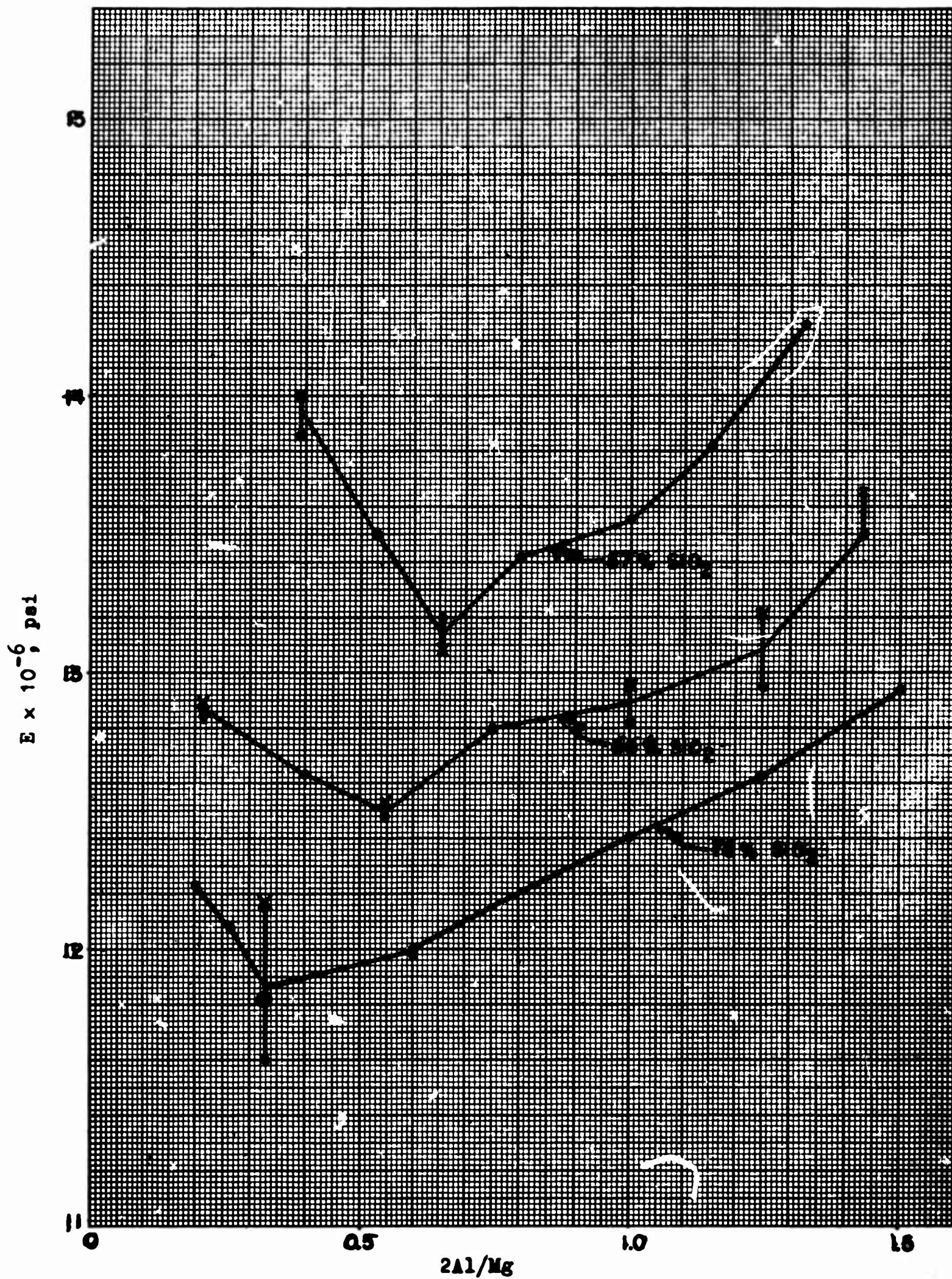


Figure 7 Modulus vs. 2Al/Mg Ratio

moduli of elasticity. In general, as demonstrated previously, decreases in  $\text{SiO}_2$  content improved modulus at a given  $\text{Al}_2\text{O}_3$  to  $\text{MgO}$  mole ratio. In general, this also produced an increase in density. The relatively high tensile strengths of the series represented by X-2322 through X-2330 were obtained through careful melting history changes and fiberization techniques. At such low  $\text{SiO}_2$  contents the glass viscosity was relatively low, which necessitated fiberization in bushings with small tips (0.040" to 0.050"). Normal fiberization techniques would have produced rather low strength fibers and in some cases the glasses could not have been fiberized at all. In conclusion, it appears that thermal history and fiberization technique is quite important with glasses of this nature.

A study of the tensile strength and modulus of AF-994 glass as affected by thermal history and fiberization technique (forming temperature, forming time, and orifice size) was made to complete the investigation of the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{MgO}$  system. It was felt that a study of this nature, taken in the context of the overall investigation, would aid in selecting optimum forming conditions for most of the glasses based on  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{MgO}$  or  $\text{R}_x\text{O}$  substitutions for  $\text{MgO}$ .

The program was carried out as follows:

1. AF-994, melted from batch at  $2900^\circ\text{F}$  for four hours, was fiberized in one of three one-hole bushings having hole sizes of 0.062", 0.050" and 0.040" respectively.
2. The glass was fiberized at temperatures as low as possible and temperature was increased in increments of  $25^\circ$  to a maximum of  $3000^\circ\text{F}$ .
3. The glass was fiberized for a period of eight hours at each temperature and fiber samples for tensile strength measurements were taken hourly.
4. Fiber samples for modulus of elasticity measurements were taken every two hours at each forming temperature over the eight-hour period.
5. The average fiber diameter for all samples was held between 0.00025" and 0.00035". Glass level was held relatively constant at 1.5" to 2.0".

The results obtained in this study are shown in Table VII. Both the tensile strength and modulus figures shown are the maximum values obtained over the eight-hour period. The sub-number in parenthesis is the number of hours the glass was fiberized at the given forming temperature before its maximum strength was obtained.

The tensile strength values show a dependence on the length of time the glass has been in the bushing. Most of the runs reached

TABLE VII  
Effect of Forming Temperature and Orifice Size on  
AF-994 Tensile Strength and Modulus of Elasticity

| Forming<br>Temp.<br>°F           | <u>2725</u> | <u>2750</u> | <u>2775</u> | <u>2800</u> | <u>2825</u> | <u>2850</u> | <u>2875</u> | <u>2900</u> | <u>2925</u> | <u>2950</u> | <u>2975</u> | <u>3000</u> |
|----------------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Orifice Size<br>= 0.040"         |             |             |             |             |             |             |             |             |             |             |             |             |
| <u>T.S. x 10<sup>3</sup> psi</u> | -           | -           | -           | 811(6)*     | 785(7)      | 660(3)      | 757(3)      | 654(2)      | 779(3)      | 700(4)      | 728(7)      | 740(7)      |
| <u>E x 10<sup>6</sup> psi</u>    | -           | -           | -           | 12.4        | 13.7        | 13.2        | 13.3        | 13.1        | 13.5        | 13.2        | 12.8        | 12.6        |
| Orifice Size<br>= 0.050"         |             |             |             |             |             |             |             |             |             |             |             |             |
| <u>T.S. x 10<sup>3</sup> psi</u> | -           | 756(6)      | 800(6)      | 791(2)      | 780(3)      | 801(7)      | 809(6)      | 790(2)      | 768(6)      | 769(7)      | 732(4)      | 704(7)      |
| <u>E x 10<sup>6</sup> psi</u>    | -           | 12.9        | 13.8        | 13.3        | 13.3        | 13.3        | 13.1        | 13.7        | 13.5        | 13.0        | 12.7        | 12.1        |
| Orifice Size<br>= 0.062"         |             |             |             |             |             |             |             |             |             |             |             |             |
| <u>T.S. x 10<sup>3</sup> psi</u> | 721<br>(4)  | 734(8)      | 768(6)      | 750(7)      | 753(8)      | 791(5)      | 786(7)      | 781(8)      | 763(2)      | 790(7)      | 760(3)      | 740(7)      |
| <u>E x 10<sup>6</sup> psi</u>    | 12.7        | 13.4        | 13.0        | 13.1        | 12.7        | 12.7        | 12.9        | 12.9        | 13.0        | 13.7        | -           | 13.1        |

\*No. of hours required to reach given strength

maximum tensile strength values in four to seven hours. It should also be noted that the highest strengths are not obtained until the forming temperature has reached 2775° F or higher. Apparently, forming temperatures above 2900° F do not produce increasingly higher tensile strengths.

There is some indication that the orifice size influences strength to a small extent. This might be a thermal history effect caused by a different quench rate for the smaller orifice. Once the forming temperature reaches 2850° F, however, there is no noticeable difference in strength. (These statements apply to comparisons between fibers from the 0.050" and 0.062" orifice. Fiber strengths from the 0.040" orifice are rather variable and were probably adversely influenced by interactions of the fiber cone and the edge of the small orifice).

The modulus does not appear to be materially affected by forming temperature, forming time or orifice diameter.

Several conclusions have been drawn from this rather extensive study of glass compositions in the  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{MgO}$  system.

1. Glasses with the best combination of tensile strength, modulus of elasticity, density, and forming characteristics are located at an approximate  $\text{Al}_2\text{O}_3$  to  $\text{MgO}$  mole ratio of 1.0 and at a  $\text{SiO}_2$  level ranging from 60 to 70% by weight.
2. The modulus of elasticity of these glasses can be increased by lowering the  $\text{SiO}_2$  content. However, this will also cause an increase in the glass density. The density can be controlled to some extent by using small amounts of  $\text{Li}_2\text{O}$ .
3. Small  $\text{ZrO}_2$  additions increase tensile strength under certain conditions, but glass density increases.
4. Compositions with less than 56 mole percent  $\text{SiO}_2$  have densities greatly in excess of 2.6 g/cc.
5. If low density is not desired, glasses can be fiberized with moduli of  $13 - 14 \times 10^6$  psi and strengths approaching 700,000 psi.

#### d. $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{Li}_2\text{O}$ System

The  $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{Li}_2\text{O}$  system was investigated rather extensively under the first year's effort and these results were reported in Technical Report AFML-TR-65-90, Part I. However, in order to investigate compositions outside optimum fiberization areas, eight additional glasses were subsequently prepared.

The compositions (X-2172, X-2245 through X-2251) and their properties are shown in Table VIII under the heading " $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ - $\text{Li}_2\text{O}$  System." The position of each composition is plotted on the three-phase diagram, Figure 8.

TABLE VIII

SiO<sub>2</sub> - Al<sub>2</sub>O<sub>3</sub> - Li<sub>2</sub>O System

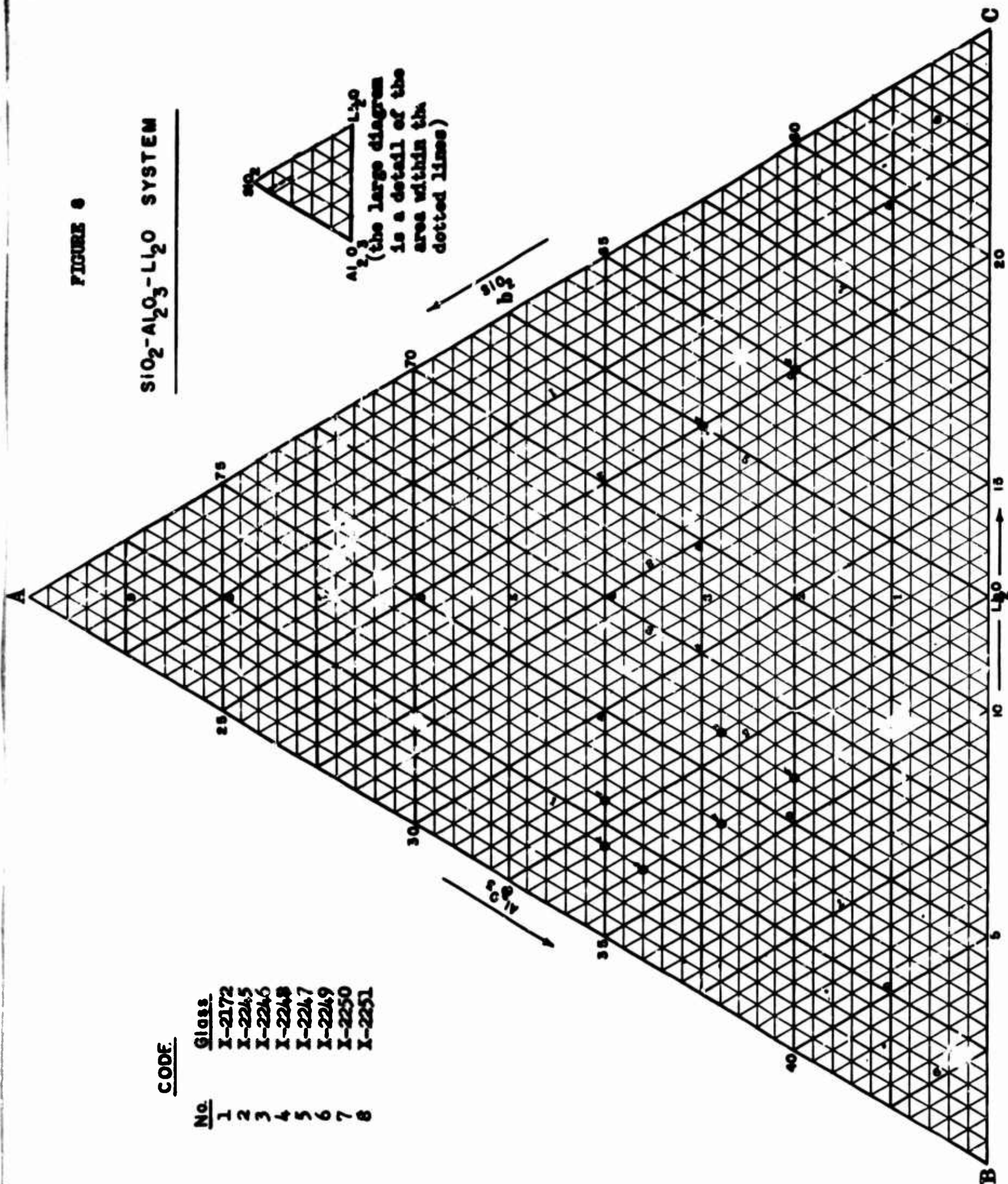
| Glass No. | Oxides<br>(a. weight %, b. mole %) |                                | Average<br>Tensile<br>Strength<br>$\times 10^3$ psi | Modulus<br>of<br>Elasticity<br>$\times 10^6$ psi | Average<br>Diameter<br>$\times 10^{-5}$ in. | Forming<br>Temp.<br>°F | No. of<br>Fibers | Bulk<br>Density<br>g/cc |
|-----------|------------------------------------|--------------------------------|-----------------------------------------------------|--------------------------------------------------|---------------------------------------------|------------------------|------------------|-------------------------|
|           | SiO <sub>2</sub>                   | Al <sub>2</sub> O <sub>3</sub> |                                                     |                                                  |                                             |                        |                  |                         |
| X-2172    | a. 65.0                            | 32.0                           | 698                                                 | 11.4                                             | 31                                          | 3115                   | 16               | 2.4                     |
|           | b. 72.3                            | 21.0                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2245    | 65.0                               | 33.0                           | 455                                                 | 10.6                                             | 32                                          | 3215                   | 15               | 2.3                     |
|           | 73.5                               | 22.0                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2246    | 64.0                               | 34.0                           | 372                                                 | 11.8                                             | 67                                          | 3190                   | 15               | 2.45                    |
|           | 72.7                               | 22.7                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2247    | 60.0                               | 34.0                           | 522                                                 | 11.2                                             | 26                                          | 2800                   | 15               | 2.4                     |
|           | 65.1                               | 21.7                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2248    | 62.0                               | 34.0                           | 655                                                 | 11.1                                             | 37                                          | 3065                   | 18               | 2.3                     |
|           | 68.8                               | 22.2                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2249    | 62.0                               | 32.0                           | 575                                                 | 10.7                                             | 17                                          | 2830                   | 15               | 2.4                     |
|           | 66.7                               | 20.3                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2250    | 62.5                               | 25.0                           | 386                                                 | 9.6                                              | 22                                          | 2390                   | 15               | 2.4                     |
|           | 61.0                               | 14.4                           |                                                     |                                                  |                                             |                        |                  |                         |
| X-2251    | 60.0                               | 25.0                           | 315                                                 | 10.1                                             | 33                                          | 2285                   | 15               | 2.4                     |
|           | 57.2                               | 14.0                           |                                                     |                                                  |                                             |                        |                  |                         |

FIGURE 8

SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Li<sub>2</sub>O SYSTEM

CODE

| No. | Glass  |
|-----|--------|
| 1   | I-2172 |
| 2   | I-2245 |
| 3   | I-2246 |
| 4   | I-2248 |
| 5   | I-2247 |
| 6   | I-2249 |
| 7   | I-2250 |
| 8   | I-2251 |



The bulk densities of these glasses were low, as expected.

