MTL TR 89-44

AD-A211 170

AD

Y-Si-Al-O-N GLASS FIBERS

DONALD R. MESSIER, ROBERT P. GLEISNER,
and RONALD E. RICH
CERAMICS RESEARCH BRANCH

May 1989

Approved for public release; distribution unlimited.

US ARMY
LABORATORY COMMAND
MATERIALS TECHNOLOGY LABORATORY

U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
Watertown, Massachusetts 02172-0001

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

Mention of any trade names or manufacturers in this report shall not be construed as advertising nor as an official indorsement or approval of such products or companies by the United States Government.

DISPOSITION INSTRUCTIONS

Destroy this report when it is no longer needed.
Do not return it to the originator.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 89-44	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)* Y-Si-Al-O-N GLASS FIBERS		5. TYPE OF REPORT & PERIOD COVERED Final Report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR*(s)* Donald R. Messier, Robert P. Gleisner, and Ronald E. Rich		8. CONTRACT OR GRANT NUMBER*(s)*
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-EMC		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: 1L16102.AH42
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE May 1989 13. NUMBER OF PAGES 11
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*		15. SECURITY CLASS. *(of this report)* Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Fiber-reinforced composites
Glass
Oxynitride glass
Glass fibers
Corrosion resistance
Mechanical properties

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

(SEE REVERSE SIDE)

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

Block No. 20

ABSTRACT

The excellent mechanical properties and outstanding water corrosion resistance of Y-Si-Al-O-N glasses indicate that they are attractive candidate materials for forming into high performance glass fibers. Fibers of glasses containing, respectively, 3.2 and 6.6 wt% N were drawn freehand in air, and from glass rods in N_2. Continuous fibers (100-m long) of the former glass were melt-drawn in N_2 while being wound in air outside of the glass-melting furnace. Some fibers had diameters as small as 10 μm, and characterization data show that the fibers retain all of the desirable properties of the bulk glass.

Keywords: continued on front

INTRODUCTION

It is now well established that the properties of many glasses are markedly improved by the substitution of N for O in the glass structure.[1-7] Properties that increase with increasing N content include glass transition temperature, viscosity, density, hardness, water corrosion resistance, and, of especial interest, elastic modulus. Reported in the literature are elastic modulus values for Y-Si-Al-O-N (up to 186 GPa),[8] and for Ca-Si-Al-O-N (135 GPa)[9] glasses that significantly exceed values obtained for similar oxide glasses and, indeed, are unprecedented for any non-Be glass. In view of the considerable potential for improvement afforded by N substitution, it appeared that oxynitride glasses should have promise for fabrication into fibers with exceptional properties, and the purpose of this investigation was to demonstrate the feasibility of making such fibers.

Interest in the particular system chosen for this investigation came from prior work on the hot pressing of Si_3N_4 indicating that the use of yttria as a sintering aid produces material with good high temperature properties.[10] Jack and his coworkers[1] appear to be the first to recognize that this and other sintering aids produced oxynitride glass phases, and that the glasses could be prepared as separate phases. Subsequent studies of the preparation of, and evidence of the outstanding properties of, glasses in the system Y-Si-Al-O-N were reported by several investigators.[2-6,8,11-14] Of particular significance, with respect to the potential usefulness of these glasses for fibers, are the high elastic modulus values for bulk glasses reported by Messier and Broz,[8] and the outstanding hot water corrosion resistance demonstrated by Loehman,[3] and by Wald et al.[13]

The present investigation is an outgrowth of earlier work[8,11,14] on the preparation and properties of Y-Si-Al-O-N glasses. That work emphasized making the materials in significant quantities (100 g). It was intended that this system, while yielding useful materials in its own right, could also serve to demonstrate the feasibility of producing oxynitride glass fibers in any number of systems. Such has indeed proved to be the case, as will be reported in future publications.

