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HIGH STRENGTH - HIGH MODULUS GLASS FIBERS

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OWENS-CORNING FIBERGLAS CORPORATION

TECHNICAL REPORT AFML-TR-65-90
March 1965

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio
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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 1964 through February 1965.

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. P. Munro Vesie, as Program Manager, with Mr. Robert McMarlin and Mr. Ralph Tiede as Project Managers. They were assisted by Messrs. Austin Walpole, Glen Lucas, and John Brookbank.

Manuscript released by authors for publication as an RTD Technical Report

This technical documentary report has been reviewed and is approved.

R. G. SPAIN, Chief
Fibrous Materials Branch
Nonmetallic Materials Division
ABSTRACT

The results of the present investigation have shown that glass fibers drawn from compositions located in the SiO₂-Al₂O₃-ZnO system possess tensile strengths in excess of 800,000 psi. Glass fibers drawn from compositions in the SiO₂-Al₂O₃-MgO system have given similar results, particularly at fiber diameters in the 0.00015 to 0.00020 inch range.

The modulus of elasticity of glass fibers obtained from compositions located in the SiO₂-Al₂O₃-MgO system can be increased to $16.0 \times 10^6$ psi. This is accomplished by lowering the fiber diameter to the 0.00015 to 0.00020 inch range.

In the SiO₂-Al₂O₃-Na₂O system, the fiber forming temperature, the tensile strength and the modulus of elasticity change with composition in a manner which gives a break in the composition vs. property curves at the point where the Al to Na ratio is 1. This is related to the change in the coordination number of the aluminum at this ratio which has been proposed in the literature.

The properties measured for glasses in the SiO₂-TiO₂-K₂O system do not appear to lead to as clear a concept of glass structure. Properties vary gradually with composition, with no evidence of a sharp break at any point. There is some indication that the rate of change of properties is dominated more by the silica content than is the case in the SiO₂-Al₂O₃-Na₂O system.
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HIGH STRENGTH - HIGH MODULUS GLASS FIBERS

INTRODUCTION

The present study was undertaken for the purpose of preparing and studying glass compositions which would produce glass fibers possessing high tensile strengths and high moduli of elasticity. An additional study was initiated which sought to establish a relationship between tensile strength and the composition and probable constitution of selected glasses. Results of this additional study are presented separately under the title "Relationship Between Composition and Properties of Selected Glasses."

Emphasis in the study of high strength - high modulus glass fibers was placed on glasses of two general classifications. In the first classification, SiO$_2$-Al$_2$O$_3$-R$_x$O glasses were prepared in which the alumina to R$_x$O ratio was maintained at one on a mol basis. In the second classification, SiO$_2$-Al$_2$O$_3$-R$_0$ glasses were prepared in which the alumina to R$_0$ ratio varied from 0.5 to 2.0. The R$_0$ component included the alkali and alkaline earth oxides. Other modifications of both classifications were made using several other glass formers and modifiers.

The study initially proceeded with the preparation of glass compositions in the SiO$_2$-Al$_2$O$_3$-MgO system, notably S-994 and related glasses. Subsequent studies were then made by substituting alkali and alkaline earth oxides for all or part of the MgO in the base SiO$_2$-Al$_2$O$_3$-MgO glasses.
II

DISCUSSION

A. Study of SiO$_2$-Al$_2$O$_3$-R$_x$ Glasses

1. Glass Preparation, Fiberization, and Testing

Each glass composition was prepared by dry mixing the appropriate end member materials which were of at least U.S.P. grade. The dry batch was melted in precious metal crucibles. Temperatures used were 2900°F for two hours and 3200°F for an additional hour or 2900°F for 24 hours. Both methods gave equivalent results. Each batch was removed from its crucible and remixed at least once during the melting operation. All glasses were cooled by plunging the hot crucible directly from the furnace into a cold water bath. Fibers were obtained from each glass composition by remelting the prepared cullet in a one-hole precious metal bushing capable of reaching temperatures in excess of 3200°F.

To test the fibers, virgin filaments were captured by hand in the usual manner and, to determine tensile strength, were wound on a stainless steel fork. Each fork normally contained 6-8 fiber lengths for study after this operation. The apparatus for measuring tensile strength was designed to test one fiber at a time, recording essentially the load elongation diagram for each fiber. The fibers were loaded by a screw-driven cross-head at a constant rate of strain of 0.25 in./in./minute applied to a 2 inch gage length of fiber. The load cell consisted of a cantilever beam with a linear variable differential transformer (LVDT) as the displacement sensing element. The load cell was calibrated by dead weight loading. The LVDT was powered by a strain gage amplifier. The signals from the LVDT were first rectified through matched pairs of germanium diodes, then passed through gain and zero potentiometers and recorded on a single channel recorder (Esterline-Angus).

The fiber diameter of each fiber length (6-8 measurements) was determined from a short length of fiber remaining on the fork after mounting. The fibers were mounted on a microscope slide in a liquid of appropriate index of refraction and their diameters measured under a 430 power microscope with the aid of a micrometer eyepiece.

The modulus of elasticity of a particular glass composition was determined by direct measurement of the elongation of an unannealed fiber under load. In most cases, these fibers had diameters of approximately 0.00060 to 0.00080.