Several had tensile strengths approaching 700,000 psi. Modulus values did not exceed  $12 \times 10^6$  psi. The higher strength glasses did not correspond with the higher modulus glasses.

All efforts with the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Li}_2\text{O}$  glasses continued to indicate that glasses with the best properties in the system contain approximately 65% by weight  $\text{SiO}_2$  and 3 to 5 weight percent  $\text{Li}_2\text{O}$ . All other compositions investigated failed to produce better properties, no matter what the fiberization parameters. The system is capable of producing low density glasses with good tensile strength properties ( $\approx 700,000$  psi), but only moderate modulus of elasticity properties ( $11\text{-}12 \times 10^6$  psi).

#### c. High Modulus Glasses

A series of compositions were prepared strictly on the basis of their high modulus properties. Low density and high strength were not initially considered. Lowenstein's(2) study of high modulus compositions was used as a guide.  $\text{BeO}$  was used in only one case to determine its effect on final properties.

The compositions studied and their properties are shown in Table IX titled "Compositions Prepared For High Modulus Studies."

As suggested by Lowenstein, initial studies of possible high modulus glasses started with those having a low content of network formers and a high content of network modifying ions. X-2066 through X-2069 were glasses of this type. X-2067 was found to have the best fiberization properties in the series. Various modifications of X-2067 were then made, an increase in the  $\text{Al}_2\text{O}_3$  content being most beneficial in increasing the modulus, as shown by X-2070. X-2073 and X-2117 represent higher and higher  $\text{Al}_2\text{O}_3$  contents with corresponding increases in the modulus. This characteristic of  $\text{Al}_2\text{O}_3$  was unexpected since it is classified as a network former, in conjunction with  $\text{SiO}_2$ , in Lowenstein's study. There also appeared to be some improvement in tensile strength with the increase in  $\text{Al}_2\text{O}_3$  content.

At this point X-2117 had the best modulus. X-2130 and X-2131 represent modifications of X-2117 and contain more  $\text{Al}_2\text{O}_3$  and changes in the  $\text{MgO}$  to  $\text{CaO}$  mole ratio. X-2131 possessed a good modulus and, unexpectedly, a high tensile strength.

The remaining glasses are modifications of X-2131. The modifications consist primarily of changes in the  $\text{MgO}$  to  $\text{CaO}$  mole ratio. X-2134 had the best modulus of all glasses studied and, again unexpectedly, a good tensile strength.

It appears that the best range of components for this particular

TABLE IX  
Compositions Prepared For High Modulus Sandies

| Glass No. | Oxides (a. weight %, b. mole %) |                                |      |      |                  |                  |                  |                   |     |  | Modulus<br>of<br>Elasticity<br>x 10 <sup>6</sup> psi | Average<br>Tensile<br>Strength<br>x 10 <sup>3</sup> psi | Forming<br>Temp.<br>°F | Bulk<br>Density<br>g/cc |
|-----------|---------------------------------|--------------------------------|------|------|------------------|------------------|------------------|-------------------|-----|--|------------------------------------------------------|---------------------------------------------------------|------------------------|-------------------------|
|           | SiO <sub>2</sub>                | Al <sub>2</sub> O <sub>3</sub> | CaO  | MgO  | TiO <sub>2</sub> | ZrO <sub>2</sub> | CeO <sub>2</sub> | Li <sub>2</sub> O | BeO |  |                                                      |                                                         |                        |                         |
| X-2066    | a. 47.9                         | 10.6                           | 10.6 | 10.6 | 16.0             | 4.3              |                  |                   |     |  | 13.1                                                 | 335                                                     | 2365                   | -                       |
|           | b. 50.2                         | 6.5                            | 11.9 | 16.6 | 12.6             | 2.2              |                  |                   |     |  |                                                      |                                                         |                        |                         |
| X-2067    | 45.0                            | 10.0                           | 10.0 | 10.0 | 15.0             | 4.0              | 6.0              |                   |     |  | 13.3                                                 | 385                                                     | 2285                   | -                       |
|           | 49.0                            | 6.4                            | 11.7 | 16.2 | 12.3             | 2.1              | 2.3              |                   |     |  |                                                      |                                                         |                        |                         |
| X-2068    | 50.0                            | 10.0                           | 5.0  | 10.0 | 15.0             | 4.0              | 6.0              |                   |     |  | 13.6                                                 | 255                                                     | 2390                   | -                       |
|           | 54.7                            | 6.4                            | 5.9  | 16.3 | 12.3             | 2.1              | 2.3              |                   |     |  |                                                      |                                                         |                        |                         |
| X-2069    | 45.0                            | 10.0                           | 5.0  | 10.0 | 16.0             | 4.0              | 10.0             |                   |     |  | 12.6                                                 | 324                                                     | 2325                   | -                       |
|           | 50.8                            | 6.6                            | 6.0  | 16.8 | 13.6             | 2.2              | 3.9              |                   |     |  |                                                      |                                                         |                        |                         |
| X-2070    | 42.9                            | 14.3                           | 9.5  | 9.5  | 14.3             | 3.8              | 5.7              |                   |     |  | 14.2                                                 | 522                                                     | 2310                   | -                       |
|           | 47.5                            | 9.3                            | 11.3 | 15.7 | 11.9             | 2.1              | 2.2              |                   |     |  |                                                      |                                                         |                        |                         |
| X-2071    | 52.7                            | 22.7                           |      | 9.1  | 9.1              | 6.4              |                  |                   |     |  | 13.4                                                 | 328                                                     | 2735                   | -                       |
|           | 58.8                            | 14.9                           |      | 15.1 | 7.6              | 3.5              |                  |                   |     |  |                                                      |                                                         |                        |                         |
| X-2072    | 54.6                            | 22.7                           |      | 9.1  | 9.1              | 4.6              |                  |                   |     |  | 13.1                                                 | 455                                                     | 2735                   | -                       |
|           | 60.2                            | 14.8                           |      | 15.0 | 7.6              | 2.4              |                  |                   |     |  |                                                      |                                                         |                        |                         |
| X-2073    | 40.9                            | 18.2                           | 9.1  | 9.1  | 13.6             | 3.6              | 5.5              |                   |     |  | 14.5                                                 | 462                                                     | 2315                   | -                       |
|           | 46.0                            | 12.1                           | 11.0 | 15.3 | 11.5             | 2.0              | 2.1              |                   |     |  |                                                      |                                                         |                        |                         |
| X-2074    | 43.1                            | 14.3                           | 10.0 | 7.6  | 7.2              | 3.9              | 10.6             | 2.7               |     |  | 14.0                                                 | 360                                                     | 2180                   | -                       |
|           | 47.5                            | 9.3                            | 12.5 | 12.5 | 6.0              | 2.1              | 4.1              | 6.0               |     |  |                                                      |                                                         |                        |                         |
| X-2117    | 38.6                            | 23.0                           | 8.6  | 8.6  | 12.9             | 3.4              | 5.0              |                   |     |  | 14.6                                                 | 413                                                     | 2340                   | -                       |
|           | 44.2                            | 15.5                           | 10.6 | 14.7 | 11.1             | 1.9              | 2.0              |                   |     |  |                                                      |                                                         |                        |                         |

**Table IX (Continued)**

| Glass No. | SiO <sub>2</sub> |      | Al <sub>2</sub> O <sub>3</sub> |      | CaO  | MgO | TiO <sub>2</sub> | ZrO <sub>2</sub>       | CeO <sub>2</sub> | Li <sub>2</sub> O | BeO  | Modulus of Elasticity |                       | Average Tensile Strength x 10 <sup>3</sup> psi | Forming Temp. °F | Bulk Density g/cc |  |  |
|-----------|------------------|------|--------------------------------|------|------|-----|------------------|------------------------|------------------|-------------------|------|-----------------------|-----------------------|------------------------------------------------|------------------|-------------------|--|--|
|           |                  |      |                                |      |      |     |                  |                        |                  |                   |      | x 10 <sup>6</sup> psi | x 10 <sup>3</sup> psi |                                                |                  |                   |  |  |
| X-2130    | 38.3             | 28.9 | 4.0                            | 6.8  | 12.4 | 3.5 | 4.9              | 1.3                    | 14.3             | 305               | 2390 | -                     |                       |                                                |                  |                   |  |  |
|           | 45.0             | 20.0 | 5.0                            | 12.0 | 11.0 | 2.0 | 2.0              | 3.0                    |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2131    | 38.2             | 28.8 | 7.1                            | 7.4  | 10.2 | 3.5 | 4.9              | 14.5-                  | 733              | 2505              | 2.91 |                       |                       |                                                |                  |                   |  |  |
|           | 45.0             | 20.0 | 9.0                            | 13.0 | 9.0  | 2.0 | 2.0              | 15.3                   |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2133    | 38.3             | 28.9 | 5.8                            | 8.4  | 10.0 | 3.5 | 4.9              | Couldn't Obtain        | 393              | 2340              | 2.86 |                       |                       |                                                |                  |                   |  |  |
|           | 45.0             | 20.0 | 7.3                            | 14.7 | 9.0  | 2.0 | 2.0              |                        |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2134    | 37.7             | 28.4 | 11.5                           | 4.1  | 10.0 | 3.4 | 4.8              | 15.7                   | 656              | 2535              | 3.4  |                       |                       |                                                |                  |                   |  |  |
|           | 45.0             | 20.0 | 14.7                           | 7.3  | 9.0  | 2.0 | 2.0              |                        |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2135    | 43.7             | 22.3 | 7.3                            | 7.6  | 10.5 | 3.6 | 5.0              | 15.0                   | 655              | 2505              | 2.9  |                       |                       |                                                |                  |                   |  |  |
|           | 50.0             | 15.0 | 9.0                            | 13.0 | 9.0  | 2.0 | 2.0              |                        |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2136    | 38.0             | 28.7 | 8.7                            | 6.2  | 10.1 | 3.5 | 4.8              | 14.7                   | 456              | 2410              | 3.0  |                       |                       |                                                |                  |                   |  |  |
|           | 45.0             | 20.0 | 11.0                           | 11.0 | 9.0  | 2.0 | 2.0              |                        |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2137    | 39.8             | 25.9 | 11.6                           | 4.2  | 10.1 | 3.5 | 4.9              | 14.0                   | 519              | 2505              | 2.9  |                       |                       |                                                |                  |                   |  |  |
|           | 47.0             | 18.0 | 14.7                           | 7.3  | 9.0  | 2.0 | 2.0              |                        |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
| X-2169    | 39.5             | 28.1 | 12.6                           | 4.5  | 8.5  | 3.8 | 3.1              | Could Not Be Fiberized |                  |                   |      |                       |                       |                                                |                  |                   |  |  |
|           | 43.0             | 18.0 | 14.7                           | 7.3  | 7.0  | 2.0 | 8.0              |                        |                  |                   |      |                       |                       |                                                |                  |                   |  |  |

glass series is:

| <u>Oxide</u>                   | <u>Mole %</u> |
|--------------------------------|---------------|
| SiO <sub>2</sub>               | 45 (approx.)  |
| Al <sub>2</sub> O <sub>3</sub> | 20            |
| CaO/MgO                        | 2/1           |
| TiO <sub>2</sub>               | 10            |
| ZrO <sub>2</sub>               | 2             |
| CeO <sub>2</sub>               | 2             |

The use of BeO, at least in this series of glasses, is detrimental to the glass as shown by the fiberization problems with X-2169. The bulk densities of all glasses investigated are high, of course, but are not unexpected.

### SECTION III

#### Relationship Between Composition and Properties of Selected Glasses

##### 1. Background Relating to Study

Glass fibers with the highest possible tensile strength and modulus of elasticity are desired for many important applications. These properties are intimately related to the ultimate structure of glass. Knowledge of the structure of a glass and its relationship to these properties would be invaluable in developing new glass compositions with superior performance.

The work reported here started as an attempt to find out whether modulus of elasticity and tensile strength of fibers could be related to glass composition and presumably structure in the same manner as has been done with certain other properties. For instance, Day and Rindone (3,4) have shown that a number of physical properties of glasses in the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Na<sub>2</sub>O field vary in a systematic manner with composition changes. Further, composition vs. property curves usually show breaks which they related to a proposed theory of changes in glass structure. In sets of related compositions, breaks occur at the points where the Al to Na ratio is one. It was postulated that at ratios below one, the aluminum is in fourfold coordination and participates in the network, while at higher ratios some of the aluminum assumes sixfold coordination and acts as a modifier. At a ratio of Al to Na of one, there are just enough oxygens present to supply two for each Si and Al ion, the ratio needed for fourfold coordination.

Earlier work on the same project<sup>(1)</sup> showed that a break at Al to Na ratios of one exists in the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Na<sub>2</sub>O field for the properties of modulus of elasticity, tensile strength, and

fiber forming temperature. These results led to the study reported here in which several other oxides from Group 1 or 2 in the periodic table were substituted for  $\text{Na}_2\text{O}$ .

## 2. Experimental Procedure

### a. Formulation of Compositions Studied

Compositions were calculated on a mole percent basis, and corresponded in general to those selected for the original study in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O}$  field. Batches were formulated at three arbitrarily selected  $\text{SiO}_2$  contents, 75, 66, and 57 mole percent. The following oxides were included in the study:  $\text{K}_2\text{O}$ ,  $\text{Li}_2\text{O}$ , mixtures of  $\text{K}_2\text{O}$  and  $\text{Li}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{ZnO}$  and  $\text{BeO}$ .

Batches were formulated from a high purity commercial potter's flint, commercial calcined alumina of 99.5% purity, and U.S.P. quality  $\text{K}_2\text{CO}_3$ ,  $\text{Li}_2\text{CO}_3$ ,  $\text{CaCO}_3$ ,  $\text{MgO}$ ,  $\text{ZnO}$  and  $\text{BeO}$ .

### b. Preparation of Melts

Batches were prepared and melted essentially as described previously in Section II. The glasses were crushed and re-melted once in each case to improve homogeneity. Melting times and temperatures varied between glasses. A combination of time and temperature was selected which was adequate to produce nearly seed-free glass. Usually melting periods were one hour each, with the temperature adjusted as required. However, longer melting times were used with the most viscous glasses. Maximum melting temperatures were around  $3150^\circ$ . Crucibles used for melting  $\text{ZnO}$  were covered with lids during the melting process to minimize volatilization of  $\text{ZnO}$ . A few crucibles with contents were weighed before and after melting the batch. The loss in weight attributable to volatilization was small enough to justify the conclusion that losses of  $\text{ZnO}$  could be ignored for purposes of the present study.

### c. Preparation of Fibers

Fibers for testing were made in a one hole bushing. In general a pulling speed of 6125 feet per minute was used for tensile strength fibers. In the case of some very viscous glasses and several of the lithia glasses, it was necessary to reduce the speed. In the case of the viscous glasses, the speed was reduced to avoid excessive bushing temperatures. Some of the lithia glasses had to be run at lower speeds because the forming cones became unstable at the temperatures required for the desired fiber diameters.

Note was taken of the forming temperature as measured by a thermocouple attached to the bushing wall near the tip. These temperatures gave some indication of relative glass viscos-

ities, but large variations were introduced because of bushing changes, drift of the thermocouple output with time, and modifications of forming procedure necessary with some of the glasses.

The target fiber diameter for tensile strength measurements was 0.00035 to 0.00040 inches. Some fibers were taken which were outside this range, particularly in the case of glasses that were especially hard to fiberize. However, all tests included fibers in this range, and it is felt that changes in fiber diameter between tests was not an important factor. Fibers were captured and mounted on forks in a manner that permitted the testing of sections of untouched fiber. In this case, each fork contained only five fibers, the maximum amount conveniently obtained in one capture with the bushing position used.

The target fiber diameter for fibers used for measuring modulus was 0.00070 to 0.00080 inches. Modulus fibers were prepared at the same temperature as tensile strength fibers. The increased diameter was obtained by slowing down the winder as required. Usually this speed was about 1000 feet per minute. The above target for fiber diameters was selected because it is a convenient size to handle and because it was noticed earlier that results were influenced somewhat by fiber diameter. Some fibers outside the above limits were used, particularly in the case of glasses that proved especially hard to fiberize. However, since in all cases some fibers were in this range, and all were close to it, it is felt that variation in fiber diameter from the target was not responsible for any serious errors.