1. JACK, K. H. *Sialon Glasses* in Nitrogen Ceramics, F. L. Riley, ed., Noordhoff International, Reading, MA, 1978, p. 257-262.
2. SHILLITO, K. R., WILLS, R. R., and BENNETT, R. B. *Silicon Metal Oxynitride Glasses.* J. Am. Ceram. Soc., v. 61, no. 11-12, 1978, p. 537.
3. LOEHMAN, R. E. *Preparation and Properties of Yttrium-Silicon-Aluminum Oxynitride Glasses.* J. Am. Ceram. Soc., v. 62, no. 9-10, 1979, p. 491-494.
4. LOEHMAN, R. E. *Oxynitride Glasses.* J. Non-Cryst. Solids, v. 42, 1980, p. 433-446.
5. DREW, R. A. L., HAMPSHIRE, S., and JACK, K. H. *Nitrogen Glasses* in Special Ceramics, D. Taylor and P. Popper, ed., Proc. Brit. Ceram. Soc., v. 31, 1981.
6. LOEHMAN, R. E. *Preparation and Properties of Oxynitride Glasses.* J. Non-Cryst. Solids, v. 56, 1983, p. 123-134.
7. MESSIER, D. R. *Review of Oxynitride Glasses.* Rev. Chim. Min., v. 22, 1985, p. 518-533.
8. MESSIER, D. R., and BROZ, A. *Microhardness and Elastic Moduli of Si-Y-Al-O-N Glasses.* J. Am. Ceram. Soc., v. 65, no. 8, 1982, p. c-123.
9. SAKKA, S., KAMIYA, K., and YOKO, T. *Preparation and Properties of Ca-Al-Si-O-N Oxynitride Glasses.* J. Non-Cryst. Solids, v. 56, 1983, p. 147-152.
10. GAZZA, G. E. *Hot Pressed Si_3N_4.* J. Am. Ceram. Soc., v. 56, 1973, p. 662.
11. MESSIER, D. R. *Preparation and Crystallization of Si-Y-Al-O-N Glasses.* in Ceramic Engineering and Science Proceedings, v. 3, no. 9-10, September - October 1982, The American Ceramic Society, Columbus, Ohio, p. 563-576.
12. HAMPSHIRE, S., DREW, R. A. L., and JACK, K. H. *Viscosities, Glass Transition Temperatures, and Microhardness of Y-Si-Al-O-N Glasses.* J. Am. Ceram. Soc., v. 67, no. 3, 1984, p. c46-c47.
13. WALD, J. W., MESSIER, D. R., and DEGUIRE, E. J. *Leaching Behavior of Si-Y-Al-O-N Glasses.* Int. J. High Tech. Ceramics, v. 2, 1986, p. 65-72.
14. MESSIER, D. R. *Preparation and Properties of Y-Si-Al-O-N Glasses.* Int. J. High Tech. Ceramics, v. 3, 1987, p. 33-41.

EXPERIMENTAL PROCEDURES

Glass Preparation

All of the glasses that were used in this study were premelted according to procedures already described.[11,14] Melting was done in BN-lined graphite crucibles in N_2 at temperatures from 1600°C to 1650°C. The resulting glass disks were subsequently broken into chunks and, if required, ground into powder for forming into glass rods. The coarse glass chunks were used as is for feed stock in melt-drawing experiments. Glass rods were formed by remelting ground glass in BN-lined graphite crucibles. The crucible was lined by coating its interior with BN powder applied as a slurry in acetone. The resulting coating was dried to remove any residual acetone. The glass rods that were obtained were 4 mm to 8 mm in diameter and about 50-mm to 100-mm long. The two glass compositions that were investigated, and to be discussed further below, are given in Table 1.