2. Tensile Strength and Modulus of Elasticity Results

a. Glasses located in the SiO$_2$-Al$_2$O$_3$-MgO system were studied extensively. The composition locations are shown in Figure 1, which represents the phase diagram of this system and shows the boundaries between primary phase fields as straight lines. The compositions studied are located at the circled points. The theoretical glass compositions studied in the system are shown
### TABLE I
THEORETICAL GLASS COMPOSITIONS AND FIBER PROPERTIES

<table>
<thead>
<tr>
<th>GLASS NO.</th>
<th>OXIDES (a. weight % - b. mol %)</th>
<th>AVERAGE TENSILE STRENGTH x 10^3 psi</th>
<th>MODULUS x 10^6 psi</th>
<th>AVERAGE DIAMETER x 10^-5 in.</th>
<th>FORMING TEMP. °F</th>
<th>NO. OF FIBERS</th>
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### TABLE I (Continued)

| GLASS NO. | SiO₂ | Al₂O₃ | MgO | Li₂O | Na₂O | K₂O | BeO | CaO | ZnO | SrO | BaO | AVERAGE TENSILE STRENGTH MODULUS | AVERAGE DIAMETER TEMP. | NO. OF FIBERS |
|-----------|------|-------|-----|------|------|-----|-----|-----|-----|-----|-----| x 10³ psi | x 10⁶ psi | x 10⁻⁵ in. °F |
| X-1831 a | 65.0 | 8.8   | 6.4 |      |      |     |     |     |     |      |      | 26.3 | 12.8 | 226 | 8.7 | 31 | 3115 | 16 |
| X-1831 b | 60.7 |       |     |     |      |     |     |     |     |      |      | 682  | 13.7 | 37  | 3000 | 16 |
| X-1851 a | 65.0 | 26.1  | 16.8|      |      |     |     |     |     |      |      | 11.5 | 26.0 | 327 | 14.0 | 34 | 3050 | 12 |
| X-1851 b | 66.3 |       |     |     |      |     |     |     |     |      |      |      | 5.9  | 26.0 | 635 | 13.2 | 39 | 3025 | 17 |
| X-1891 a | 71.2 | 5.9   | 14.4|      |      |     |     |     |     |      |      | 635  | 13.2 | 39  | 3025 | 17 |
| X-1891 b | 67.1 |       |     |     |      |     |     |     |     |      |      | 407  | 12.1 | 36  | 2735 | 17 |
| X-1835 a | 65.8 | 24.4  | 5.1 | 3.7 |      |     |     |     |     |      |      | 532  | 11.0 | 40  | 2850 | 15 |
| X-1835 b | 66.7 | 5.1   | 15.6| 7.9 | 7.9  |     |     |     |     |      |      | 413  | 10.6 | 42  | 2955 | 17 |
| αX-1836 a| 63.3 | 4.9   | 7.5 |      |      |     |     |     |     |      |      | 604  | 12.6 | 40  | 2795 | 17 |
| αX-1836 b| 66.7 | 4.9   | 7.5 |      |      |     |     |     |     |      |      | 542  | 12.9 | 39  | 2955 | 16 |
| X-1837 a | 60.7 | 15.6  | 7.9 | 7.9 |      |     |     |     |     |      |      | 391  | 11.7 | 43  | 2900 | 18 |
| X-1837 b | 66.7 | 15.6  | 7.9 |      |      |     |     |     |     |      |      | 16.7 | 7.9 | 485 | -   | 44 | 2900 | 16 |
| X-1840 a | 60.2 | 11.9  | 7.9 |      |      |     |     |     |     |      |      | 638  | 12.4 | 33  | 2850 | 16 |
| X-1840 b | 60.7 | 11.9  | 7.9 |      |      |     |     |     |     |      |      | 504  | 11.7 | 33  | 2795 | 16 |

**Notes:**
- The table continues with more rows of similar data.
- The content represents a compilation of oxide compositions and mechanical properties of various glasses.
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<td>P₂O₅</td>
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<td>GeO₂</td>
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in Table 1, which also lists pertinent physical properties for each glass. On Table 1, see glass numbers X-994, X-1751 through X-1763, X-1888 through X-1890, X-1898 through X-1903, X-1657 and X-1659.

Most of the data presented describes glass compositions with either constant weight percentages of SiO₂ and variable Al₂O₃ to MgO ratios or constant Al₂O₃ to MgO ratios and variable weight percentages of SiO₂. These data are summarized graphically in Figures 2 and 3.

Figure 2 indicates that at a constant SiO₂ content of 65% by weight, the tensile strength of the series of compositions increases with an increasing Al₂O₃ to MgO ratio. The increase continues until a maximum is reached at an Al₂O₃ to MgO ratio of 1. At this point it is postulated that additional Al₂O₃ enters sixfold coordination in the glass structure. The data show a reverse change in slope at this point in the curve, which indicates such a structural change is possible and does take place. Increasingly higher forming temperatures and much greater devitrification tendencies above the Al₂O₃ to MgO ratio of 1.0 also make it difficult to interpret the data obtained, but there is a good indication that beyond 1.5 mol % the tensile strength is beginning to increase again at a lower rate.