#### d. Tensile Strength Measurement

Fibers were transferred on the forks on which they were collected to the tensile strength tester described in Section II and shown as Figure 1 (Page 3). Usually, fibers were tested within an hour after being prepared. A normal test consisted of three sets of three forks each, with five breaks per fork. In a few cases where fibers were extremely difficult to obtain, only three forks were prepared.

Fiber diameters were determined on fragments of fibers remaining on the forks, since in most cases the sections tested were completely destroyed. Diameters were measured on the microscope by means of an eyepiece with movable crosshair. Fibers to be measured were immersed in an oil of slightly lower index, selected so that the fiber edges appeared reasonably sharp. In some cases, measurements were cross-checked using the Vickers Image-Splitting Eyepiece. The same values were obtained.

#### e. Modulus of Elasticity Measurement

Modulus of elasticity was measured on sections of fibers

about 14 inches long by determining the amount of elongation under loads of 5 and 10 grams. Normally measurements were made at these two loadings on five fibers, so that each point reported is the average of ten determinations. The apparatus is pictured in Figure 2 (Page 4).

Fiber diameters were measured optically in the same manner as were those used for tensile strength.

f. Density Measurement

In a few cases, glass densities were run. Pieces of cullet about a gram in weight were annealed and run by the Archimedes method, using distilled water as the immersion medium. It is possible that some of the glasses were slightly soluble in water, and that precision could have been improved by selection of a more inert liquid. However, in view of the short time of immersion involved and the precision required, it was felt that results with the distilled water were acceptable.

3. Results

a.  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-K}_2\text{O}$  System

Compositions studied and results obtained are given in Table X. In general, glasses in this system were more difficult to fiberize than those in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Na}_2\text{O}$  system. They were more viscous than the corresponding  $\text{Na}_2\text{O}$  glasses, and devitrified more readily and at higher temperatures. The glass at the 75%  $\text{SiO}_2$  level and Al/K ratio of 1.0 could not be fiberized at temperatures attainable because of its high viscosity. The glass at the 57%  $\text{SiO}_2$  level and Al/K ratio of 1.0 devitrified too readily to permit fiber forming. All glasses at Al/K ratios higher than 1.0 were too viscous to fiberize at normal pulling speeds. Specimens obtained from them were formed at reduced speeds. Consequently, the temperatures reported in Table X do not represent accurate indications of relative glass viscosities. However, they indicate that there is a break in the viscosity-composition curve at or near the point at which the Al/K ratio is 1.0.

Tensile strength results are consistent with the expectation that there should be a break at an Al/K ratio of 1.0, but are too variable to establish this with certainty. Data could not be obtained for two of the series at the points at which the Al/K ratio is 1.0. Further, results shown for the higher ratios were obtained on fibers pulled at reduced speeds, so that strengths obtained cannot be reliably compared with those pulled at normal speeds.

In Figure 9, modulus is plotted as a function of composition. Part of the curves for the 75% and the 57%  $\text{SiO}_2$  levels is

**TABLE X**  
**Compositions and Properties of Silica-Alumina-Potash Glasses**

| <u>Glass #</u>                  | <u>X-2001</u>       | <u>X-2002</u> | <u>X-2003</u> | <u>X-2004</u> | <u>X-2005</u>              | <u>X-2006</u> | <u>X-2007</u> |                  |
|---------------------------------|---------------------|---------------|---------------|---------------|----------------------------|---------------|---------------|------------------|
| <b>Composition</b>              |                     |               |               |               |                            |               |               |                  |
| Mole % SiO <sub>2</sub>         | 75.00               | 75.00         | 75.00         | 75.00         | 75.00                      | 75.00         | 75.00         |                  |
| Al <sub>2</sub> O <sub>3</sub>  | -                   | 3.13          | 6.25          | 9.38          | 12.50                      | 15.00         | 16.25         |                  |
| K <sub>2</sub> O                | 25.00               | 21.88         | 18.75         | 15.63         | 12.50                      | 10.00         | 8.75          |                  |
| <b>Ave. Tensile</b>             |                     |               |               |               |                            |               |               |                  |
| Strength x 10 <sup>-3</sup> psi | 94                  | 175           | 235           | 385           | Could not form test fibers |               |               |                  |
| <b>Ave. Modulus</b>             |                     |               |               |               |                            |               |               |                  |
| x 10 <sup>-6</sup> , psi        | 5.44                | 6.18          | 7.11          | 7.85          | -                          | 8.83*         | 9.83*         |                  |
| <b>Forming Temp °F</b>          |                     |               |               |               |                            |               |               |                  |
|                                 | 2435                | 2760          | 3010          | 3200          | Over<br>3200               | 3205*         | 3205*         |                  |
| <u>Glass #</u>                  | <u>X-2008</u>       | <u>X-2009</u> | <u>X-2010</u> | <u>X-2011</u> | <u>X-2012</u>              | <u>X-2013</u> | <u>X-2014</u> |                  |
| <b>Composition</b>              |                     |               |               |               |                            |               |               |                  |
| Mole % SiO <sub>2</sub>         | 66.00               | 66.00         | 66.00         | 66.00         | 66.00                      | 66.00         | 66.00         |                  |
| Al <sub>2</sub> O <sub>3</sub>  | -                   | 6.00          | 12.00         | 17.00         | 20.00                      | 22.00         | 24.00         |                  |
| K <sub>2</sub> O                | 34.00               | 28.00         | 22.00         | 17.00         | 14.00                      | 12.00         | 10.00         |                  |
| <b>Ave. Tensile</b>             |                     |               |               |               |                            |               |               |                  |
| Strength x 10 <sup>-3</sup> psi | 76                  | 162           | 228           | 177*          | 240*                       | 192*          | -             |                  |
| <b>Ave. Modulus</b>             |                     |               |               |               |                            |               |               |                  |
| x 10 <sup>-6</sup> psi          | 4.45                | 5.56          | 7.41          | 7.55*         | 8.39*                      | 9.44*         | 9.94*         |                  |
| <b>Forming Temp °F</b>          |                     |               |               |               |                            |               |               |                  |
|                                 | 2235                | 2670          | 3060          | 3200          | 3220                       | 3200          | 3200          |                  |
| <u>Glass #</u>                  | <u>X-2015</u>       | <u>X-2016</u> | <u>X-2017</u> | <u>X-2018</u> | <u>X-2019</u>              | <u>X-2020</u> | <u>X-2021</u> | <u>X-2022</u>    |
| <b>Composition</b>              |                     |               |               |               |                            |               |               |                  |
| Mole % SiO <sub>2</sub>         | 57.00               | 57.00         | 57.00         | 57.00         | 57.00                      | 57.00         | 57.00         | 57.00            |
| Al <sub>2</sub> O <sub>3</sub>  | -                   | 6.00          | 12.00         | 17.00         | 21.50                      | 24.50         | 27.50         | 30.50            |
| K <sub>2</sub> O                | 43.00               | 37.00         | 31.00         | 26.00         | 21.50                      | 18.50         | 15.50         | 12.50            |
| <b>Ave. Tensile</b>             |                     |               |               |               |                            |               |               |                  |
| Strength x 10 <sup>-3</sup> psi | Too weak<br>to test | 134           | 232           | 333           | Could<br>not run           | 384           | 395           | Could<br>not run |
| <b>Ave. Modulus</b>             |                     |               |               |               |                            |               |               |                  |
| x 10 <sup>-6</sup> psi          | Too weak<br>to test | 5.00          | 6.56          | 8.13          | -<br>Over<br>3200          | 9.10          | 9.63          |                  |
| <b>Forming Temp °F</b>          |                     |               |               |               |                            |               |               |                  |
|                                 | -                   | 2505          | 2975          | 3205*         | 3200                       | 3205*         | 3205*         |                  |

\*Pulled fibers at speeds lower than normal

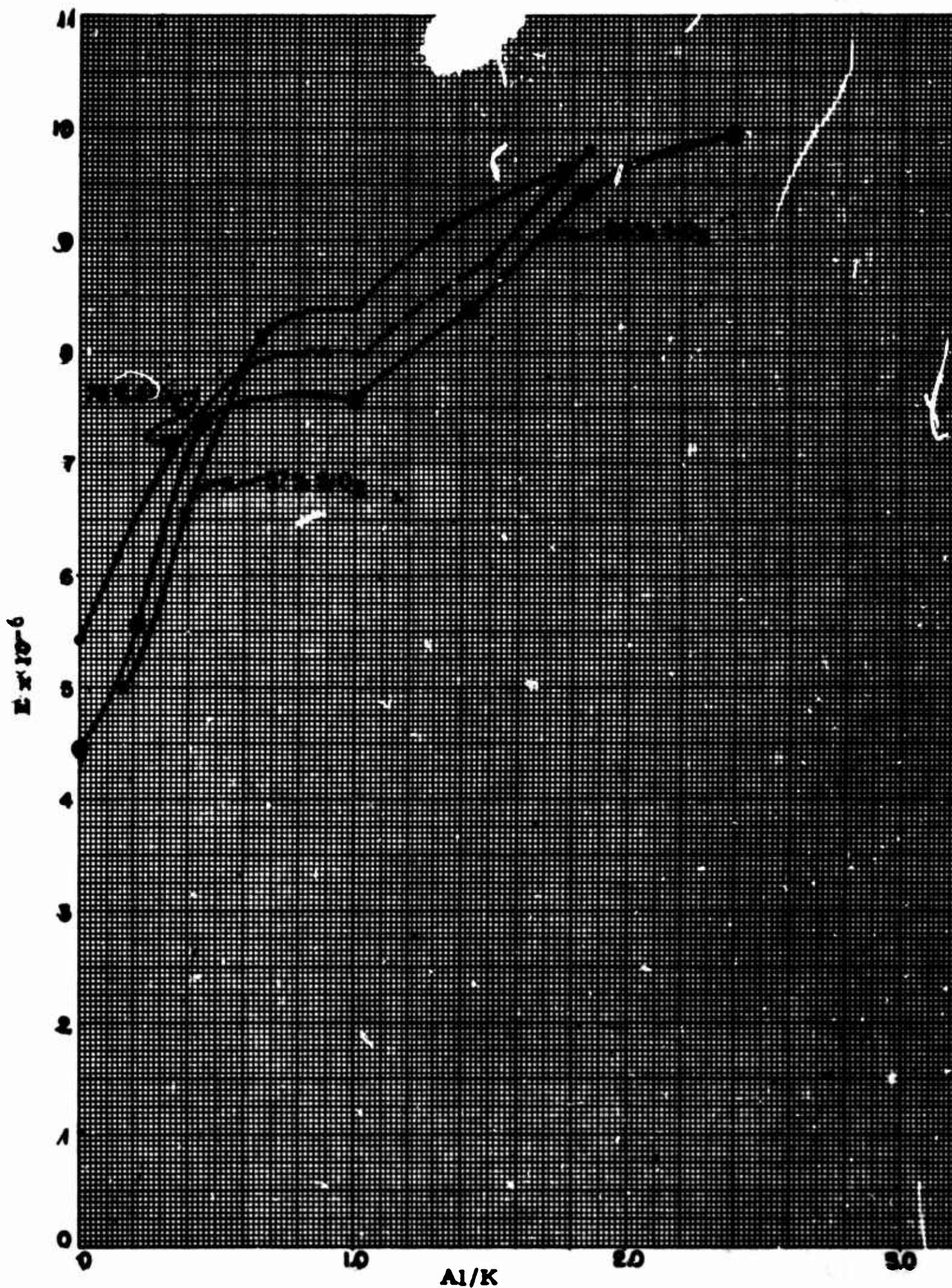


Figure 9 Modulus Vs. Al/K Ratio

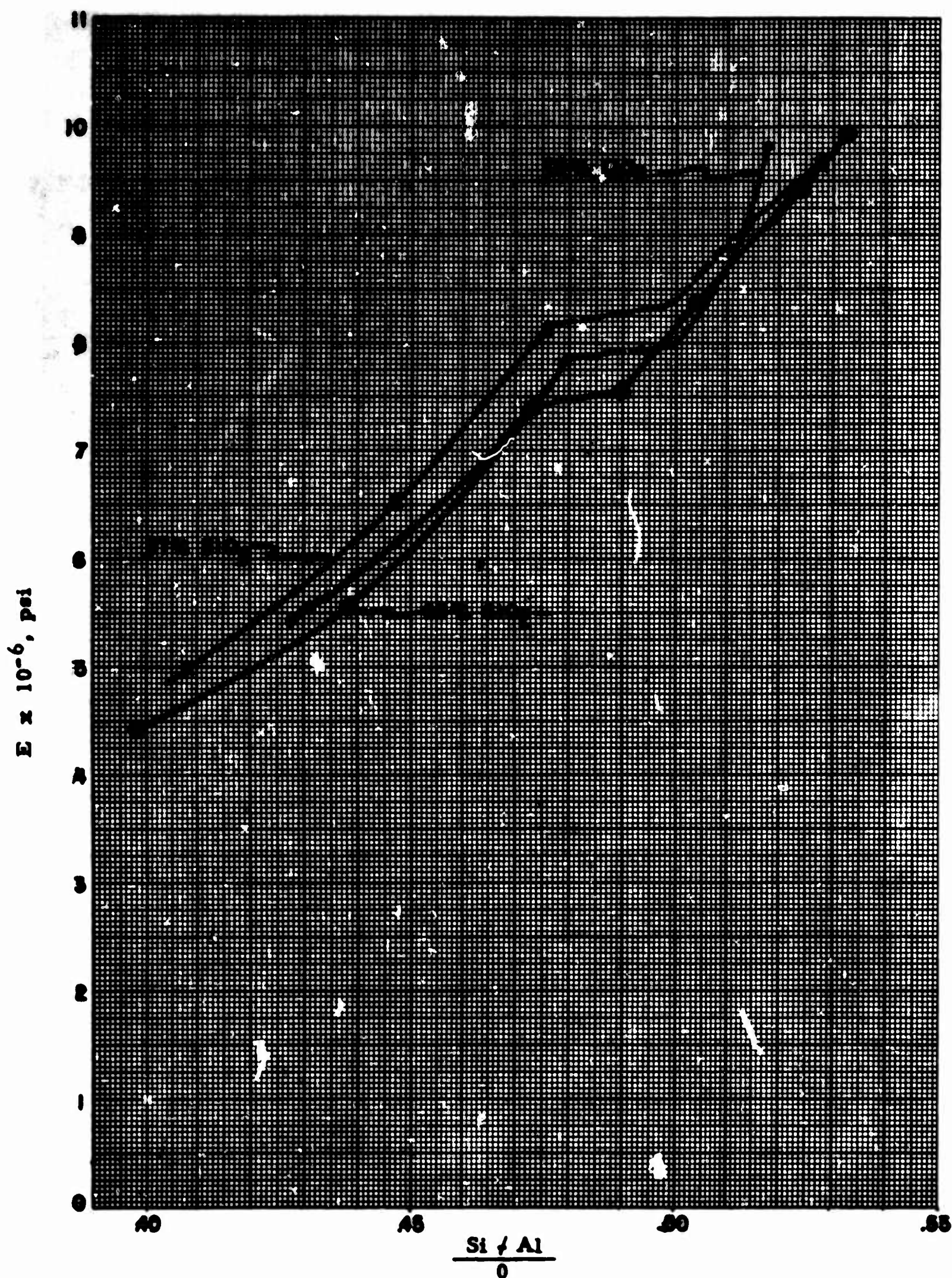


Figure 10 Modulus Vs.  $\frac{\text{Si} + \text{Al}}{0}$  Ratio For Glasses Containing  $\text{K}_2\text{O}$

dotted because of missing data. They are drawn to show a break at an Al/K ratio of 1.0. It seems probable that such a break exists from the location of the other points and because of the existence of a break at the 66% SiO<sub>2</sub> level.

Modulus is plotted as a function of the ratio of Si plus Al to O in Figure 10. This relationship was used in studying the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Na<sub>2</sub>O system to indicate that there was some dependence of the properties on the amount of Si present also. In both systems, the curves show two breaks, one at 0.5 (where the Al/K ratio is 1.0) and one at about 0.475. The significance of the latter value has not been determined.

The moduli of the potash glasses are lower than those of the corresponding soda glasses.

b. SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Li<sub>2</sub>O System

Compositions studied and results obtained are given in Table XI.