Table 1. COMPARISON OF Y-Si-Al-O-N GLASS NOMINAL COMPOSITIONS WITH COMPOSITIONS OBTAINED ON BULK GLASSES AND GLASS FIBERS VIA CHEMICAL AND SCANNING ELECTRON MICROSCOPE (SEM) ANALYSES

	Weight Percent (of Cation Total)			Weight Percent (of Spec. wt)
Specimen	Al	Si	Y	N
Nominal "a" Composition	9.3	29.1	61.6	3.2
"a" Chemical Analysis	9.7	26.8	63.5	3.3
"a" Bulk Glass (SEM)	8.7	31.6	59.8	2.7
"a" Fiber No. 11 (SEM)	9.1	27.9	63.0	4.1
"a" Fiber No. 12 (SEM)	8.9	27.7	63.4	3.5
Nominal "b" Composition	18.7	19.5	61.8	6.6
"b" Chemical Analysis	18.4	19.2	62.4	6.4
"b" Bulk Glass (SEM)	18.2	18.6	63.1	9.4
"b" Fiber (SEM)	18.4	17.2	64.4	8.4

Fiber Drawing in Air

Initial fiber drawing attempts involved simply heating abutting pieces of glass in air with an oxyacetylene torch until they partially fused together, and rapidly pulling the pieces apart. The fibers, although relatively easily formed, were variable in length and cross-sectional size and shape.

Fiber Pulling from Rods in N_2

The first fibers that were drawn in N_2 were made in the same furnace (Figure 1) that was subsequently used for drawing from the melt. The procedure involved heating the tips of two abutting glass rods, one of which was fixed and the other of which could be withdrawn to pull a short fiber. One of the rods was held rigidly in a drill chuck, while the other was affixed to a steel rod that could be pulled up through a compression fitting in the top of the apparatus. In order to draw a short (250 mm) fiber, the apparatus was assembled with the tips of the two glass rods abutting in the hot zone, and the assembly heated to the point where the glass softened and the rod tips fused together. The moveable rod was then

quickly withdrawn to form the fiber, the removal of which required disassembly of the apparatus after cooling to room temperature. Because of obvious difficulties, this fiber-drawing method was abandoned in favor of the melt-drawing technique described below.

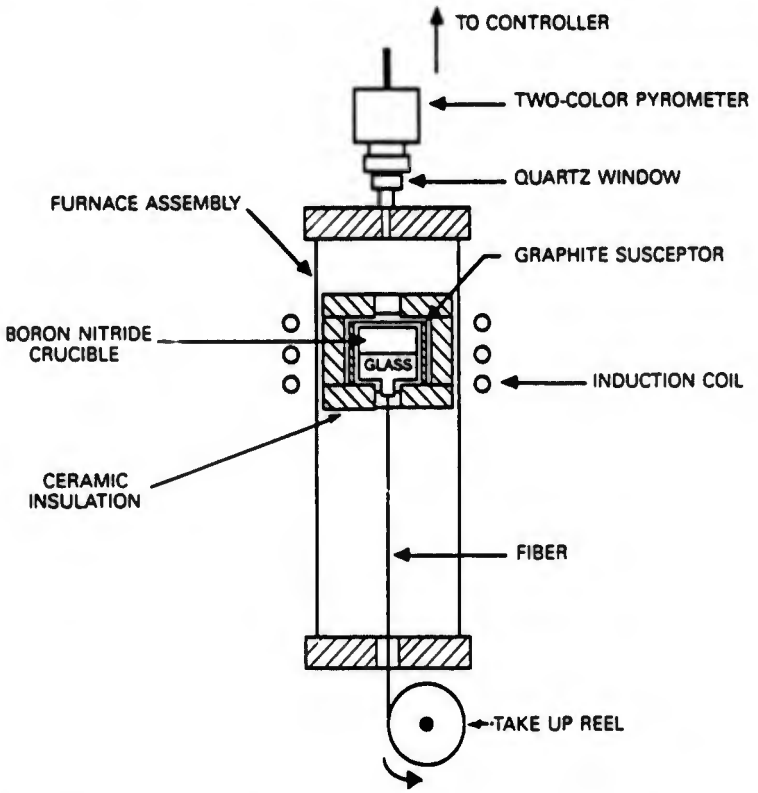

Figure 1. Schematic representation of oxynitride glass fiber-drawing system.