Tensile strength results for X-994 as listed in Table 1 show a range of values. It is felt that these results are indicative of the structural sensitivity of the glass at this point (Al₂O₃/MgO = 1.0). Strength values for other glasses at this ratio have shown similar variability and reinforce the view that slight changes in composition produce fairly large changes in strength.

The modulus values plotted in Figure 2 show no clear-cut trend. There appears to be an over-all increase in modulus with increasing Al₂O₃ to MgO ratio.

Figure 3 indicates that at a constant Al₂O₃ to MgO ratio of 1.0 and a variable SiO₂ content by weight the tensile strength of the series of compositions increases with an increase in the SiO₂ content. The increase is continuous up to a SiO₂ level of from 60-65% where the tensile strength begins to drop again. Again increased forming temperatures and greater devitrification tendencies are found at the higher SiO₂ levels. The plotted value beyond 70% by weight SiO₂ may therefore not be entirely correct.

The high tensile strength peak for X-1760 and the range of values for X-994 are again indicative of the composition sensitivity of these glasses at this point.

The modulus values appear to follow the trend established by the strength values, with maximum values located in the 60-70% SiO₂ range.
Tensile Strength, Forming Temperature, and Modulus of Elasticity as Effected by the Mol. Ratio of Al₂O₃ to MgO (SiO₂-Al₂O₃-MgO Glass; Constant 65% by wt. SiO₂)

FIGURE 2 Tensile Strength, Forming Temperature, and Modulus as Effected by the Mol Ratio of Al₂O₃ to MgO
Tensile Strength, Forming Temperature, and Modulus as Effected by the SiO₂ Content
(SiO₂-Al₂O₃-MgO
Class: Constant Al₂O₃/MgO = 1.0)

FIGURE 3 Tensile Strength, Forming Temperature, and Modulus as Effected by the SiO₂ Content
The tensile strength values of the remaining glasses which are not plotted tend to join out the trends established in Figures 2 and 3. These are as follows:

1. The glasses with the best forming characteristics and highest tensile strengths are located at an approximate Al$_2$O$_3$ to MgO ratio of approximately 1.0 and at a SiO$_2$ level ranging from 60-70% by weight.

2. Glasses with Al$_2$O$_3$ to MgO ratios much less than 1.0 do not have desirable tensile strengths.

3. Glasses with Al$_2$O$_3$ to MgO ratios much greater than 1.0 do not have high strengths because of fiberization difficulties and greater devitrification tendencies.

4. Glasses within the desirable composition limits (1) have tensile strength values which are sensitive to slight structural changes because of the changes in coordination of the Al$^{+++}$ ion in this area. It is therefore postulated that there is a structural link between the coordination of the Al$^{+++}$ ion in the glass and the tensile strength and modulus of filaments made from the glass.

b. SiO$_2$-Al$_2$O$_3$-R$_x$O Compositions

Table 1 lists the theoretical compositions and properties of a series of glasses containing SiO$_2$, Al$_2$O$_3$, and R$_x$O (where R$_x$O is Li$_2$O, K$_2$O, BeO, CaO, ZnO, SrO, or BaO.) Attention is directed to glasses numbered X-1764 through X-1770, X-1785 and X-1786, X-1795 through X-1799, X-1600, X-1620 through X-1623, X-1824 through X-1631, X-1651 and X-1852, and X-1691 through X-1693.

Figure 4 summarizes the properties of fibers produced from these glasses. The highest average strength and modulus value in each series of compositions has been compared to the range of values established for the SiO$_2$-Al$_2$O$_3$-MgO series. The tensile strength values plotted for the alkali oxide substitutions show a continuous strength decrease in going from MgO to Li$_2$O to K$_2$O. A similar decrease occurs in the modulus values. It is postulated that the Li$^+$ ion causes a reduction because its valence is only one and it has a much lower field strength than Mg$^{++}$. Similarly, the $X^+$ ion has a valence of only one, has an even lower field strength than Li$^+$, and has an ionic radius about twice as large as Mg$^{++}$.

The alkaline earth oxide substitutions for MgO show a continuous decrease in strength. Ranking the alkaline earth ions by ionic size and field strength may help account for the trend in the tensile strength.
<table>
<thead>
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<th>Ion</th>
<th>Ionic Size</th>
<th>Field Strength</th>
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<td>Be$^{++}$</td>
<td>0.31</td>
<td>16.7</td>
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<tr>
<td>Mg$^{++}$</td>
<td>0.65</td>
<td>4.5</td>
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<tr>
<td>Zn$^{++}$</td>
<td>0.74</td>
<td>5.6</td>
</tr>
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<td>Ca$^{++}$</td>
<td>0.99</td>
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<td>Sr$^{++}$</td>
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<td>1.6</td>
</tr>
<tr>
<td>Ba$^{++}$</td>
<td>1.35</td>
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</table>

The Zn$^{++}$ ion, though not an alkaline earth ion, is included to show its similarity to Mg$^{++}$. It would thus be expected to act like Mg$^{++}$. This is the case, as shown in Figure 4, at least for tensile strength values.

With such a trend established, it would be expected that a SiO$_2$-Al$_2$O$_3$-BeO glass would show outstanding strength properties. There is some indication that such may be the case, but no final values are yet available. Glasses of this type have enhanced devitrification and phase separation tendencies, which would be explained by the extremely high field strength of Be$^{++}$. Currently, such tendencies have made fiberization of good quality fibers difficult.