Glasses in this system were easier to fiberize than those in the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-K<sub>2</sub>O system, and ran at lower temperatures. However, most glasses devitrified readily. Also, they behaved in a peculiar manner, in that fibers of the sizes normally used for testing could be produced only by substantially reducing the pulling speed (2,000 vs. 3,600 rpm for a 6" diameter wheel). Normally, as the temperature is raised, the fiber diameter increases. With these glasses, raising the temperature increased the fiber diameter very little, up to the point at which the forming cone became too unstable to permit production of uniform fibers. Two possible reasons for this behavior suggested themselves: (1) these glasses might have an usually flat viscosity curve, that is, one in which viscosity changes relatively slowly with temperature; or (2) there might be an unusual surface tension effect.

To check these possibilities, viscosity measurements were made on several of the glasses. Results are presented in Figure 11. It would be desirable to run softening points (log  $\eta$  = -7.6) to extend the length of the curves to determine their slopes more accurately. However, the ease with which these glasses devitrified made all attempts to do so unsuccessful except for glass X-2083, which had a softening point of 1160° F. Inspection of all the available viscosity data suggests that these glasses have curves with about the slope that would be expected, and that this does not account for their forming behavior. A recent paper<sup>(5)</sup> indicates that the lithium ion increases surface tension of glasses compared to the sodium and potassium ions. Perhaps this is related to the unusual forming behavior.

Forming temperatures reported do not very accurately reflect

TABLE XI

Compositions and Properties of Silica-Alumina-Lithia Glasses

| <u>Glass #</u>                                      | <u>X-2075</u> | <u>X-2076</u> | <u>X-2077</u> | <u>X-2078</u> | <u>X-2079</u> | <u>X-2080</u> | <u>X-2081</u> | <u>X-2106</u> | <u>X-2107</u> |
|-----------------------------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| <b>Composition</b>                                  |               |               |               |               |               |               |               |               |               |
| Mole % SiO <sub>2</sub>                             | 75.00         | 75.00         | 75.00         | 75.00         | 75.00         | 75.00         | 75.00         | 75.00         | 75.00         |
| Al <sub>2</sub> O <sub>3</sub>                      | -             | 3.13          | 6.25          | 9.38          | 12.50         | 15.00         | 16.25         | 17.50         | 18.75         |
| Li <sub>2</sub> O                                   | 25.00         | 21.88         | 18.75         | 15.63         | 12.50         | 10.10         | 8.75          | 7.50          | 6.25          |
| <b>Ave. Tensile Strength x 10<sup>-3</sup>, psi</b> |               |               |               |               |               |               |               |               |               |
|                                                     | 270           | 315           | 340*          | 428*          | 455*          | 527*          | 561*          | 612*          | 666*          |
| <b>Ave. Modulus x 10<sup>-3</sup>, psi</b>          |               |               |               |               |               |               |               |               |               |
|                                                     | 9.38          | 10.05         | 10.38         | 10.52         | 10.47         | 11.37         | 11.70         | 11.95         | 12.20         |
| <b>Forming Temp. °F</b>                             |               |               |               |               |               |               |               |               |               |
|                                                     | 2265          | 2505          | 2850*         | 3155*         | 3230*         | 3105*         | 3040*         | 2975*         | 3095*         |
| <b>Liquidus, °F</b>                                 |               |               |               |               |               |               |               |               |               |
|                                                     | -             | -             | 1890          | -             | -             | -             | -             | -             | -             |
| <u>Glass #</u>                                      |               | <u>X-2082</u> | <u>X-2083</u> | <u>X-2084</u> | <u>X-2085</u> | <u>X-2086</u> | <u>X-2087</u> | <u>X-2088</u> |               |
| <b>Composition</b>                                  |               |               |               |               |               |               |               |               |               |
| Mole % SiO <sub>2</sub>                             |               | 66.00         | 66.00         | 66.00         | 66.00         | 66.00         | 66.00         | 66.00         |               |
| Al <sub>2</sub> O <sub>3</sub>                      |               | -             | 6.00          | 12.00         | 17.00         | 20.00         | 22.00         | 24.00         |               |
| Li <sub>2</sub> O                                   |               | 34.00         | 28.00         | 22.00         | 17.00         | 14.00         | 12.00         | 10.00         |               |
| <b>Ave. Tensile Strength x 10<sup>-3</sup> psi</b>  |               |               |               |               |               |               |               |               |               |
|                                                     |               | 274*          | 293*          | 325*          | 370*          | 415*          | 465*          | 512*          |               |
| <b>Ave. Modulus x 10<sup>-3</sup>, psi</b>          |               |               |               |               |               |               |               |               |               |
|                                                     |               | 10.06         | 10.46         | 10.80         | 10.59         | 11.20         | 11.60         | 11.96         |               |
| <b>Forming Temp. °F</b>                             |               |               |               |               |               |               |               |               |               |
|                                                     |               | 1955*         | 2125*         | 2445*         | 2850*         | 2810*         | 2915*         | 3095*         |               |
| <b>Liquidus, °F</b>                                 |               |               |               |               |               |               |               |               |               |
|                                                     |               | 1893          | 1840          | 2120          | 2610          | 2550          | -             | -             |               |
| <u>Glass #</u>                                      |               | <u>X-2089</u> | <u>X-2090</u> | <u>X-2091</u> | <u>X-2092</u> | <u>X-2093</u> | <u>X-2094</u> | <u>X-2095</u> | <u>X-2096</u> |
| <b>Composition</b>                                  |               |               |               |               |               |               |               |               |               |
| Mole % SiO <sub>2</sub>                             |               | 57.00         | 57.00         | 57.00         | 57.00         | 57.00         | 57.00         | 57.00         | 57.00         |
| Al <sub>2</sub> O <sub>3</sub>                      |               | -             | 6.00          | 12.00         | 17.00         | 21.50         | 24.50         | 27.50         | 30.50         |
| Li <sub>2</sub> O                                   |               | 43.00         | 37.00         | 31.00         | 26.00         | 21.50         | 18.50         | 15.50         | 12.50         |
| <b>Ave. Tensile Strength x 10<sup>-3</sup> psi</b>  |               |               |               |               |               |               |               |               |               |
|                                                     |               | Could not run | Could not run | 259*          | 319*          | 448*          | 367*          | Could not run | Could not run |
| <b>Ave. Modulus x 10<sup>-3</sup>, psi</b>          |               |               |               |               |               |               |               |               |               |
|                                                     |               | "             | "             | 10.13         | 10.40         | 10.28         | 11.30         | 11.73         | "             |
| <b>Forming Temp °F</b>                              |               |               |               |               |               |               |               |               |               |
|                                                     |               | -             | -             | 2075*         | 2445*         | 2560*         | 2505*         | -             | -             |
| <b>Liquidus, °F</b>                                 |               |               |               |               |               |               |               |               |               |
|                                                     |               | 2145          | 2027          | 1905          | 2385          | 2587          | 2530          | -             | -             |

\*Run at speeds lower than usual

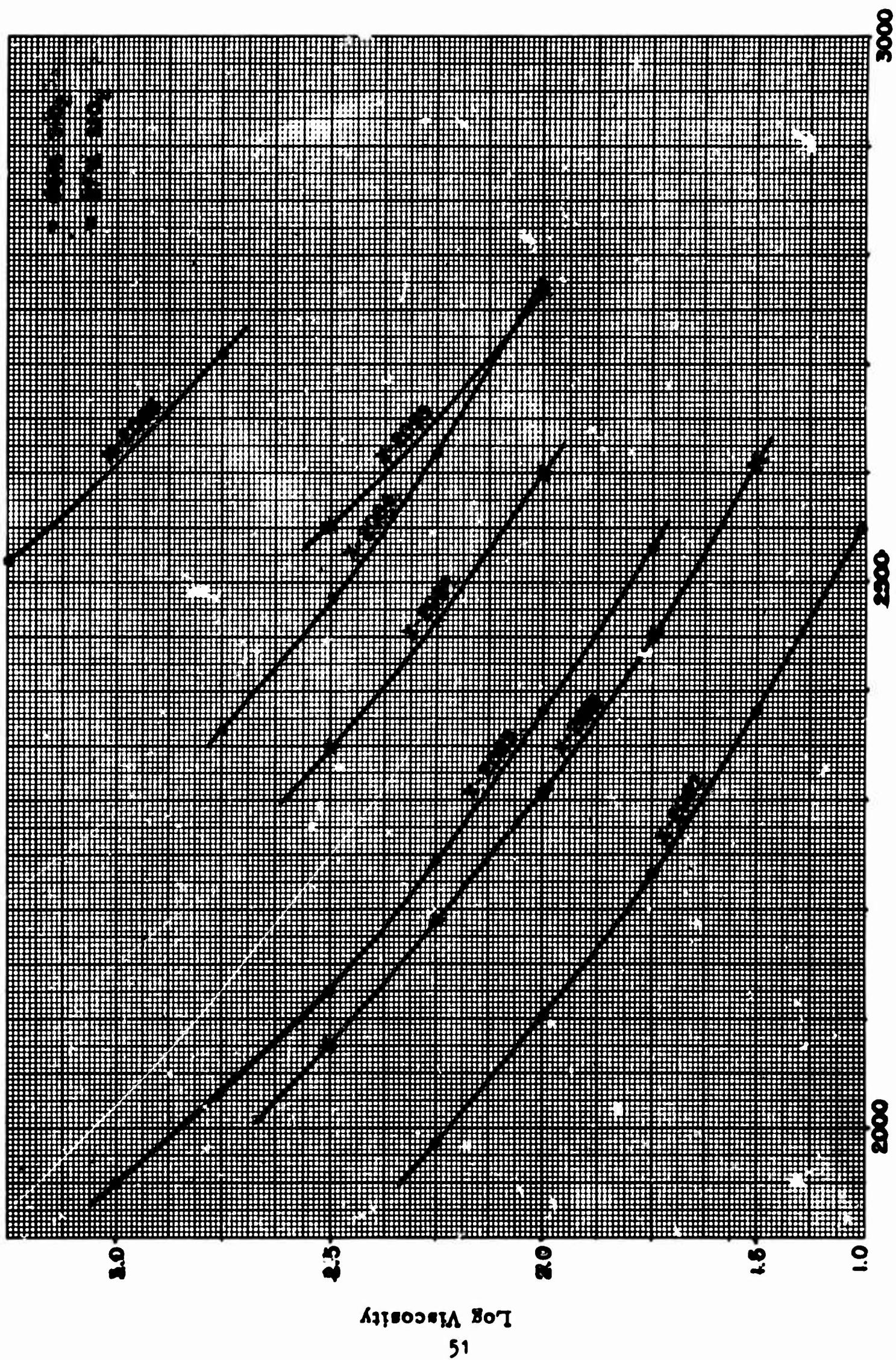


Figure 11 Log Viscosity Vs. Temperature For Glasses In The  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-Li}_2\text{O}$  System

differences in viscosity between glasses, because of the previously discussed relationship between forming temperature and fiber diameter. They do indicate a break at or near an Al/Li ratio of 1.0, such as that found for the other alkalis. The values obtained for glasses with the higher Al/Li ratios are the most uncertain because they were run at temperatures selected to prevent devitrification rather than to give the optimum viscosity.

There is no clear indication of a break in the tensile strength vs. composition curve at an Al/Li ratio of 1.0. Tensile strengths are rather higher for the lithia glasses than for the corresponding soda and potash glasses.

In view of the observed tendency of these glasses to devitrify readily, it was felt that knowledge of the relationship between liquidus and forming temperature might help explain the different behavior of these glasses compared to the corresponding soda and potash glasses. This difference was apparent both in forming behavior and in the failure to show a definite break in the tensile strength vs. composition curve. Accordingly, attempts were made to measure liquidus on most of these glasses. Liquidus is most conveniently measured by the so-called gradient boat method. Crushed glass is put in a long shallow platinum boat, which is introduced into a furnace constructed so as to have a nearly linear temperature gradient. The boat is left in the furnace for a time sufficient to permit the formation of devitrification, then withdrawn and quickly quenched in air. If the gradient was properly chosen, the boat will contain a region of clear glass and a boundary below which crystalline material may be found. The temperature pertaining to this boundary may be determined from the temperature distribution in the furnace. When this technique was applied to the lithia glasses, it was noted that the clear glass above the boundary devitrified during cooling. This process could not be completely stopped even by quenching the boat in water. Consequently, the boundary was estimated visually on the glass in the hot boat immediately after it was removed from the furnace. This procedure could not be very accurate. However, results checked well with available data reported in the literature, which were obtained by the more accurate but more tedious quenching technique. Results are included in Table XI (Page 50). In most cases, the liquidus was well below the forming temperature, so that results should not have been affected by devitrification during forming. In a few cases, particularly at the 57%  $\text{SiO}_2$  level, forming temperatures were barely over the liquidus. Results for these glasses might be questionable in view of the relative inaccuracy of liquidus and forming temperature measurements. However, they seem to be consistent with measurements in the other series and other systems.

Modulus is plotted as a function of composition in Figure 12. A break at an Al/Li ratio of 1.0 is clearly indicated. It is noteworthy that the curves do not fall in the same order as

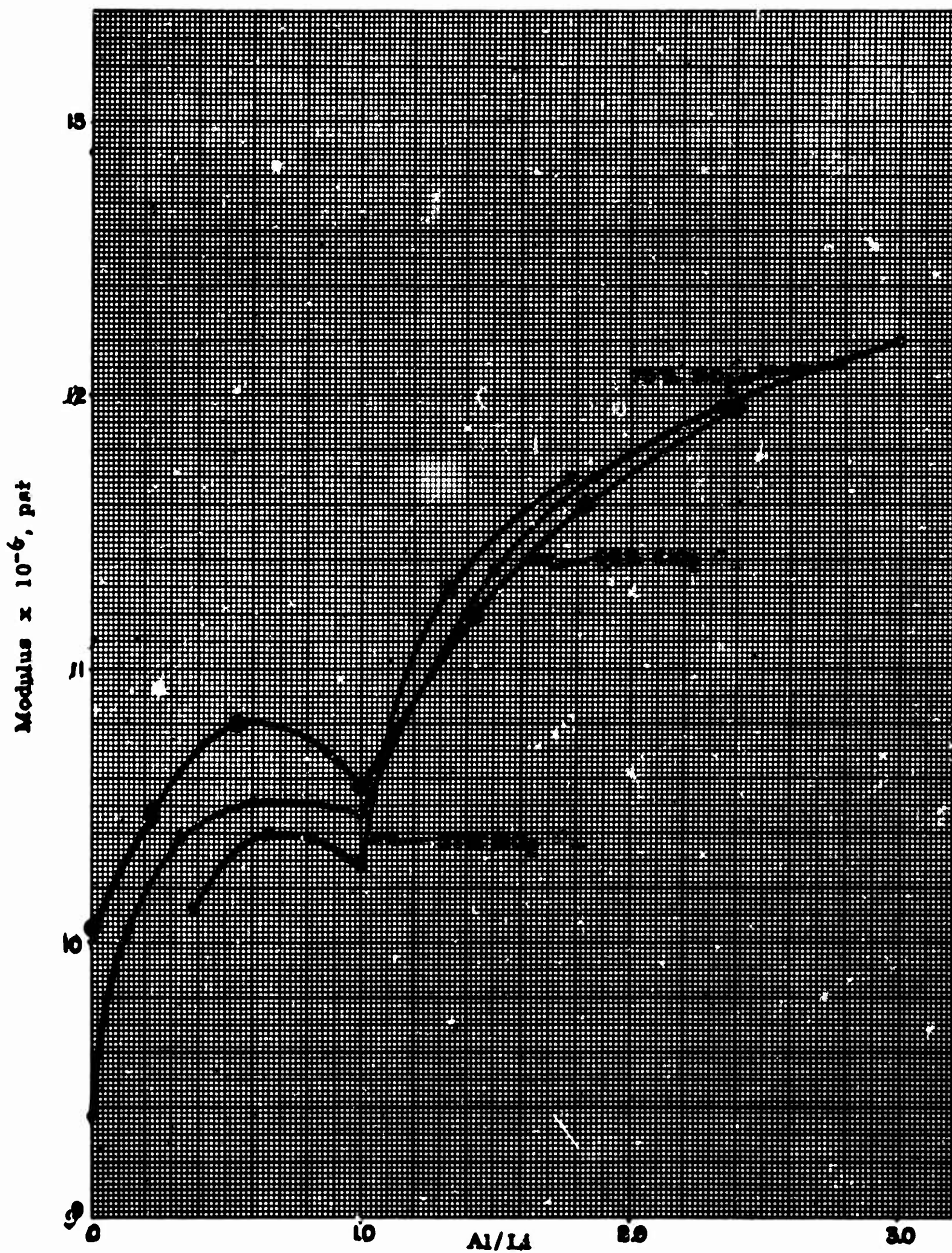


Figure 12 Modulus Vs. Al/Li Ratio

SiO<sub>2</sub> contents and that they cross above an Al/Li ratio of 1.0. Figure 13 shows the same data with modulus plotted as a function of the ratio of Si plus Al to O. Again, two breaks are found, one at 0.5, the other near 0.475. The lithia glasses clearly show higher moduli than the corresponding soda or potash glasses.