Fiber Drawing from the Melt in N_2

The apparatus used for drawing Y-Si-Al-O-N glass fibers from the melt is illustrated in Figure 1. Earlier experience taught us the necessity for precise temperature programming and control, and the present setup includes a two-color pyrometer* that provides temperature measurement as well as a feedback signal for control by a microprocessor programmer/controller.† The furnace employs a graphite susceptor heated by a 400-MHz induction generator‡ whose power is regulated by a silicon-controlled rectifier.** Insulation is provided by ceramic foam type refractory material◊ that is easily formed with hand tools. The glass-melting crucible, 30-mm orifice diameter x 50-mm long, is machined from hot-pressed BN.◊◊ The nozzle in the bottom of the crucible is 3-mm long with an orifice diameter of 2.8 mm. The fiber is wound on a 100-mm-diameter plastic spool by means of a laboratory motor whose speed is continuously variable from 0 to 3000 rpm. The furnace is enclosed in a vacuum-tight fused silica tube, and the fiber is withdrawn through a slightly loosened compression fitting in the bottom of the furnace tube.

*R Series, Ircon, Skokie, IL.
†Micricon, Research Inc., Minneapolis, MN.
‡Tocco, Ohio Crankshaft Co., Cleveland, OH.
**Barber Colman Co., Rockford, IL.
◊ Zircar Products, Inc., Florida, NY.
◊◊ Carborundum Co., Niagara Falls, NY.

In order to draw a fiber, the crucible is filled with crushed premelted glass, loaded into the furnace, and heated in flowing N_2 to a temperature suitable for fiber drawing. Heating is continued to the point where the glass has melted, and a bead of molten glass has formed at the nozzle tip. For the glasses investigated in this study, that temperature is in the range of 1500°C to 1600°C. At this stage, a fiber is started by inserting an Al_2O_3 thermocouple insulator tube axially through the fitting in the bottom of the furnace to the point at which its tip just touches the molten glass bead. The Al_2O_3 tube is then withdrawn through the fitting, pulling a fiber with it. The tip of the fiber is then attached to the take-up spool, and winding begun. Winding continues until the fiber breaks, or the glass in the crucible has been consumed. Winding times are typically from 5 to 10 minutes, at which time we estimate that 100 m or more of fiber have been drawn. Fiber diameters for different runs have ranged from 30 μm to 100 μm with the smallest diameter feasible at present being limited by inadequacies (presently being corrected) in our experimental system.

Characterization

The characterization data reported herein were obtained by various means. Density measurements on bulk specimens were obtained by the usual Archimedes technique, and X-ray diffraction patterns were run on various glass specimens that had been crystallized by heating in N_2 for 2 hours at 1300°C. Optical photomicrographs were taken with transmitted light on fiber specimens in immersion oil.

Microhardness measurements were done on polished sections with a Knoop indenter and 0.98 N load. The technique used for elastic modulus measurements on bulk specimens has already been discussed.[8] Modulus values for the air-drawn fibers were obtained from stress-strain curves,[15] and for the N_2-drawn fibers from sonic velocity measurements.[16]

Conventional chemical analyses that were done included cation determination by spectrochemical techniques, and analyses for O and N by the Kjeldahl inert gas fusion technique.[14] Cation analysis in the scanning electron microscope was done by energy dispersive X-ray spectroscopy (EDX), and N analysis by wavelength dispersive spectroscopy (WDS).

RESULTS AND DISCUSSION

Air-Drawn Fibers

Although the Y-Si-Al-O-N glass fibers drawn in air were somewhat crude and misshapen, the results are instructive as to the behavior of the glass when heated in air. Table 2 summarizes the results of chemical analyses for N on several samples of Y-Si-Al-O-N glass corresponding to composition "a" in Table 1. The heating times undergone by the fiber specimens, although not measured exactly, are estimated to be in the range from a few seconds to a minute or so at temperatures near the melting range of the glass. In comparison, heating times for the extensively heated material are estimated to be of the order of 10 minutes or more. The specimens heated for short times retain significant amounts of their original N contents, and decomposition is incomplete even for glass heated extensively in air.

15. ASTM Standard D 3379. *Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials.* American Society for Testing and Materials, 1975.
16. Dynamic Modulus Tester, PPM-5R, H. M. Morgan Co., Norwood, MA.