The plot of modulus of elasticity values for the alkaline earth oxide substitutions show a variable pattern. The BeO addition produces the best results, with SrO, surprisingly, a close second. Unfortunately, the high modulus values do not correspond to the strongest glasses in the series.

Summarizing the results of the study in which alkali oxides and alkaline earth oxides were substituted for MgO in the SiO$_2$-Al$_2$O$_3$-MgO series of glasses, it should be noted that:

1. The substitution of ZnO for MgO produced the only glasses with tensile strength values comparable to those in the SiO$_2$-Al$_2$O$_3$-MgO system.
2. When substituting alkali oxides for MgO, the tensile strength decreased as the size of the ion increased.
3. When substituting alkaline earth oxides for MgO, the tensile strength decreased as the size of the ion increased. The effect of BeO has not yet been definitely determined and should not be included in this conclusion.
4. None of the oxide substitutions for MgO improved the modulus of elasticity above that associated with glasses in the SiO$_2$-Al$_2$O$_3$-MgO system, except for the BeO substitution.

This is attributed to the small size and high field strength of the Be$^{++}$ ion.

**Table 1**: lists the theoretical compositions and properties of a series of glasses based on the X-994 composition where R$_x$O.
FIGURE 4 Variation in Tensile Strength and Modulus with $R_x0$ Substitutions for MgO

(Base Glass: $SiO_2$-$Al_2O_3$-$MgO)$
component is substituted for half the MgO on a mol basis. 
($\text{P}_x\text{O}_y = \text{Li}_2\text{O}, \text{Na}_2\text{O}, \text{K}_2\text{O}, \text{RbO}, \text{CsO}, \text{ZnO}, \text{SrO}, \text{BaO}$.) Attention 
is directed to glasses numbered X-1635 through X-1896.

Figure 5 summarizes the property data. The value of tensile 
strength and modulus for each oxide addition is compared to the 
range of values established for the SiO$_2$-Al$_2$O$_3$-MgO glass series. 
No trend is evident except to note that all of the additions 
tend to reduce the tensile strength of the SiO$_2$-Al$_2$O$_3$-MgO series 
of glasses. The trend of the modulus curve cannot be explained, 
except to note that the glasses with higher modulus values do not 
correspond to those with high tensile strengths, except in the 
case of the SiO$_2$-Al$_2$O$_3$-MgO-ReO glass.

1. SiO$_2$-Al$_2$O$_3$-MgO Glasses with Additions of Other Glass formers 
and Intermediates

Table 1 lists the theoretical compositions and properties of a 
series of glasses containing SiO$_2$-Al$_2$O$_3$-MgO and one other glass 
former or intermediate oxide (P$_2$O$_5$, TiO$_2$, ZrO$_2$, HfO$_2$, or GeO$_2$).

On Table 1 see the following glass numbers:

- Glasses containing P$_2$O$_5$: X-1842 through X-1845, X-1849 through X-1850, and X-1856.
- Glasses containing TiO$_2$: X-1846 through X-1848.
- Glasses containing ZrO$_2$: X-1853 through X-1855, X-1857 through X-1859, X-1893 through X-1894.
- Glasses containing HfO$_2$: X-1867.
- Glasses containing GeO$_2$: X-1860, X-1865, X-1886.

The X-994 composition was used as the base glass. In general, 
the added oxide was either substituted for part of the SiO$_2$ 
content of the X-994 composition or for part of the SiO$_2$, 
Al$_2$O$_3$, and MgO content on a weight basis. In all cases the mol 
 ratio of Al$_2$O$_3$ to MgO remained approximately equal to 1.

A systematic study of the exact effect of each addition on the 
tensile strength of the base glass was not attempted. In general, 
however, the tensile strength results indicate that the addition 
of P$_2$O$_5$ for a portion of the SiO$_2$ resulted in a general strength 
decrease as the P$_2$O$_5$ content increased. An even greater strength 
drop resulted when P$_2$O$_5$ was substituted for a portion of the SiO$_2$, 
Al$_2$O$_3$, and MgO. Modulus values were not greatly affected by any 
of the additions and cannot be explained.

TiO$_2$ substitutions for part of the SiO$_2$ caused the greatest drop 
in strength of all the additions used. The strength decreased as 
the TiO$_2$ content increased. Again, modulus values were not greatly 
affected and this response cannot be explained.

17
Variation in Tensile Strength and Modulus with R₂O Substitutions for 1/2 the MgO on a Mol Basis (Base Glass: SiO₂ - Al₂O₃ - MgO)

FIGURE 5 Variation in Tensile Strength and Modulus with R₂O Substitutions for 1/2 the MgO on a Mol Basis
Interpretation of the data for the glasses containing ZrO$_2$ is difficult. However, it appears that small ZrO$_2$ additions improve modulus values slightly. Even such small additions, however, decrease tensile strength. HfO$_2$ has the same effect.

Finally, the addition of GeO$_2$ for part of the SiO$_2$ greatly reduces the tensile strength below that associated with X-994. Modulus does not seem to be affected, but again no explanation is offered.