Because of the uncertainty of the glass viscosity measurement as indicated by forming temperatures, and because of the failure to find breaks in the tensile strength vs. composition curves, density was run on these glasses. It was hoped that density vs. composition curves would show a break at an Al/Li ratio of 1.0, thus tending to corroborate the conclusions that might be drawn from the breaks in the modulus vs. composition curves. The data obtained are plotted in Figure 14. The curves are drawn to the average values for several determinations. The spread in results for each point is indicated by the lengths of the vertical bars. The results clearly indicate that a break exists at all three silica levels in the curve of density vs. the Al/Li ratio at a value of 1.0. This corroborates the finding on modulus.

c. SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-K<sub>2</sub>O-Li<sub>2</sub>O System

The properties of the glasses containing Na<sub>2</sub>O were intermediate between those of the K<sub>2</sub>O and Li<sub>2</sub>O glasses, a not unexpected result. It seemed likely that the properties of Na<sub>2</sub>O glasses could be duplicated by mixtures of Li<sub>2</sub>O and K<sub>2</sub>O at comparable ratios of alkali ions to the other ions. It was hoped that something could be learned regarding the role of the alkali ions themselves by doing this.

Table XII gives results for a series of glasses in which the ratio of ions selected corresponds to equal volumes of cations, that is, the volume of the Li<sup>+</sup> plus K<sup>+</sup> ions equalled that of an equal number of Na<sup>+</sup> ions. The compositions corresponded to the ones with Al/R ratios of one and the two nearest to it for each SiO<sub>2</sub> level. Comparison of the data in Table XII with results obtained on glasses containing Na<sub>2</sub>O reveals that the properties are obviously not a linear function of the volume of the alkali cation.

Table XIII gives the results obtained on the second series of glasses. These were selected on the basis of ionic field strengths, using the relationship:

$$(1) \frac{Li}{R^2_{Li}} + \frac{K}{R^2_K} = \frac{1}{R^2_{Na}}$$

where Li and K represent relative numbers of ions of lith-

Modulus  $\times 10^{-6}$ , psi

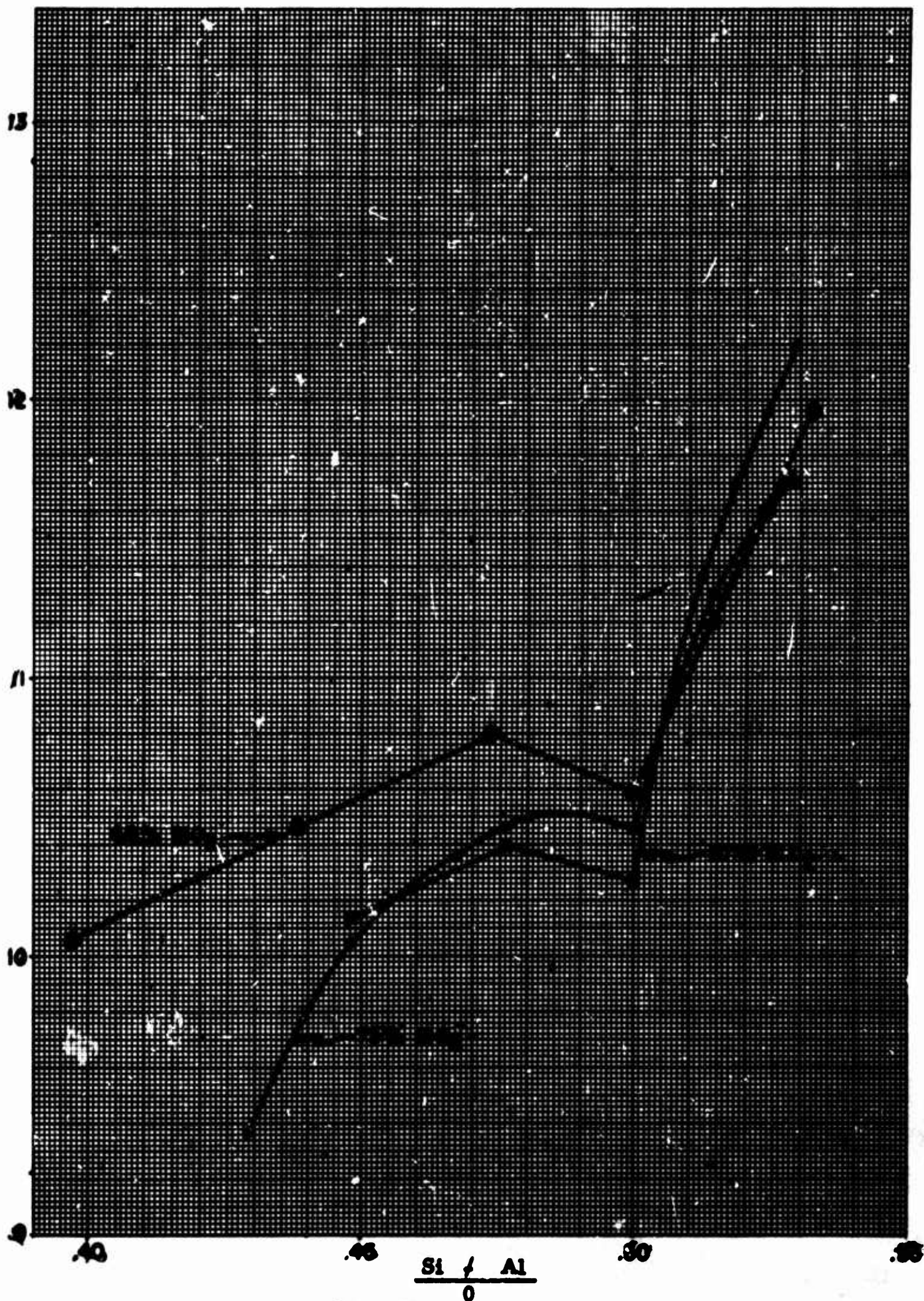


Figure 13 Modulus Vs.  $\frac{\text{Si} + \text{Al}}{0}$  Ratio For Glasses Containing  $\text{Li}_2\text{O}$

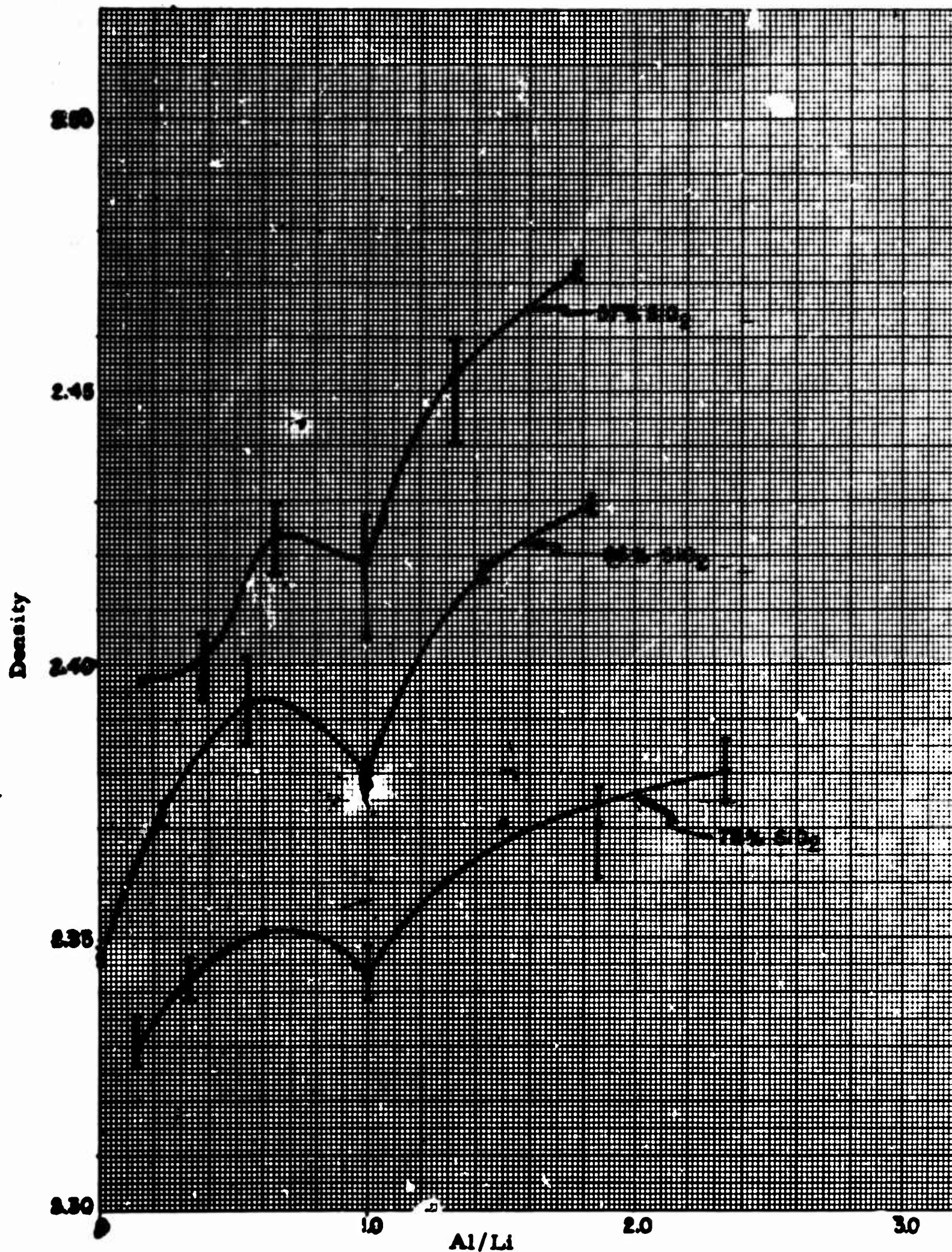


Figure 14  $\epsilon$  Al-Li Glasses Density Vs. Composition

TABLE XII

COMPOSITION AND PROPERTIES OF  
SILICA - ALUMINA - POTASH - LITHIA GLASSES  
SERIES I

| <u>Class #</u>                               | <u>X-2108</u> | <u>X-2109</u> | <u>X-2110</u>                                               | <u>X-2111</u> | <u>X-2112</u> | <u>X-2113</u> | <u>X-2114</u> | <u>X-2115</u> | <u>X-2116</u> |
|----------------------------------------------|---------------|---------------|-------------------------------------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|
| Composition                                  |               |               |                                                             |               |               |               |               |               |               |
| Mole % SiO <sub>2</sub>                      | 75.00         | 75.00         | 75.00                                                       | 66.00         | 66.00         | 66.00         | 57.00         | 57.00         | 57.00         |
| Al <sub>2</sub> O <sub>3</sub>               | 9.38          | 12.50         | 15.00                                                       | 12.00         | 17.00         | 20.00         | 17.00         | 21.50         | 24.50         |
| K <sub>2</sub> O                             | 3.91          | 3.12          | 2.50                                                        | 5.50          | 4.25          | 3.50          | 6.50          | 5.38          | 4.63          |
| Li <sub>2</sub> O                            | 11.72         | 9.38          | 7.50                                                        | 16.50         | 12.75         | 10.50         | 19.50         | 16.12         | 13.87         |
| Modulus x 10 <sup>-6</sup> , psi             | 9.84          | 9.74          | 10.48                                                       | 9.38          | 8.98          | 10.13         | 10.32         | 10.15         | 11.10         |
| Tensile Strength<br>x 10 <sup>-3</sup> , psi | 399           | 519           | 377                                                         | 350           | 480           | 487           | 325           | 428           | 444           |
| Forming Temperature<br>°F, ( 2000 rpm)       | 3095          | 3115          | 3155                                                        | 2530          | 2915          | 2975          | 2505          | 2645          | 2735          |
|                                              | Seedy         | Seedy         | Very Seedy<br>(probably<br>affected<br>tensile<br>strength) |               |               |               |               |               |               |

\*Repeat

TABLE XIII

COMPOSITION AND PROPERTIES OF  
SILICA - ALUMINA - POTASH - LITHIA GLASSES  
SERIES 2

| Glass #                                      | X-2143        | X-2144 | X-2145 | X-2146 | X-2147 | X-2148 | X-2149 | X-2150 | X-2151 |
|----------------------------------------------|---------------|--------|--------|--------|--------|--------|--------|--------|--------|
| Composition                                  |               |        |        |        |        |        |        |        |        |
| Mole % SiO <sub>2</sub>                      | 75.00         | 75.00  | 75.00  | 66.00  | 66.00  | 66.00  | 57.00  | 57.00  | 57.00  |
| Al <sub>2</sub> O <sub>3</sub>               | 9.38          | 12.50  | 15.00  | 12.00  | 17.00  | 20.00  | 17.00  | 21.50  | 24.50  |
| K <sub>2</sub> O                             | 10.50         | 8.41   | 6.73   | 14.80  | 11.44  | 9.42   | 17.49  | 14.47  | 12.45  |
| Li <sub>2</sub> O                            | 5.12          | 4.09   | 3.27   | 7.20   | 5.56   | 4.58   | 8.51   | 7.03   | 6.05   |
| Modulus, Average<br>psi x 10 <sup>-6</sup> , | 9.02          | 8.73   | 9.58   | 9.43   | 9.42   | 10.03  | 9.26   | 9.45   | 9.95   |
| Tensile Strength<br>psi x 10 <sup>-3</sup>   | 418           | N.D.*  | N.D.*  | 343    | 372    | 427    | 334    | 443    | 416    |
| Forming Temp.<br>°F.                         | 3060-<br>3080 | 3250** | 3250   | 2975   | 3040   | 3170   | 2975   | 3040   | 2915   |

\*Not Determined

\*\*Reduced Speed

ium and potassium, the sum of which is 1.0.  $r$  is the ionic radius of the indicated ion.

In general, the tensile strength of the glasses with mixed alkalis is near that of the lithia glasses, and among the higher values. It is difficult to generalize further about these data because of the scatter in results.

Data on forming temperatures were not plotted because they were not accurate enough reflections of viscosity to be meaningful. In general, they were consistent with the forming temperatures found for the other glasses.

Modulus data are plotted in Figures 15, 16, and 17. Figure 16 shows two sets of data for the mixed alkali glasses of Table XII. The second set, labeled "repeat" seems to fit better. It was obtained by rerunning the glasses after noting that the first set of data seemed inconsistent. Although the discrepancy is relatively small, it is greater than the error usually encountered. No explanation was apparent at the time the data were obtained.

The moduli of glasses in the second set, formulated on the basis of field strengths, lie rather close to those of the corresponding  $\text{Na}_2\text{O}$  glasses. However, they are consistently above the  $\text{Na}_2\text{O}$  curves. This led to an attempt to determine from the data the most likely ratio of  $\text{K}_2\text{O}$  to  $\text{Li}_2\text{O}$  to match the properties of  $\text{Na}_2\text{O}$ . Data were plotted to show the relationship between modulus and the proportions of  $\text{Li}_2\text{O}$  and  $\text{K}_2\text{O}$  for each  $\text{SiO}_2$  level, and each ratio of aluminum to total alkali ions. There appears to be very nearly a straight line relationship between modulus and the  $\text{Li}/\text{K}$  ratio. Assuming a straight line relationship, the  $\text{Li}_2\text{O}$ - $\text{K}_2\text{O}$  proportions that should give the same modulus as the corresponding  $\text{Na}_2\text{O}$  glass can be determined. The proportions of  $\text{Li}_2\text{O}$  and  $\text{K}_2\text{O}$  so determined vary quite widely from curve to curve. The precision of the data perhaps is not great enough for one to expect otherwise. However, there are enough data to suggest that an average value might be meaningful. On the average, mixtures of  $\text{Li}_2\text{O}$  and  $\text{K}_2\text{O}$  simulate the effect of  $\text{Na}_2\text{O}$  at the point at which the proportion of  $\text{Li}_2\text{O}$  is 21.7% of the total alkali.