Table 2. COMPARISON OF ANALYZED N CONTENTS OF AIR-DRAWN Y-SI-AL-O-N GLASS FIBERS WITH NOMINAL STARTING COMPOSITION AND WITH N ANALYSIS ON BULK GLASS USED FOR FIBER DRAWING

Specimen	Weight Percent N
Nominal Glass Compostion	3.20
Bulk Glass	4.12
Air-Drawn Fibers	2.60
Glass Heated Extensively in Air	0.26

Although the fibers that were drawn in air were of poor quality, that such fibers could be produced at all while retaining a significant N content is noteworthy. Rapid thermal decomposition would be expected in air, and the observed result suggests that the heating and drawing rates were so fast as to preclude extensive thermal decomposition during fiber drawing. At any rate, heating the glass with a torch to the high temperature required for drawing was difficult, as was controlling the process. Air drawing was not, therefore, pursued further as a viable means of making oxynitride glass fibers.

Fibers Pulled from Rods in N_2

Numerous problems were encountered in attempting to draw short fibers from glass rods in N_2. The rods that were fabricated were often imperfect and some tended to break at pre-existing cracks when stressed even moderately. The best fiber drawing procedure that could be devised consisted of fusing together the ends of short rods inserted into the hot zone from opposite ends of the furnace, cooling to yield a single rod held at both ends, and reheating to soften the glass enough to stretch the rod into a fiber by pulling from one end. Judging the correct viscosity for pulling, however, proved difficult, and the rods frequently broke from accidentally induced bending stresses. Barring that, the rod often pulled out of the chuck holding it if pulling was started before the glass was soft enough. At the other extreme, heating to too high a temperature for fiber drawing resulted in separation of the two rod halves without any fiber being produced. The temperature problem was compounded by the difficulty of temperature measurement; the presence of the rod interfered with the axial sight path required for optical pyrometry. Despite these difficulties, however, Figure 2 presents evidence that a few good fibers were produced by pulling from rods.

Some of the above mentioned problems with regard to pulling fibers from oxynitride glass rods are almost certainly composition related. For example, the Y-Si-Al-O-N glass that was used had a narrow temperature range in which its viscosity was suitable for fiber drawing. A further problem relates to the necessity of imposing a temperature gradient on the rod during fiber drawing; that gradient caused devitrification to occur in the part of the rod which was in the temperature range (approximately 900°C to 1100°C) where devitrification occurs fairly readily. Our past experience has been that devitrified glass is difficult to remelt, and such devitrification would make it difficult, at best, to pull fibers in a manner that required the rod tip to be fed continuously into the hot zone to furnish additional molten glass to keep the process going. The foregoing problems could probably be minimized or eliminated by suitable modifications of the glass composition, but the investigation of such modifications was beyond the scope of the present study.

Figure 2. Transmitted light optical photomicrograph of parts of short (100-mm long) Y-Si-Al-O-N glass fibers drawn from a glass rod. Fiber quality and uniformity are good, and the small diameter (10 μm) of one fiber indicates that fine fibers can be formed from this glass.

Fibers Melt Drawn in N_2

The experimental setup shown in Figure 1, although similar to arrangements used to make many types of oxide glass fibers,[17] differs in some important respects. A unique feature of the present fiber-drawing process is that a flowing N_2 atmosphere is maintained in the furnace chamber to prevent thermal decomposition of the molten glass[18] while the fiber is wound in the ambient atmosphere. Also unique is the use of a BN crucible. Because of the N_2 atmosphere and corrosiveness of the glass, the noble metals commonly used for oxide glasses would be unsuitable as crucible materials.

Although graphite has proven satisfactory as a susceptor material, it may promote thermal decomposition by making the atmosphere excessively reducing.[18] From that standpoint, a susceptor capable of operating in a more oxidizing environment might be desirable, and other possible susceptor materials are currently being evaluated.