5. The Effect of Fiber Diameter on Tensile Strength and Modulus of Elasticity

Preliminary results from several studies of the forming parameters associated with the fiberization of SiO$_2$-Al$_2$O$_3$-MgO glasses have indicated that tensile strength and modulus properties were improved when the diameter of fibers under test was reduced. The tensile strength of virgin X-994 fiber is normally 700-725,000 psi for a 0.00040 inch diameter fiber. This figure is apparently raised to 800-850,000 psi for a fiber with an apparent diameter of 0.00015 to 0.00020 inch. Since there are problems encountered with accurately measuring fibers of this diameter with the micrometer eyepiece, further work in this area is warranted.

Modulus of elasticity values are also apparently improved for lower diameter fibers. The following modulus measurements have been made:

<table>
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<th>Glass</th>
<th>Modulus x 10$^6$ psi</th>
<th>Method Employed</th>
<th>Measured Diameter</th>
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<tr>
<td>X-994</td>
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<td>Elongation</td>
<td>0.00065 in.</td>
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<tr>
<td>X-994</td>
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<td>Elongation</td>
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<td>Elongation</td>
<td>0.00016</td>
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</table>

Note the modulus value, measured by the sonic method, of a fiber with a diameter of 0.00027 inches. The sonic method does not require a diameter measurement. This finding reinforces the view that the lower diameter fibers do have improved moduli. Further work in this area is definitely desirable.

Glasses in systems other than the SiO$_2$-Al$_2$O$_3$-MgO series have been checked to determine the effect of reductions in fiber diameter on both tensile strength and modulus. The properties of glasses in the SiO$_2$-Al$_2$O$_3$-Li$_2$O and SiO$_2$-Al$_2$O$_3$-CaO systems did not change with reductions in fiber diameter. Improved properties were evident, however, in the SiO$_2$-Al$_2$O$_3$-ZnO system.

B. Relationship Between Composition and Properties of Selected Glasses

1. General

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
The work reported here was undertaken to provide some insight into the relationship between glass structure and the properties of tensile strength and modulus of elasticity. In papers by Day and Rindone (1,2), it has been shown that a number of physical properties of certain glasses in the SiO₂-Al₂O₃-Na₂O field vary in a systematic manner with composition changes. Further, composition vs. property curves usually showed breaks which were related to proposed changes in glass structure. It was found that in a set of related compositions in which the Al to Na ratio varied, a break occurred at the point where this ratio was one. It was postulated that at ratios below one, the aluminum was in fourfold coordination and participated in the glass network, while at higher ratios some of the aluminum assumed sixfold coordination. This proposition was based in part upon the total number of oxygen ions available. At Al to Na ratios less than one, there are less than two. 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 had been done with other properties.

Considerable property data have also been published for glasses in the SiO₂-TiO₂-K₂O system (3). These glasses were also reported to show deviations in curves of properties vs. composition which were tentatively related to changes in coordination number of the titanium from six to eight. These observations, plus the superficial similarity between the two systems, led to the extension of the work by including a study of this composition field also.

The SiO₂-Al₂O₃-K₂O system is an attractive one for further study, inasmuch as it differs from each of the previously studied systems by only one constituent. It may be possible to relate the two through study of this system.

**Experimental Procedure**

a. SiO₂-Al₂O₃-Na₂O System

(1) Compositions Studied

Table II gives the compositions in mol percentages for all glasses studied. The glasses are divided into five related series. Series II and IV comprise sets of compositions similar to those given the same designation in the papers by Day and Rindone (1,2). In general, all glasses in each series contain the same percentage of silica. The exception is Series IV in which all components vary simultaneously.

(2) Preparation and Testing of Specimens

Batches compounded from relatively pure commercial raw materials were melted in crucibles to form the glass used to
### Table 1

**COMPOSITIONS IN MOL PER CENT**

**SiO$_2$-Al$_2$O$_3$-Na$_2$O SYSTEM**

#### Series I

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charge the bushing. Fibers between 0.00035 and 0.00040 inch in diameter were formed for tensile strength measurements. Virgin fibers were collected on forks, placed in a desiccator, and tested promptly in the manner previously described. At least three sets of fibers sufficient to give fifteen breaks each were tested for each glass. Results reported are usually the average of the results for the two closest sets. In a few cases, additional sets of fibers were tested, in which case the averages reported are for more than two sets.

Modulus of elasticity measurements were made on fibers in the 0.00075 to 0.00085 inch diameter range by measuring the elongation under load. This size of fiber was selected because it was convenient to form and to measure. Some difficulty was found in obtaining reproducible results with fibers varying widely in fiber diameter. Although the source of difficulty was never completely isolated, it was found that reproducibility was reasonably good when measurements were performed on fibers of this selected diameter range only. Each point reported is the average of at least eight measurements on four fibers.

(3) A rough measurement of relative viscosities of the glasses was made by noting the bushing temperature at which fibers were formed. Results are approximate only, since no effort was made to calibrate thermocouples, or to compensate for any drift in thermocouple output that may have occurred as they aged. Also, several different bushings were used, with possible bushing-to-bushing variation.
### TABLE III

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**b. SiO₂-TiO₂-K₂O System**

(1) **Compositions Studied**

Table III gives the compositions studied in mol percentages.