Attempts to relate the average value for the point of intersection of the curves with ionic sizes are complicated by the precision with which ionic radii are known. For example, Table XIV gives ionic radii computed by four investigators for lithium, sodium, and potassium ions. For laying out the ratios of  $\text{Li}_2\text{O}$  to  $\text{K}_2\text{O}$  used in the series of mixed alkali glasses studied, average values were used. When these are used, equation (1) gives a value of 33% for the proportion of lithia. This is significantly higher than the average value

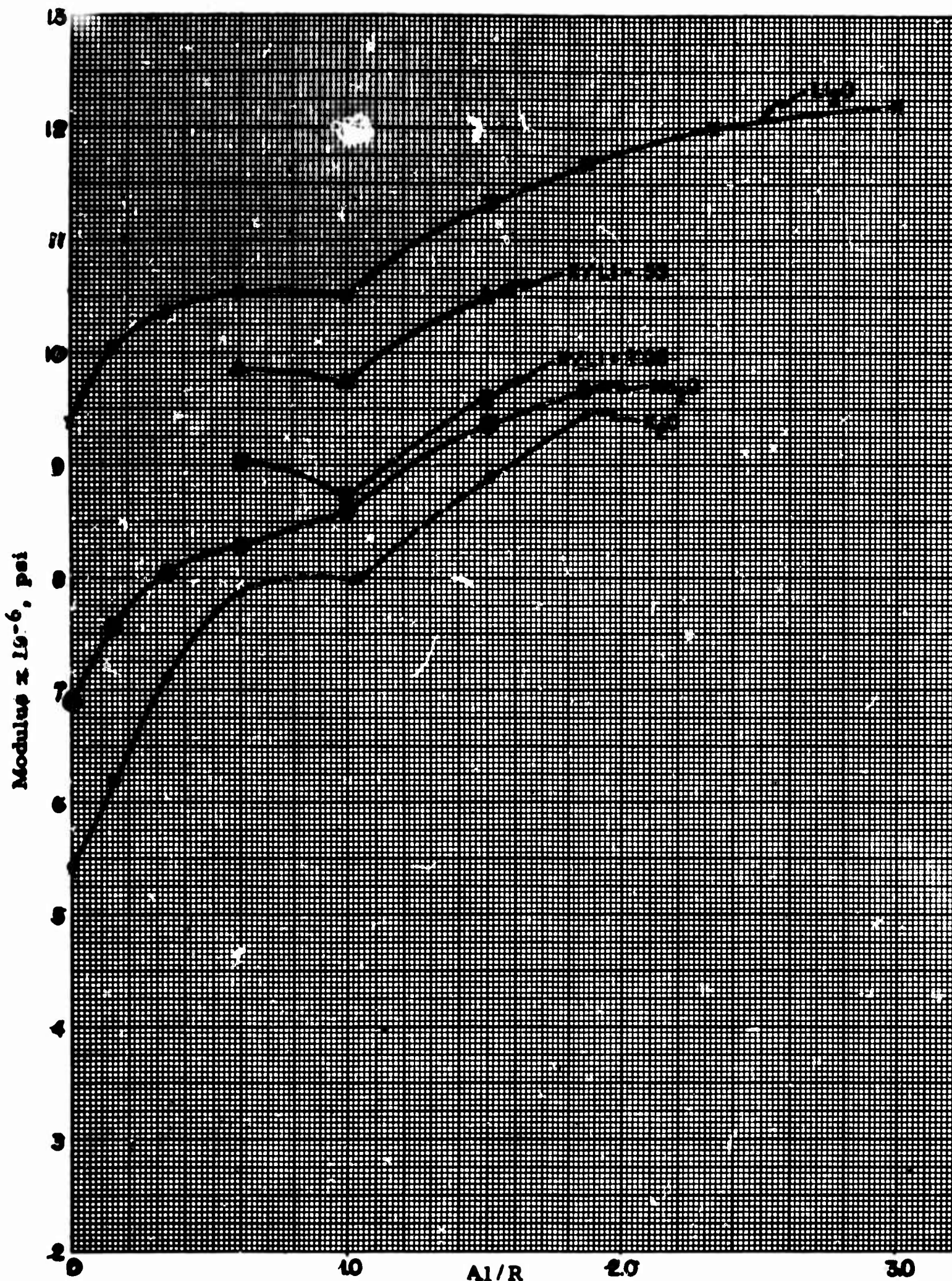


Figure 15 Silica-Alumina-Potash-Lithia Glasses Modulus Vs. Al/R Ratio at 75% SiO<sub>2</sub>

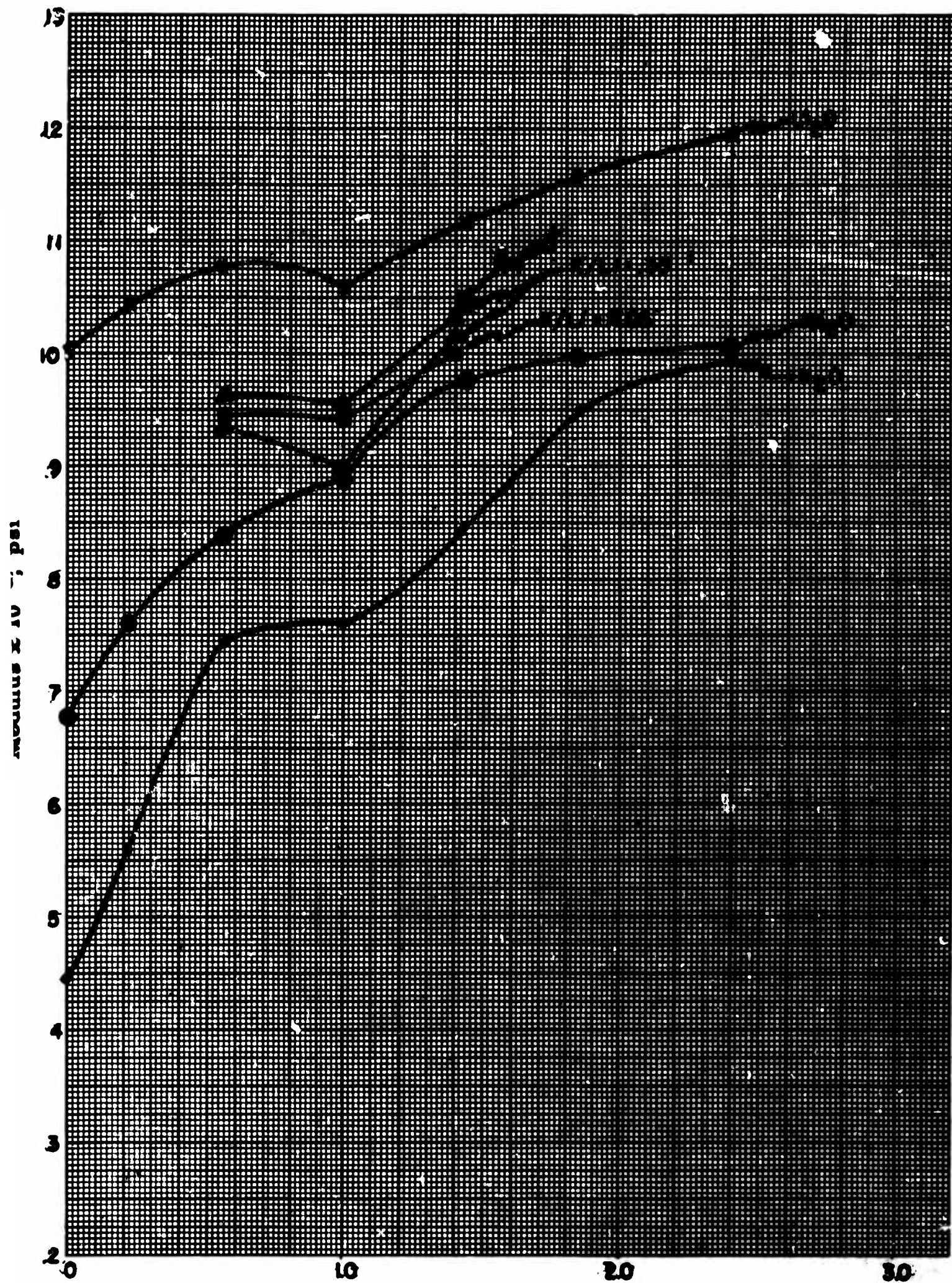


Figure 16 Silica-Alumina-Potash-Lithia Glasses Modulus Vs. Al/R Ratio at 66% SiO<sub>2</sub>

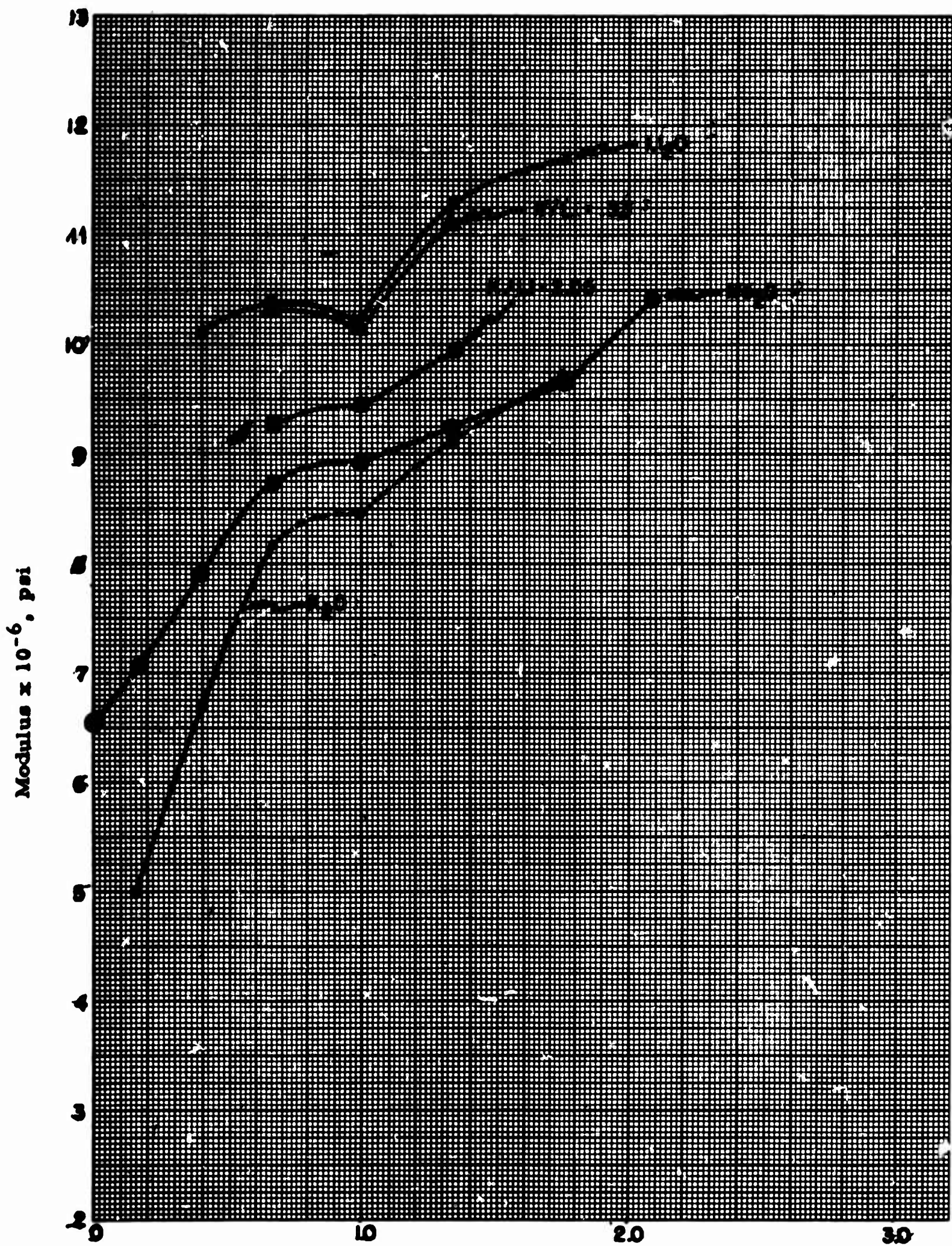


Figure 17 Silica-Alumina-Potash-Lithia Glasses Modulus Vs. Al/R Ratio at 57% SiO<sub>2</sub>

of 21.7% obtained from the curves. Inspection of equation (1) shows that the value for the proportion of  $\text{Li}_2\text{O}$  would drop if powers of the ionic radius higher than two were used. For the fourth power,  $\text{Li}_2\text{O}$  would amount to 20.1% of the alkali, a rather good correlation with the value of 21.7% picked from the curves. On the other hand, if the ionic radii proposed by Pauling are used, equation (1) gives a value of 24.6%  $\text{Li}_2\text{O}$ , which also is fairly close to the average observed value of 21.7%. Thus, the data from both the current

TABLE XIV

IONIC RADII

| <u>Source</u> | <u><math>\text{Li}^+</math></u> | <u><math>\text{Na}^+</math></u> | <u><math>\text{K}^+</math></u> |
|---------------|---------------------------------|---------------------------------|--------------------------------|
| Goldschmidt   | .78                             | .98                             | 1.33                           |
| Pauling       | .60                             | .95                             | 1.33                           |
| Abrens        | .68                             | .94                             | 1.33                           |
| Zachariasen   | .68                             | .98                             | 1.33                           |
| Average       | .685                            | .962                            | 1.33                           |

work and from work involving determination of ionic radii appear to be too inaccurate to permit establishing with certainty a valid relationship between ionic radii of the cations and modulus of elasticity, if one exists.

d.  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  System

Three series of compositions in the  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  system were formulated and tested. Most of these glasses were made up to have the same mole percentages as glasses previously studied except that  $\text{MgO}$  was substituted for the Group 1 oxides. One mole of  $\text{MgO}$  is equivalent to one mole of Group 1 oxides in that it introduces the same amount of oxygen. Therefore, the same mole percentages were selected for study in spite of the difference in valence of the added cation.

The compositions prepared and the properties obtained are listed in Table XV.

Beyond a general tendency for the higher silica glasses to be stronger, there does not appear to be a meaningful relationship between composition and tensile strength. It is felt that fiber forming difficulties caused the production of fibers so variable in quality that any relationship that might exist could not be found. Some glasses were not run at the optimum viscosity because of bushing temperature limitations. Others devitrified very readily at the optimum viscosity and were run at higher temperatures than normal. Some were probably actually fiberized below the liquidus.

TABLE XV

Compositions and Properties of  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  Glasses

| <u>Glass #</u>                                | <u>X-2374</u>  | <u>X-2369</u> | <u>X-2287</u>                | <u>X-2200</u>  | <u>X-2201</u>  | <u>X-2286</u>                 | <u>X-2202</u>  |
|-----------------------------------------------|----------------|---------------|------------------------------|----------------|----------------|-------------------------------|----------------|
| Composition                                   |                |               |                              |                |                |                               |                |
| Mole % $\text{SiO}_2$                         | 75.00          | 75.00         | 75.00                        | 75.00          | 75.00          | 75.00                         | 75.00          |
| $\text{Al}_2\text{O}_3$                       | 4.20           | 5.25          | 6.25                         | 9.38           | 12.50          | 13.40                         | 15.00          |
| $\text{MgO}$                                  | 20.80          | 19.75         | 18.75                        | 15.63          | 12.50          | 11.10                         | 10.00          |
| Ave. Tensile<br>Strength $\times 10^{-3}$ psi | 512            | N. D.         | 434                          | 630            | 568            | 611                           | 609            |
| Ave. Modulus<br>$\times 10^{-6}$ , psi        | 12.22          | 12.08         | 11.60, 11.82<br>11.82, 12.16 | 12.00<br>11.98 | 12.40          | 12.62                         | 12.94          |
| Forming Temp °F                               | 3215           | 3155          | 3095                         | 3215           | 3095           | 3075                          | 3060           |
| <u>Glass #</u>                                | <u>X-2289</u>  | <u>X-2373</u> | <u>X-2203</u>                | <u>X-2372</u>  | <u>X-2204</u>  | <u>X-2288</u>                 | <u>X-2205</u>  |
| Composition                                   |                |               |                              |                |                |                               |                |
| Mole % $\text{SiO}_2$                         | 66.00          | 66.00         | 66.00                        | 66.00          | 66.00          | 66.00                         | 66.00          |
| $\text{Al}_2\text{O}_3$                       | 6.00           | 9.71          | 12.00                        | 14.57          | 17.00          | 18.90                         | 20.00          |
| $\text{MgO}$                                  | 28.00          | 24.29         | 22.00                        | 19.43          | 17.00          | 15.10                         | 14.00          |
| Ave. Tensile<br>Strength $\times 10^{-3}$ psi | 525            | N. D.         | 525                          | N. D.          | 468            | 515                           | 449            |
| Ave. Modulus<br>$\times 10^{-6}$ , psi        | 12.84<br>12.88 | 12.63         | 12.48<br>12.52               | 12.80          | 12.82<br>12.95 | 12.95<br>13.22                | 13.50<br>13.66 |
| Forming Temp °F                               | 2830           | 2770          | 3020                         | 2975           | 2875           | 2770                          | 3095           |
| <u>Glass #</u>                                | <u>X-2291</u>  | <u>X-2376</u> | <u>X-2206</u>                | <u>X-2375</u>  | <u>X-2207</u>  | <u>X-2290</u>                 | <u>X-2208</u>  |
| Composition                                   |                |               |                              |                |                |                               |                |
| Mole % $\text{SiO}_2$                         | 57.00          | 57.00         | 57.00                        | 57.00          | 57.00          | 57.00                         | 57.00          |
| $\text{Al}_2\text{O}_3$                       | 12.00          | 14.90         | 17.00                        | 19.10          | 21.50          | 23.00                         | 24.50          |
| $\text{MgO}$                                  | 31.00          | 28.10         | 26.00                        | 23.90          | 21.50          | 20.00                         | 18.50          |
| Ave. Tensile<br>Strength $\times 10^{-3}$ psi | 541            | 545           | 510                          | 494            | 430            | Could not form<br>test fibers |                |
| Ave. Modulus<br>$\times 10^{-6}$ , psi        | 13.86<br>14.00 | 13.50         | 13.20<br>13.08               | 13.42          | 13.56          | 13.82                         | 14.26          |
| Forming Temp. °F                              | 2505           | 2585          | 2735                         | 2560           | 2850           | 2735                          | 2975           |

The difficulty of forming fibers also made it impractical to relate composition with viscosity as shown by forming temperatures. In addition, there was a change of bushings during the series, resulting in an abrupt change in bushing temperature readings, probably as a result of the drift in reading of the older thermocouple.