After initial successful attempts at drawing short fibers from molten glass in a BN crucible, the apparatus was configured as shown in Figure 1 for winding continuous fibers. While a number of successful fiber-drawing runs were conducted with Y-Si-Al-O-N glass of composition "a" (Table 1), attempts at drawing continuous fibers of composition "b" were unsuccessful. Although short fibers of the latter composition were produced, the fibers were so stiff, they resisted winding.

Photomicrographs of sections of continuous fibers of composition "a" are shown in Figure 3. Although difficult to see in Figure 3, imperfections found in the fibers typically included occasional transparent crystalline inclusions, metallic Si-rich particles, and elongated axial pores.

17. LOWENSTEIN, K. L. *The Manufacturing Technology of Continuous Glass Fibers.* Glass Science and Technology, Elsevier, Amsterdam-Oxford-New York, v. 6, 1983.
18. MESSIER, D. R., and DEGUIRE, E. J. *Thermal Decomposition in the System Si-Y-Al-O-N.* J. Am. Ceram. Soc., v. 67, no. 9, 1984, p. 602-605.

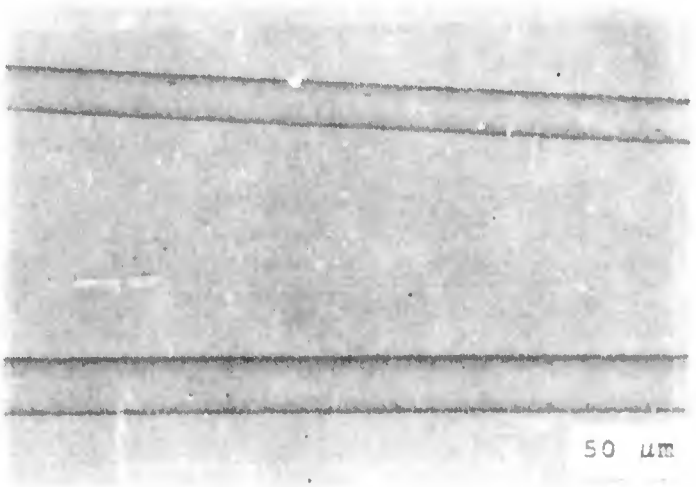

Figure 3. Transmitted light optical photomicrograph of segments of a continuous Y-Si-Al-O-N "a" fiber drawn from the melt.

Composition-related effects such as discussed above regarding pulling fibers from rods also must be considered with respect to drawing fibers from the melt. While continuous fibers were successfully drawn from Y-Si-Al-O-N glass of one composition ("a" in Table 1), the usable temperature range for fiber drawing was very narrow. As already noted, with the higher N composition ("b" in Table 1) it was impossible to make long, continuous fibers with our experimental setup. In both instances, the fiber-forming ability of the glass could probably be improved by modifying the glass compositions with additives that would make the viscosity-temperature characteristics of the glass more suitable for fiber drawing and winding.

Characterization

The results of chemical analyses performed on several bulk glass and fiber specimens are shown in Table 1. All of the cation analyses are in good agreement with each other as well as with the nominal starting composition. The N values obtained by WDS are generally higher than the nominal and Kjeldahl values, but in reasonably good agreement with the latter. The comparison of WDS results presented in Figure 4 illustrates the similarity of spectra on bulk glass and fiber specimens of the same nominal composition. All in all, the chemical analysis results indicate strongly that the chemistry of the fibers is identical to that of the bulk glasses from which they were drawn, confirming that composition changes are negligible during fiber drawing in N_2.

Although the WDS results are considered to be semiquantitative, the good agreement between the WDS and chemical analysis data is encouraging. Furthermore, the adaptability of WDS to small specimens indicates that it should be a very convenient and useful technique for the characterization of oxynitride glass fibers.

Table 3 compares physical property data on fibers with data on bulk glasses. The elastic and microhardness data were obtained as indicated in the previous section. The X-ray diffraction data show the existence of N-containing phases in the crystallized glasses, additional evidence for the presence of N in the fibers, including fibers drawn in air.