(2) **Preparation and Testing of Specimens**

Glasses were prepared, fiberized, and tested as described for the glasses in the silica-alumina-soda system.
Figure 6 Modulus Versus Al/Na Ratio
5. **Experimental Results**

a. **SiO$_2$-Al$_2$O$_3$-Na$_2$O System**

(1) **Tensile Strength**

Tensile strength results are summarized in graph form in Figure 6. A separate scale was used for each series of glasses, as indicated on the right of the graph, so that all data could be presented on one sheet without having curves overlap. Tensile strength increases in all cases as the Al to Na ratio increases. It appears that there is an inflection in all curves at or near an Al to Na ratio of one, such that the rate of increase of strength is less at the higher ratios.

(2) **Viscosity Measurements**

In Figure 7 are given the data for viscosity (fiber forming temperature) vs. the Al to Na ratios of the glasses.

Viscosity increases rapidly with increasing Al to Na ratio up to a ratio of one. Above this, the viscosity levels off or decreases slightly.

(3) **Modulus of Elasticity**

The data on modulus measurements are plotted in Figure 8. Again a separate scale is used for each series of glasses, so that all curves may be plotted on the same sheet without overlapping. There is an inflection point for all curves at an Al to Na ratio of one. All curves have approximately the same shape. Curves for Series VI and VII may be exceptions, since the locations of the last points indicate that they may rise quite steeply at the higher Al to Na ratios. However, the glasses for which these points were obtained were fiberized with difficulty because of devitrification. These points are therefore questionable. The last parts of the curves are dotted to indicate this.

The data were plotted in various ways to find whether some other significant relationship might be found. Figure 9 shows modulus plotted vs. the ratio of (Si + Al) to O. These curves show an inflection where the ratio is 0.50. This corresponds to the compositions for which the Al to Na ratio is one. However, the curves in Figure 9 show another inflection, where the ratio of (Si + Al) to O is 0.46 or 0.47. Several additional melts were made in Series VI to establish more accurately the location of the inflection and to determine how sharp the break might be. For this series, the inflection appears to be at 0.475 and to follow a smooth curve rather than to have a sharp break. The data are not complete enough or precise enough to show the exact location and nature of the inflections in the other curves. However, all seem to have some kind of a break in about the same region. When plotted in the same way, tensile
strength and viscosity curves may also show similar behavior, but results are less well defined. The significance of the breaks in this region has not yet been established.

A number of modulus measurements were made on annealed fibers. Work on annealed fibers was dropped when it became apparent that the information obtained did not justify the additional effort. In general, annealed fibers had moduli about 1,000,000 psi higher than those of fibers as drawn. Table IV lists moduli obtained on fibers as drawn and annealed for those glasses for which data are available.

Modulus data were plotted in a manner such that interpolated values could be obtained for constructing a diagram showing lines of constant modulus vs. composition. Results are shown in Figure 10. The dotted lines are lines of constant modulus of the indicated values. The five series of glasses studied fall on the dashed lines labelled with the appropriate Roman numerals. (Only the top portion of the three-component system from SiO$_2$ - 50% upwards is shown.) The phase boundaries and primary phases for this system reported in the literature are also shown. This was done to show whether transitions from regions characteristic of one phase to another would be accompanied by breaks in the constant modulus curves. The data do not show any clear indication of such behavior. The change of modulus from 8.0 to 7.0 x 10^6 psi requires a larger change in composition than a change of
Figure 10
Phase Diagram of the SiO₂-Al₂O₃-Na₂O System
similar magnitude at either higher or lower values of modulus. The constant modulus curve for modulus of 8.5 x 10^6 psi shows two breaks, whereas all other lines are nearly straight with only slight curvatures. The region in which this break occurs corresponds to the compositions for which the (Si + Al) to 0 ratio is 0.46 to 0.47.

This is the range of values in which a second break in the modulus curves was shown in Figure 9.

b. SiO₂-TiO₂-K₂O System

(1) Tensile Strength

Tensile strength results are summarized in graph form in Figure 11. Results are plotted vs. the TiO₂ to K₂O ratio. Any change in properties resulting from a change in the coordination number of the titanium might be expected to be a function of this ratio, since it is related to the amount of oxygen available in the system for coordinating with the titanium.

At the 60% SiO₂ level, it appears that there is a break in the curve at a ratio of one. At the 70% level, the curve is smooth, but it does have an inflection in this region. At the lower SiO₂ levels, it is not possible to be certain whether or not there is any meaningful break in the curves. The precision of the measurements is lower at the very low strengths involved. In addition, the glasses with the lower ratios have such poor durability that chemical attack on the fibers by the moisture of the air during the testing time could contribute to inaccuracies.

Examination of the published data on other physical properties of glasses in this system suggested that the position at which properties changed was dependent also upon the SiO₂ content. For this reason, the curves shown in Figure 12 were prepared showing the tensile strength as a function of the ratio of SiO₂ + TiO₂ to K₂O. The curves for 70 and 60% SiO₂ glasses are approximately parallel, but the other two do not appear to fit this pattern.

(2) Viscosity Measurements

The fiber forming temperatures are plotted as a function of TiO₂ to K₂O ratio in Figure 13. There is a suggestion of a break in the curve for the 60% SiO₂ series at a ratio of somewhat less than one. The rest of the curves approach a straight line relationship very closely.