The preparation of fibers suitable for modulus measurement is less difficult than the preparation of good fibers for tensile strength tests. As far as is known, fiber modulus is not greatly affected by forming difficulties, so long as seeds and crystalline material do not appear in the fibers. Therefore, it seems probable that the results obtained on the modulus measurements are valid within the limitations of the measurement as described later. Results are shown graphically in Figure 7 (Page 33). Note that modulus is plotted vs. 2 Al/Mg. On the basis of the oxygen available for coordination with the silicon and aluminum ions, this ratio is equivalent to the ratio Al/Na<sup>+</sup> used on the first series of glasses studied. It is apparent that results for this system are far different from those for Group 1 alkalis. It is possible that there is an inflection of the curves at the point where the 2 Al/Mg ratio is one, but it is very small if present at all. However, there is a marked break in the curves at some lower ratio. The location of the break varies with the silica content, being at a lower value for the higher silica levels. When modulus is plotted vs. (Si + Al)/O, the curves resemble those for modulus vs. 2 Al/Mg. They do not show inflections at either 0.5 or 0.475 as did the curves for glasses containing Group 1 oxides.

Density measurements were made on most of the MgO glasses in an effort to find another property which showed breaks when plotted against composition similar to those found in the modulus vs. composition curves. Results are shown in Figure 16. It is apparent that density changes very little as the 2 Al/Mg ratio changes. The spread in results is great enough to mask any effects that might be present, although there appears to be an inflection at the 57% SiO<sub>2</sub> level corresponding to that found for modulus. It would be possible to reduce the experimental errors leading to the spread in results by greater care in all details starting with the preparation of the glass. However, it appears that any breaks that may exist would be so slight that even precise data would be rather inconclusive. Further work was therefore not done.

#### e. SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-CaO System

A number of glasses in this system were prepared to determine whether other oxides in Group 2 behave in a manner similar to magnesia. The compositions melted and results obtained are given in Table XVI.

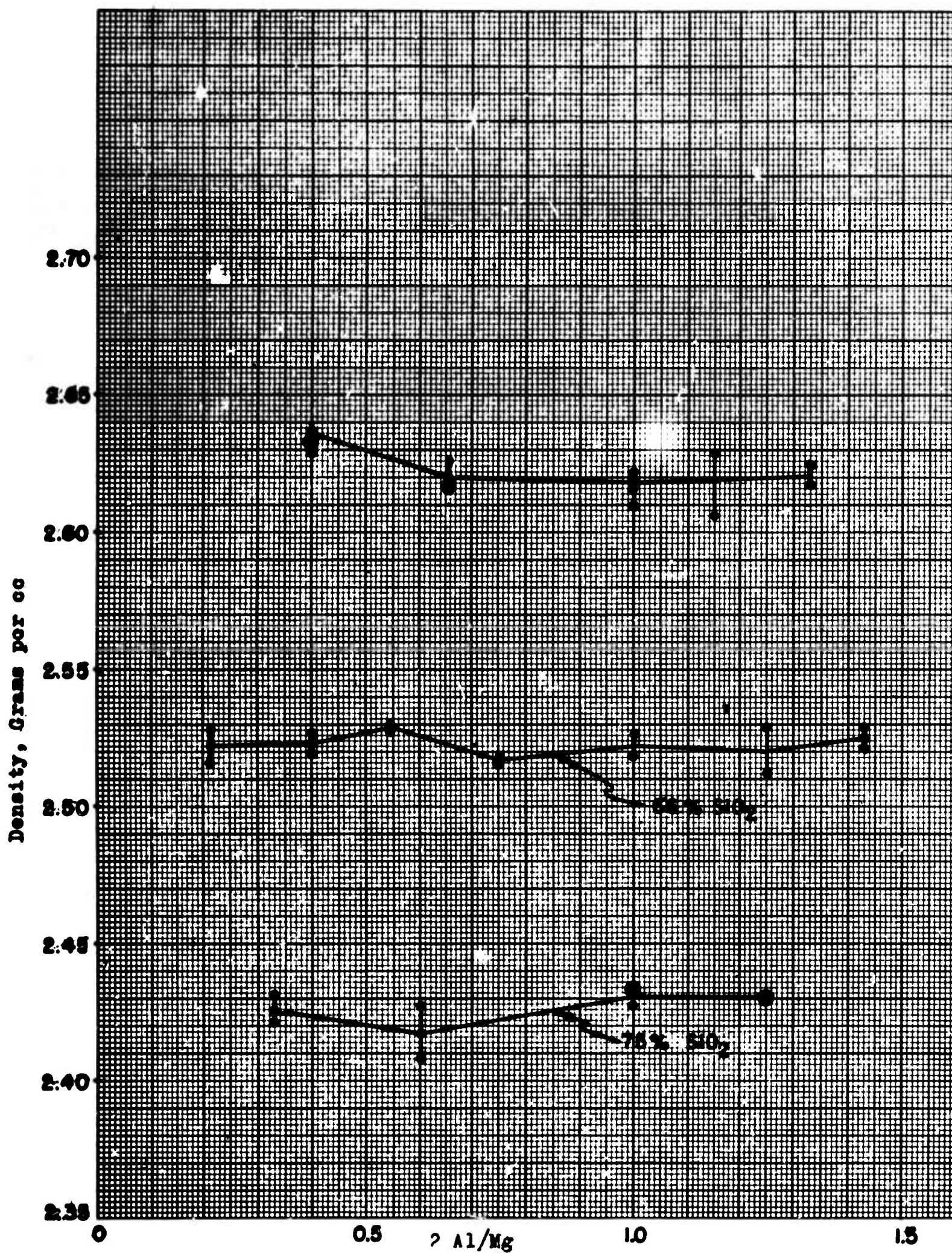


Figure 18 Density Vs. 2 Al/Mg Ratio

TABLE XVI

COMPOSITION AND PROPERTIES OF GLASSES IN THE  
SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-CaO SYSTEM

|                                              | <u>X-2315</u> | <u>X-2228</u> | <u>X-2229</u> | <u>X-2314</u> | <u>X-2230</u> | <u>X-2317</u> |
|----------------------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|
| Mole %                                       |               |               |               |               |               |               |
| SiO <sub>2</sub>                             | 75.00         | 75.00         | 75.00         | 75.00         | 75.00         | 66.00         |
| Al <sub>2</sub> O <sub>3</sub>               | 6.25          | 9.38          | 12.50         | 13.90         | 15.00         | 6.00          |
| CaO                                          | 18.75         | 15.63         | 12.50         | 11.10         | 10.00         | 28.00         |
| 2 Al/Ca                                      | .33           | .60           | 1.00          | 1.25          | 1.50          | .214          |
| Modulus, Ave.<br>x 10 <sup>-6</sup> , psi    | 11.30         | 11.10         | 10.92         | 11.28         | 11.50         | 12.20         |
| Modulus, Re-<br>check                        |               | 11.30         | 11.02         |               |               |               |
| Tensile Strength<br>x 10 <sup>-3</sup> , psi | 478           | 215           | 274           | 348           | 457           | 357           |
| Tensile Strength,<br>Recheck                 |               | 370           | 524           |               |               |               |
| Forming Temp,<br>°F                          | 3105          | 3215          | 3215          | 3215          | 3215          | 2645          |

TABLE XVI, CONTINUED)

|                                              | <u>X-2231</u> | <u>X-2232</u> | <u>X-2316</u> | <u>X-2233</u> | <u>X-2319</u> | <u>X-2234</u>    | <u>X-2235</u>    | <u>X-2318</u> | <u>X-2236</u>    |
|----------------------------------------------|---------------|---------------|---------------|---------------|---------------|------------------|------------------|---------------|------------------|
| Mole %                                       |               |               |               |               |               |                  |                  |               |                  |
| SiO <sub>2</sub>                             | 66.00         | 66.00         | 66.00         | 66.00         | 57.00         | 57.00            | 57.00            | 57.00         | 57.00            |
| Al <sub>2</sub> O <sub>3</sub>               | 12.00         | 17.00         | 18.90         | 20.00         | 12.00         | 17.00            | 21.50            | 23.00         | 24.50            |
| CaO                                          | 22.00         | 17.00         | 15.10         | 14.00         | 31.00         | 26.00            | 21.50            | 20.00         | 18.50            |
| 2 Al/Ca                                      | .545          | 1.00          | 1.25          | 1.43          | .397          | .654             | 1.00             | 1.15          | 1.324            |
| Modulus, Ave.<br>x 10 <sup>-6</sup> , psi    | 12.40         | 12.22         | 12.62         | 12.96         | 12.22         | 12.88            | 12.76            | 13.10         | 13.40            |
| Tensile Strength<br>x 10 <sup>-3</sup> , psi | 362           | 423           | 492           | 543           | 326           | 416              | 475              | 414           | 511              |
| Forming Temp,<br>°F                          | 2890          | 2955          | 2953          | 3000          | 2480          | 2735             | 2745             | 2795          | 2770             |
|                                              |               |               |               |               |               | Slight<br>Devit. | Slight<br>Devit. |               | Slight<br>Devit. |

A rather striking example of the dependence of tensile strength on the thermal history of the glass was observed. While running the center three members of each series, an unexpected result was obtained in that the glasses with the highest  $\text{SiO}_2$  contents appeared to give the lowest strengths. When additional glasses were run, the first glass in the 75%  $\text{SiO}_2$  series gave results higher than the others, which made the first results seem out of line. Two of the compositions were remelted for 24 hours at 2900° F. These conditions had earlier been found to give improved strengths with other glasses as reported in Section II. This melting treatment resulted in marked increases in the tensile strengths as indicated by the values for the tensile strength rechecks in Table XVI. There still appears to be a dip in the curve for the 75%  $\text{SiO}_2$  level at a 2 Al/Ca ratio of 0.6. However, results were too erratic to justify the conclusion that this was significant. Rather, it was concluded that the tensile strengths actually measured showed dependence upon thermal history great enough to mask any effects caused by glass structure changes resulting from glass composition alone.

Modulus is plotted vs. composition in Figure 19. There is definitely an inflection in the curves at the point where the 2 Al/Ca ratio is one. The curves were not extended over as large a composition range as was done for most of the series containing Group 1 oxides because of forming difficulties. However, enough points were obtained to show that the curves for the calcia glasses resemble those for the glasses containing Group 1 oxides, and that they do not resemble the curves for the series of glasses containing magnesia. Modulus values are higher, however, than those for the glasses containing the Group 1 oxides. In this, the calcia series is more similar to the magnesia series.

f.  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-ZnO}$  System

The unique results obtained for the modulus vs. composition relationship with the glasses containing MgO suggested that it would be interesting to study a similar system with ZnO replacing MgO.

The valence and ionic radii of zinc and magnesium are similar. The compositions studied and the results obtained are given in Table XVII.

Tensile strength and forming temperature data are not very reliable because of the difficulty associated with the forming of suitable fibers.

Modulus is plotted vs. composition in Figure 4 (Page 9). The curves are rather similar to those found for the MgO series of glasses. There is no clear indication of an inflection at the point where the 2 Al/Zn ratio is one. There is an inflec-

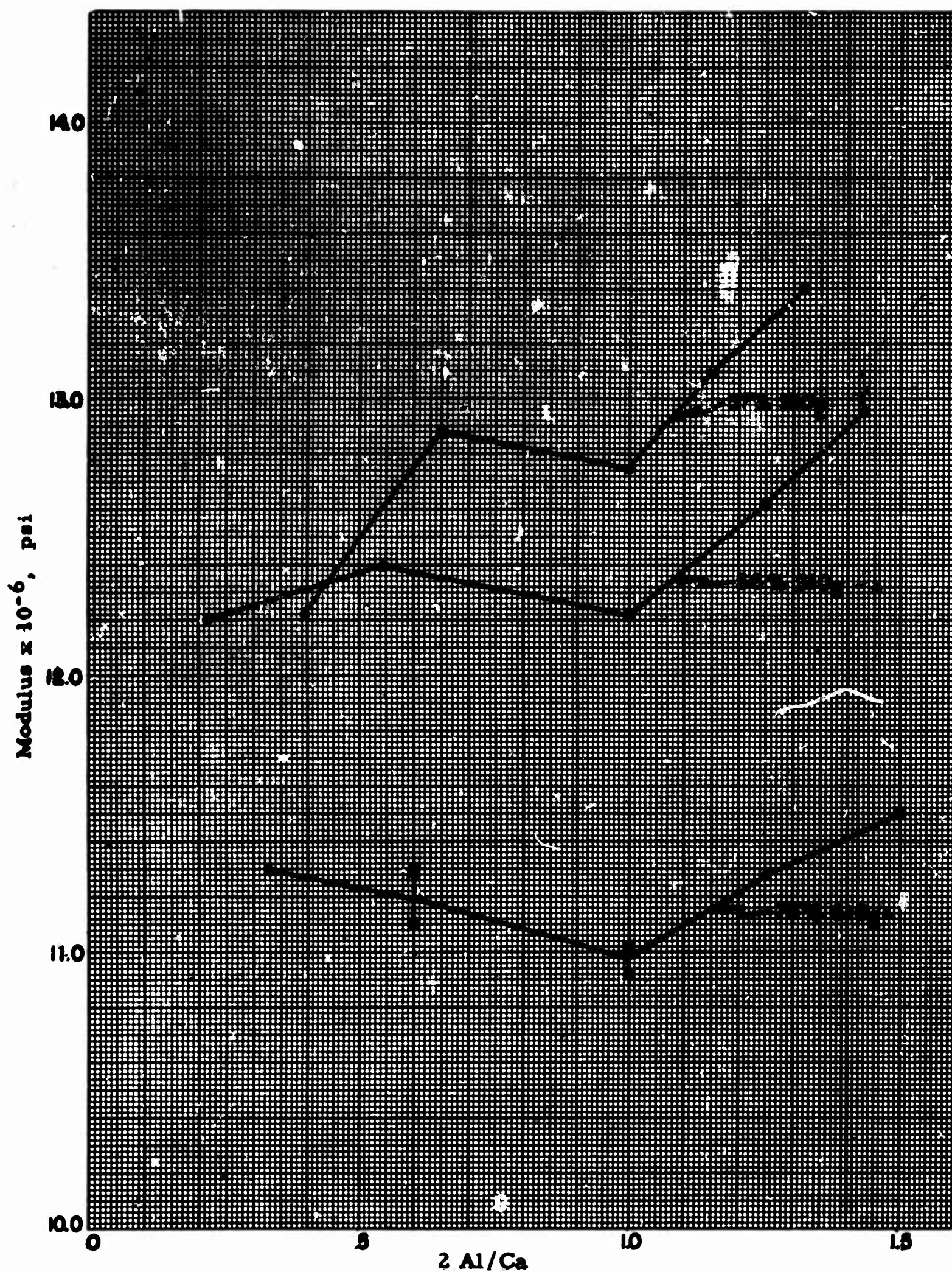


Figure 19 Modulus Vs.  $2 \text{ Al/Ca}$  Ratio for Silica-Alumina-Calcia Glasses