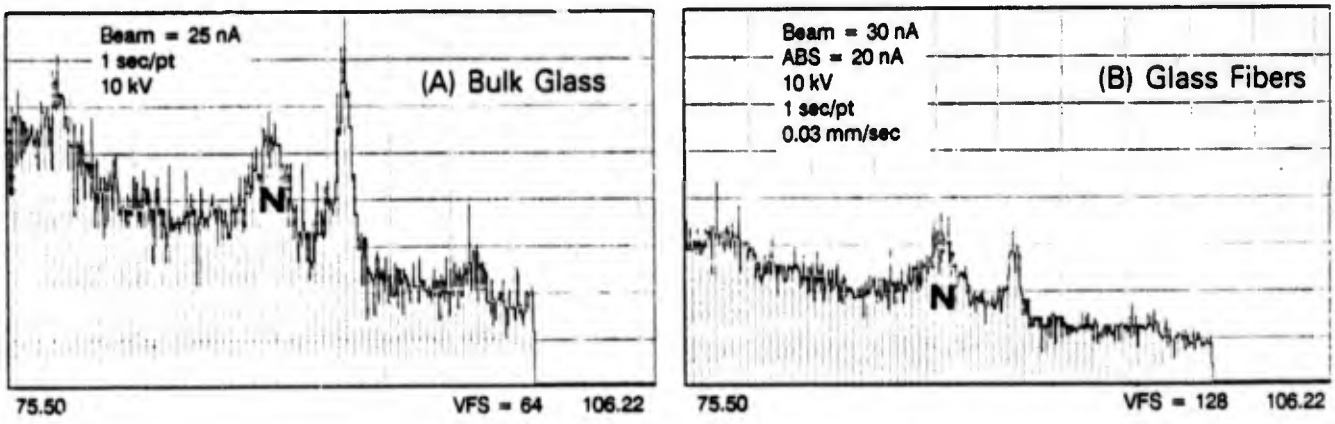

Figure 4. WDS spectra showing N peaks from polished sections of Y-Si-Al-O-N "b" glass and glass fibers. Spectrum of bulk glass (A), is similar to that of fiber (B), indicating that N contents are the same in both.

Table 3. CHARACTERIZATION OF Y-Si-Al-O-N GLASS FIBERS

Composition Designation	"a" Bulk	"a" Fiber (Air)	"a" Fiber (N_2)	"b" Bulk	"b" Fiber (Air)	"b" Fiber (N_2)
Nominal wt% N	3.2	3.2	3.2	6.6	6.6	6.6
Bulk Density (kg/m^3)	3720	-	-	3920	-	-
Microhardness (in GPa, Knoop, 0.98 N Load)	9.01	8.82	9.14	10.4	10.6	9.88
Elastic Modulus (GPa)	141	138	135	157	-	146
Phases in Crystallized Glass	$Y_2Si_2O_7$	-	-	Various Oxynitrides & Oxides	YAG, "H," β-Si_3N_4	-

As with the chemical analysis results, the physical properties of the fibers are essentially the same as those of the parent glasses. The measured microhardnesses and elastic moduli of the fibers are a little lower than those of the bulk glasses, a result consistent with literature data comparing the properties of the two forms of glass in other systems.[19,20] All of the data are also consistent with earlier findings that microhardness and elastic modulus of many glasses increase with the increasing N content.[7] Although the densities of glasses in the system Y-Si-Al-O-N are rather high, their properties are still impressive on a specific, i.e., property/density, basis. Furthermore, the general effect of N in increasing hardness and stiffness for glasses in many systems suggests that the properties of many glass-fiber compositions could be improved by adding N to them, and further research is currently being pursued along those lines.

19. LOWENSTEIN, K. L. *Studies in the Composition and Structure of Glasses Possessing High Young's Moduli, Part 1. The Composition of High Young's Moduli Glasses and the Function of Individual Irons in the Glass Structure.* Phys. Chem. Glasses, v. 2, no. 3, 1961, p. 69-82.

20. LOWENSTEIN, K. L. *Studies in the Composition and Structure of Glasses Possessing High Young's Moduli, Part 2. The Effect of Changes in the Configuration Temperature.* Phys. Chem. Glasses, v. 2, no. 4, 1961, p. 119-125.