Two curves are given for the 60% SiO₂ series. The one drawn as a solid line was obtained when the fibers tested for strength and modulus were run. When it became apparent that these results were inconsistent, the glasses were re-run to
Figure 11  Tensile Strength Versus TiO₂/K₂O Ratio

TENSILE STRENGTH x 10^7 psi

60% Soda

50% Soda
determine fiber forming temperatures only. The results are shown on the curve drawn in a dotted line. The re-run indicated that these glasses must have a very flat viscosity curve, that is, a relatively large change in temperature is required to produce a given change in fiber diameter. This could account in part for the unusual shape of the original curve. Care was taken to obtain forming temperatures at very nearly the same fiber diameters on the second run. Therefore, this curve is to be regarded as more reliable. The second curve was obtained with a different bushing. Small differences in bushing construction or thermocouple output may have caused the position of the curve to be generally higher than that obtained on the first run. The slope of the curve seems to be less at the 60% SiO₂ level than at either higher or lower levels.

The point at the lowest TiO₂ to K₂O ratio for the 50% SiO₂ series was obtained the day the thermocouple on the bushing failed. Its accuracy is therefore suspect.

Forming temperature data were re-plotted in Figure 14, showing forming temperature vs. the SiO₂ + TiO₂ to K₂O ratio. The curves have about the same shape and relationship to each other as shown on Figure 13.

(3) **Modulus of Elasticity**

Modulus results are plotted as a function of the TiO₂ to K₂O ratio in Figure 15. The curves are all similar in shape. The curve at the 70% SiO₂ level appears to be out of place at the upper end in that it crosses those for the 50% and 60% SiO₂ levels. The other curves seem to approach the same value at a TiO₂ to K₂O ratio of 3.0.

None of the curves show a sharp break, although all curve downward in the general vicinity where the TiO₂ to K₂O ratio is one.

When modulus is plotted vs. the SiO₂ + TiO₂ to K₂O ratio, Figure 16, all curves seem to fall into a consistent pattern. One point on the curve for the 50% SiO₂ level appears to be off, but it did not fit the apparent curve in Figure 15 either.

4. **Discussion of Results**

In the SiO₂-Al₂O₃-Na₂O system, the three properties measured—fiber forming temperature, tensile strength, and modulus of elasticity—appear to be dependent upon glass structure in the same manner as other physical properties reported in the literature. A break in composition vs. property curves was found where the Al to Na ratio for the glass was one. The suggestion that this is related to a change in coordination number of the alumina is consistent with these observations. Glass viscosity (as shown by fiber forming temperature)
Figure 16. Modulus Versus (SiO₂·TiO₂)/K₂O
increases at first as $\text{Al}_2\text{O}_3$ is substituted for $\text{Na}_2\text{O}$. This is logical if the $\text{Al}_2\text{O}_3$ enters the network and behaves much as $\text{SiO}_2$ would. At higher $\text{Al}_2\text{O}_3$ contents where the Al to Na ratio exceeds one, the aluminum added enters the structure with a coordination number of six, and acts as most glass modifiers do, by lowering the viscosity. Similarly, substitution of $\text{Al}_2\text{O}_3$ for $\text{Na}_2\text{O}$ increases the tensile strength, by increasing the proportion of ions participating in the network. Strength continues to increase, but at a lower rate, at ratios of Al to Na higher than one. It could be expected that viscosity might be related to tensile strength in that both are dependent upon the forces holding the network together, and that if a change in composition caused the viscosity to drop, the tensile strength should drop also. Two possible reasons for the results actually obtained are suggested:

a. Forming temperature may not be a simple function of viscosity. Surface tension, for instance, can affect forming behavior, and might change in such a way as to counteract changes in viscosity.

b. The cooling of the glass may be accompanied by further structural changes which make it behave differently at room temperature than it would at elevated temperatures.

Measurements of the modulus of elasticity show the break at the Al to Na ratio of one more clearly and consistently than to tensile strength or forming temperature. This is not surprising. Tensile strength of fibers is known to be closely related to the conditions under which they were formed. Attempts were made to keep forming conditions similar for the fibers tested in this study. However, it is quite possible that if enough conditions had been tried to find the optimum strength for each composition, some inconsistencies in the data would have disappeared. The measurement of forming temperature was just incidental, and was not the primary purpose of the work. Hence, precautions that could have been taken to increase accuracy were not observed, and results are expected to contain small errors. Modulus results, on the other hand, seem to be less sensitive to variations in forming conditions. The precision is believed to be relatively good, at least when the diameters of the fibers tested lie in a small range. The spread in results for the eight or more determinations made for each point reported was usually not more than $0.3 \times 10^6$ psi, and normally less than this.

The nature of the change in modulus as the Al to Na ratio increases is somewhat surprising. A structural change which results both in lowered viscosity and a reduction in slope of the tensile strength curve causes an increase in the modulus. It may be that the larger size structural units resulting from six-coordinated aluminum can increase the rigidity of the structure without increasing its strength.