TABLE XVII

Compositions and Properties of SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-ZnO Glasses

| <u>ass #</u>                                   | <u>X-2386</u> | <u>X-2385</u>  | <u>X-2360</u>  | <u>X-2361</u>  | <u>X-2362</u>  |                |
|------------------------------------------------|---------------|----------------|----------------|----------------|----------------|----------------|
| Composition                                    |               |                |                |                |                |                |
| mole % SiO <sub>2</sub>                        | 75.00         | 75.00          | 75.00          | 75.00          | 75.00          |                |
| Al <sub>2</sub> O <sub>3</sub>                 | 4.20          | 6.25           | 9.38           | 12.50          | 15.00          |                |
| ZnO                                            | 20.80         | 18.75          | 15.63          | 12.50          | 10.00          |                |
| ve. Tensile<br>strength x 10 <sup>-3</sup> psi | 482           | 459            | 469            | 609            | 622            |                |
| ve. Modulus<br>10 <sup>-6</sup> , psi          | 11.62         | 11.26          | 11.66          | 11.78          | 12.38          |                |
| forming Temp, °F                               | 3150          | 3215           | 3215           | 3155           | 3040           |                |
| <u>ass</u>                                     | <u>X-2382</u> | <u>X-2381</u>  | <u>X-2363</u>  | <u>X-2380</u>  | <u>X-2364</u>  | <u>X-2365</u>  |
| Composition                                    |               |                |                |                |                |                |
| mole % SiO <sub>2</sub>                        | 66.00         | 66.00          | 66.00          | 66.00          | 66.00          | 66.00          |
| Al <sub>2</sub> O <sub>3</sub>                 | 6.00          | 9.71           | 12.00          | 14.57          | 17.00          | 20.00          |
| ZnO                                            | 28.00         | 24.29          | 22.00          | 19.43          | 17.00          | 14.00          |
| ve. Tensile<br>strength x 10 <sup>-3</sup> psi | N. D.         | 508            | 446            | 530            | 492            | 641            |
| ve. Modulus<br>10 <sup>-6</sup> , psi          | 13.58         | 13.22          | 12.80          | 12.90          | 13.15          | 13.96          |
| forming Temp. °F                               | -             | 2620           | 2850           | 2700           | 2850           | 3155           |
|                                                | Fibers opal.  |                |                |                |                | Devit.         |
| <u>ass #</u>                                   | <u>X-2387</u> | <u>X-2388</u>  | <u>X-2366</u>  | <u>X-2389</u>  | <u>X-2367</u>  | <u>X-2368</u>  |
| Composition                                    |               |                |                |                |                |                |
| mole % SiO <sub>2</sub>                        | 57.00         | 57.00          | 57.00          | 57.00          | 57.00          | 57.00          |
| Al <sub>2</sub> O <sub>3</sub>                 | 12.00         | 14.90          | 17.00          | 19.10          | 21.50          | 24.50          |
| ZnO                                            | 31.00         | 28.10          | 26.00          | 23.90          | 21.50          | 18.50          |
| ve. Tensile<br>strength x 10 <sup>-3</sup> psi | 418           | 567            | 408            | 545            | N. D.          | 441            |
| ve. Modulus<br>10 <sup>-6</sup> , psi          | 13.64         | 13.56          | 13.50          | 13.60          | 13.83          | 14.62          |
| forming Temp. °F                               | 2570          | 2505<br>Devit. | 2680<br>Devit. | 2610<br>Devit. | 3050<br>Devit. | 2850<br>Devit. |

tion in all curves at some lower value. The inflection points correspond to those found in the MgO system.

#### 4. Discussion of Results

Of all the data obtained, modulus measurements appear to be the most valuable. Tensile strength and fiber forming temperatures appear to be quite useful in the earlier study of the glasses containing  $\text{Na}_2\text{O}$ . Most of the glasses in that system ran well at attainable temperatures and required no special fiber forming techniques. The reliability of tensile strength and forming temperature data became less for the  $\text{K}_2\text{O}$  and  $\text{Li}_2\text{O}$  containing glasses because of the high forming temperatures of the former and the high devitrification rate and peculiar forming behavior of the latter. Difficulties in getting good data became even greater for the glasses containing divalent ions. Many of these glasses had to be run at the maximum safe temperature of the bushing, and even there perhaps at reduced speeds. Under these conditions, it is not likely that the glass can be conditioned to produce fibers of optimum strength, or that the fiber forming conditions themselves can be optimum. There is no opportunity for adjusting forming variables so as to find the conditions which produce optimum strengths. Other glasses devitrify so readily that they must be formed at the opposite boundary conditions for fiber formation, and here again no opportunity exists to change variables to determine optimum strengths. The measurement of tensile strengths under these conditions appeared justified for two reasons:

- a. It could not be known in advance how many glasses in a series would have to be formed under conditions such that strength values would not be near enough to the optimum to permit valid comparisons.
- b. Even non-optimum values might uncover areas of particular interest which would warrant more extensive investigation.

Certain of the variables associated with measuring forming temperature, such as drift in thermocouple EMF with aging or differences between bushings could have been taken account of and compensated for by known techniques. This was not done for two reasons:

- a. So many of the glasses had to be fiberized by resorting to arbitrary adjustments in temperature and pulling speed that useful comparisons between glasses would still not have been possible.
- b. Forming temperatures were of secondary interest, and were noted principally in the hope that they might serve as an additional variable sensitive to glass structure which might be used to corroborate conclusions drawn from modulus or strength measurements.

The modulus of elasticity seems to be relatively insensitive

to forming variables. Values obtained on fibers drawn with difficulty seemed generally to fall in line with results from more easily fiberized glasses provided only that the fibers were not hollow and did not contain crystalline inclusions. The spread in results for a given set of modulus determinations (ten measurements on five fibers) rarely exceed  $0.3 \times 10^6$  psi. On several occasions, a complete recheck of modulus measurements was made, as indicated in the tables, particularly Table XV (Page 64) which contains the data for the MgO series. Usually these repeat measurements checked each other within  $0.3 \times 10^6$  psi, exactly. In some cases, the repeat measurements were made on fibers produced from different bushings. The largest variation was found for glass X-2267 (Table XV, Page 64) for which average values of 11.60, 11.82, 11.82, and 12.16 were obtained. The total spread in average results here is  $0.56 \times 10^6$  psi. This does allow the possibility that differences in forming or testing conditions could influence results by this much. However, the variation was not related to any obvious difference in forming conditions.

For these reasons it is felt that in this study modulus measurements provided the most reliable and most valuable data for correlating properties with glass structure. It is apparent that for glasses containing the oxides  $\text{Li}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  and  $\text{CaO}$ , additions of  $\text{Al}_2\text{O}_3$  increases the modulus in a non-uniform manner. The rate increase is relatively large with small additions, becomes smaller or even negative with intermediate additions, and increases again at still larger additions. The change from small to larger rates of increase takes place where the ratio of aluminum ions to alkali ions is one. This is consistent with the theories of glass structure previously developed for the silica-alumina-soda glasses only. For increasing the modulus of a glass the desirability of these alkalis falls in the following order:  $\text{CaO}$ ,  $\text{Li}_2\text{O}$ ,  $\text{Na}_2\text{O}$ , and  $\text{K}_2\text{O}$ . This is in line with the ionic strength of these ions. Lithium, with its small radius, is the best of the Group 1 alkalis. The charge of two for the calcium ion explains its position ahead of lithium, even though it is a large ion.

$\text{MgO}$  and  $\text{ZnO}$  are nearly equivalent in effect and both are superior to the other ions for increasing modulus. The ratio of ionic charge to radius is higher for these ions than for those in the preceding group. It is logical that these ions should tend to tighten the glass structure and thus raise the modulus. It is probable that these ions participate somewhat in the glass network. This is inferred from the observation that the modulus vs. composition curves do not show a break at an aluminum to alkali ion ratio of one. These ions apparently interfere with the change in coordination number of the aluminum as oxygen is added, probably because they tie up some of the oxygen themselves. That these ions may possibly participate in the network itself has been postulated by other investigations on the basis of other evidence.

Ainsworth<sup>(6)</sup>, for instance, discussed this possibility in connection with his work on the relationship between glass compositions and hardness. He also found an anomaly with magnesium and zinc oxides when comparing the effect various oxides had on the diamond pyramid hardness of glass. He noted a general tendency for certain oxides from Groups 1, 2, and 3 to increase the hardness of a base glass by amounts that seemed usually to be in the same order as ionic strengths. He found that magnesium and zinc oxides increased hardness rather less than expected on this basis, and postulated that this might be explained by some participation by these ions in the glass network. It is noteworthy that he found these oxides rather less effective in increasing hardness than expected. Since hardness is likely to be related to modulus, this evidence seems to be contradictory to the findings of this study, which showed these oxides to be more effective than others in raising modulus. The explanation may lie in the base glass used. Ainsworth added the oxides studied to a base glass containing  $\text{SiO}_2$  and  $\text{Na}_2\text{O}$ , while the base glass used in the current study contained  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ .

## REFERENCES

- (1) R. M. McMarlin, R. L. Tiede, F. M. Veazie, High Strength - High Modulus Glass Fibers. Technical Report AFML-TR-65-90, Part I, March, 1965.
- (2) K. L. Lowenstein. Studies in the Composition and Structure of Glasses Possessing High Young's Moduli: Part 1. The Composition of High Young's Modulus Glass and the Function of Individual Ions in the Glass Structure. Physics and Chemistry of Glasses, Vol. 2, No. 3, p. 69-82, June, 1961.
- (3) D. E. Day and G. E. Rindone. Properties of Soda Aluminasilicate Glasses: I Refractive Index Density Molar Refractivity, and Infrared Absorption Spectra. J. Am. Ceram. Soc., V. 45, No. 10, p.489-496 (1962).
- (4) D. E. Day and G. E. Rindone. Properties of Soda Aluminasilicate Glasses: II. Internal Friction, Ibid, p. 496-504.
- (5) A. A. Appen and S. S. Kayalova. Surface Tension of Alkali-Silicate Melts, Dokl. Akad. Nauk. SSSR, 145(3) 592-94 (1962). Ceram. Abs. V. 48, No. 4, Apr 1965, p. 140.
- (6) L. Ainsworth. The Diamond Pyramid Hardness of Glass in Relation to Strength and Structure of Glass. Part II. Silicate Glasses. J. Soc. of Glass Tech., V 38, 1954, p. 501-535T.

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |  |                                                                                                                                                   |                      |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--|---------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|
| 1. ORIGINATING ACTIVITY (Corporate author)<br>Owens-Corning Fiberglas Corp.<br>Granville, Ohio                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |  | 2a. REPORT SECURITY CLASSIFICATION<br>Unclassified                                                                                                |                      |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |  | 2b. GROUP                                                                                                                                         |                      |
| 3. REPORT TITLE<br>High Strength, High Modulus Glass Fibers                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |  |                                                                                                                                                   |                      |
| 4. DESCRIPTIVE NOTES (Type of report and inclusive dates)<br>Technical Documentary Report March 1965 - June 1966                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |  |                                                                                                                                                   |                      |
| 5. AUTHOR(S) (Last name, first name, initial)<br>McMarlin, R. M., Tiede, R. L., Veazie, F. M.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |  |                                                                                                                                                   |                      |
| 6. REPORT DATE<br>June 1966                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |  | 7a. TOTAL NO. OF PAGES<br>74                                                                                                                      | 7b. NO. OF REFS<br>6 |
| 8a. CONTRACT OR GRANT NO.<br>AF 33(615)-1370<br>A. PROJECT NO. 7320<br>c. 7340<br>d.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |  | 8a. ORIGINATOR'S REPORT NUMBER(S)<br>None<br>8b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)<br>AFML-TR-65-90 Part II |                      |
| 10. AVAILABILITY/LIMITATION NOTICES<br>Qualified requestors may obtain copies of this report from the Defense Documentation Center (DDC), (formerly ASTIA), Cameron Station, Bldg. 5, 5010 Duke St., Alexandria 4, Va. Dissemination outside the Department of Defense or to recipients other than government defense contractors is prohibited. DDC release to CFSTI not authorized.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |  |                                                                                                                                                   |                      |
| 11. SUPPLEMENTARY NOTES                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |  | 12. SPONSORING MILITARY ACTIVITY                                                                                                                  |                      |
| 13. ABSTRACT<br>The purpose of this investigation has been the study of glass compositions capable of producing glass fibers having high tensile strength-high modulus of elasticity properties which are also low in density. Of the many compositions studied, those which have the best promise of meeting the required properties are located in the $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$ -BeO system. In particular, compositions have been developed in this system with tensile strengths of 680-715,000 psi, moduli of $14 \times 10^6$ psi, and bulk densities of 2.41-2.50. These properties produce fibers with specific strengths and specific moduli in excess of any currently available glass fiber. Successful multifilament manufacture of glasses of this nature was demonstrated and unidirectional filament wound composites were fabricated and tested. In a basic study aimed at relating glass properties, composition, and structure, three and four component systems were studied consisting of $\text{SiO}_2$ and $\text{Al}_2\text{O}_3$ plus one of the following: $\text{K}_2\text{O}$ , $\text{Li}_2\text{O}$ , $\text{K}_2\text{O} + \text{Li}_2\text{O}$ , $\text{MgO}$ , $\text{CaO}$ , $\text{ZnO}$ and $\text{BeO}$ . For glasses containing $\text{K}_2\text{O}$ , $\text{Li}_2\text{O}$ , $\text{K}_2\text{O} + \text{Li}_2\text{O}$ , or $\text{CaO}$ , curves of modulus vs. the ratio of aluminum to added ion showed breaks at the point where one oxygen ion had been added per two aluminum ions. This was related to a proposed glass structure involving a change of coordination number of aluminum from four to six, with a corresponding change in the function of this ion in the structure. $\text{BeO}$ , $\text{MgO}$ and $\text{ZnO}$ were similar to each other in behavior, but differed from the other oxides. They showed breaks in composition vs. modulus curves at different points than the other oxides, with the inflection point changing with $\text{SiO}_2$ content. These oxides increased modulus in the following order (BeO highest): $\text{BeO}$ , $\text{MgO}$ , $\text{ZnO}$ , $\text{CaO}$ , $\text{Li}_2\text{O}$ , $\text{Na}_2\text{O}$ , $\text{K}_2\text{O}$ . |  |                                                                                                                                                   |                      |

| 14. KEY WORDS                                         | LINK A |    | LINK B |    | LINK C |    |
|-------------------------------------------------------|--------|----|--------|----|--------|----|
|                                                       | ROLE   | WT | ROLE   | WT | ROLE   | WT |
| Fibers, glass, oxides, composition, strength, modulus |        |    |        |    |        |    |

#### INSTRUCTIONS

1. **ORIGINATING ACTIVITY:** Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization (corporate author) issuing the report.
- 2a. **REPORT SECURITY CLASSIFICATION:** Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.
- 2b. **GROUP:** Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.
3. **REPORT TITLE:** Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.
4. **DESCRIPTIVE NOTES:** If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.
5. **AUTHOR(S):** Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.
6. **REPORT DATE:** Enter the date of the report as day, month, year, or month, year. If more than one date appears on the report, use date of publication.
- 7a. **TOTAL NUMBER OF PAGES:** The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.
- 7b. **NUMBER OF REFERENCES:** Enter the total number of references cited in the report.
- 8a. **CONTRACT OR GRANT NUMBER:** If appropriate, enter the applicable number of the contract or grant under which the report was written.
- 8b, 8c, & 8d. **PROJECT NUMBER:** Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.
- 9a. **ORIGINATOR'S REPORT NUMBER(S):** Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.
- 9b. **OTHER REPORT NUMBER(S):** If the report has been assigned any other report numbers (either by the originator or by the sponsor), also enter this number(s).
10. **AVAILABILITY/LIMITATION NOTICES:** Enter any limitations on further dissemination of the report, other than those

imposed by security classification, using standard statements such as:

- (1) "Qualified requesters may obtain copies of this report from DDC."
- (2) "Foreign announcement and dissemination of this report by DDC is not authorized."
- (3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through \_\_\_\_\_."
- (4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through \_\_\_\_\_."
- (5) "All distribution of this report is controlled. Qualified DDC users shall request through \_\_\_\_\_."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. **SUPPLEMENTARY NOTES:** Use for additional explanatory notes.

12. **SPONSORING MILITARY ACTIVITY:** Enter the name of the departmental project office or laboratory sponsoring (paying for) the research and development. Include address.

13. **ABSTRACT:** Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.