Recently Lacy\(^{(5)}\) offered another proposal for the structure of glasses in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{Na}_2\text{O}$ system. He suggested that the logical packing
of ions favors the formation of what he calls "triclusters." These triclusters consist of three tetrahedral groups having a common oxygen, which is shared by an AlO₄ group and two SiO₄ groups or an SiO₄ and two AlO₄ groups. Each two triclusters conserve one oxygen. When the Al to Na ratio is greater than one, there are not enough oxygens for all glass formers to be in tetrahedral groups. According to Lacy, the formation of triclusters compensates for this oxygen shortage. This theory would probably account for the observed results as well as the concept of change in coordination numbers if we assign suitable properties to the triclusters. The data do not seem to support one concept over the other.

The properties measured on the glasses in the SiO₂-TiO₂-K₂O system do not appear to lead to as good a concept of glass structure. Examination of Figures 15 and 16 shows that the modulus increases as either the TiO₂ to K₂O or the SiO₂ + TiO₂ to K₂O ratio increases. The rate of increase drops off at the higher ratios. The absence of any sharp break in the curves suggests that the change in slope does not result from a sudden change in structure at some particular composition. If, for instance, a change in coordination number of the titanium is responsible, it seems likely that titanium is present to some degree in both states in all glasses studied. Changes in composition thus probably change the relative amounts in some rather gradual fashion. That the change in structure is affected also by the SiO₂ content is suggested by the fact that the data seem to fall into a systematic system of curves in Figure 16, while there is some crossing of curves in Figure 15, in which no account is made of the SiO₂ content.

It might be argued that Figures 11 and 12 do not support the latter contention in that curves for tensile strength seem to show a more orderly relationship when plotted vs. the TiO₂ to K₂O ratio than when plotted vs. the SiO₂ + TiO₂ to K₂O ratio. However, examination of Figure 12 suggests that it is possible that the proper order of the curves for the various series of glasses might be the same as that found for modulus. At the very low ratios of SiO₂ + TiO₂ to K₂O, the curves tend to fall in the same order as the modulus curves in Figure 16. The peculiar shapes of the tensile strength curves at the 50% and 60% SiO₂ levels do not seem logical and may indicate experimental error.

Figure 17 was plotted to assist in plotting lines of constant modulus on a three-component diagram. It appears that at any SiO₂ level the modulus may be a linear function of the K₂O content of the glass.

The constant modulus curves are plotted in Figure 18. These curves appear to offer no particularly significant implications relative to glass structure.

The SiO₂-Al₂O₃-K₂O system should be studied as the SiO₂-Al₂O₃-Na₂O and the SiO₂-TiO₂-K₂O systems have been. Results should be studied to determine if some correlation between the two systems can be found.
Differences between the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{K}_2\text{O}$ system and the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{Na}_2\text{O}$ system should be examined to see if they can be related to the size of the alkali ion added. If such relation is found, study of a system utilizing a third alkali ion, probably lithium, should be undertaken.
III
CONCLUSIONS

The results of this investigation indicate that glass fibers possessing the highest tensile strength and modulus of elasticity values are located in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{MgO}$ and the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{ZnO}$ systems. There is some indication that the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{BeO}$ system may also possess desirable properties.

The best glass compositions in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{MgO}$ system fall at an $\text{Al}_2\text{O}_3$ to $\text{MgO}$ mol ratio of approximately 1.0 and contain from 60 to 70% by weight of $\text{SiO}_2$. The $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{ZnO}$ system has not been investigated extensively, but it is expected that the glasses with the best properties are located within a similar range of values. Investigation of the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{BeO}$ system should be continued, but is complicated by the extreme devitrification tendencies of this glass.

The addition of a fourth oxide component to a base glass consisting of the S-994 composition does not improve the tensile strength or modulus of elasticity of the resultant glass. The addition of minute quantities of a fourth oxide component has received only preliminary study, but might prove beneficial.

The exact effect of fiber diameter on both tensile strength and modulus is still open to question. However, there is some evidence that a reduction in diameter of $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{MgO}$ and $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{ZnO}$ glass fibers does improve properties, especially the modulus of elasticity.
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**ABSTRACT**
The results of the present investigation have shown that glass fibers drawn from compositions located in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{ZnO}$ system possess tensile strengths in excess of 800,000 psi. Glass fibers drawn from compositions in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{MgO}$ system have given similar results, particularly at fiber diameters in the 0.00015 to 0.00020 inch range. The modulus of elasticity of glass fibers obtained from compositions located in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{MgO}$ system can be increased to $16.0 \times 10^6$ psi. This is accomplished by lowering the fiber diameter to the 0.00015 to 0.00020 inch range.

In the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{K}_2\text{O}$ system, the fiber forming temperature, the tensile strength and the modulus of elasticity change with composition in a manner which gives a break in the composition vs. property curves at the point where the Al to Na ratio is 1. This is related to the change in the coordination number of the aluminum at this ratio which has been proposed in the literature.

The properties measured for glasses in the $\text{SiO}_2$-$\text{TiO}_2$-$\text{K}_2\text{O}$ system do not appear to lead to as clear a concept of glass structure. Properties vary gradually with composition, without evidence of a sharp break at any point. There is some indication that the rate of change of properties is dominated more by the silica content than is the case in the $\text{SiO}_2$-$\text{Al}_2\text{O}_3$-$\text{Na}_2\text{O}$ system.
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