SUMMARY

In summary, the following may be concluded:

1. Y-Si-Al-O-N glass fibers may be drawn in air while retaining a significant portion of their N content providing that the heating and drawing processes are relatively fast.

2. Fibers may be pulled in N_2 from glass rods, demonstrating the potential for producing high quality, small diameter (10 μm) oxynitride glass fibers.

3. Long (100 m), small diameter (30 μm - 40 μm), continuous Y-Si-Al-O-N glass fibers may be drawn from the melt in N_2 and wound on a spool in ambient air.

4. The chemical and physical properties of Y-Si-Al-O-N fibers drawn from the melt in N_2 are the same as those of the as-prepared bulk glass.

5. The stiffness and hardness of Y-Si-Al-O-N glass fibers surpass those of any existing glass fibers.

ACKNOWLEDGMENTS

The assistance of Jim Marzik in providing the WDS data is gratefully acknowledged as is that of Gail Meyers in furnishing the EDX analyses.

DISTRIBUTION LIST

No. of Copies	To
	Office of the Under Secretary of Defense for Research and Engineering, The Pentagon, Washington, DC 20301
1	ATTN: Mr. J. Persh
1	Dr. W. E. Snowden
	Director, Defense Advanced Research Project Agency, 1400 Wilson Boulevard, Arlington, VA 22901
1	ATTN: LTC P. H. Sullivan
1	Dr. J. Richardson
1	Dr. B. Wilcox
1	LTC D. Wance
	Commander, U.S. Army Laboratory Command, 2800 Powder Mill Road, Adelphi, MD 20783-1145
2	ATTN: AMSLC-IM-TL, Mr. R. Vitali
	Commander, Defense Technical Information Center, Cameron Station, Bldg. 5, 5010 Duke Street, Alexandria, VA 22304-6145
2	ATTN: DTIC-FDAC
1	Metals and Ceramics Information Center, Battelle Columbus Laboratories, 505 King Avenue, Columbus, OH 43201
	Commander, Army Research Office, P.O. Box 12211, Research Triangle Park, NC 27709-2211
1	ATTN: Information Processing Office
1	SLCRD-MD, Dr. I. Ahmad
	Commander, U.S. Army Materiel Command, 5001 Eisenhower Ave. Alexandria, VA 22333
1	ATTN: AMCLD
1	AMCDE-SR, Dr. R. Chait
1	Mr. R. Zigler
	Commander, U.S. Army Electronics Research and Development Command, Fort Monmouth, NJ 07703-5000
1	ATTN: AMDSD-L
1	AMDSD-E
	U.S. Army Research, Development and Standardization Group (UK), Box 65, FPO, New York 09510
1	ATTN: Dr. W. C. Simmons
	Commander, U.S. Army Missile Command, Redstone Scientific Information Center, Redstone Arsenal, AL 35898-5241
1	ATTN: AMSMI-RD-CS-R/Doc
1	AMSMI-RLM
	Commander, U.S. Army Natick Research, Development, and Engineering Center, Natick, MA 01760
1	ATTN: Technical Library
1	Dr. R. Lewis
	U.S. Department of Energy, Forrestal Building CE-151, 1000 Independence Avenue, Washington, DC 20585
1	ATTN: Mr. R. B. Schulz
	Commander, U.S. Army Tank-Automotive Command, Warren, MI 48397-5000
2	ATTN: AMSTA-TSL, Technical Library
1	AMSTA-ZSK
1	AMSTA-RSK, Sam Goodman
1	AMSTA-RSK, David Tenenbaum
1	AMSTA-RSK, Dr. James L. Thompson
1	AMCPM-M113-T, G. B. Singh
1	AMCPM-M113, Mr. Joseph Loiselle
1	AMCPM-BFVS, Gary Chamberlain
	Commander, U.S. Army Aviation Systems Command, Aviation Research and Technology Activity, Aviation Applied Technology Directorate, Fort Eustis, VA 23604-5577
1	ATTN: SAVRT-TY-ASV, Mr. McAllister
1	SAVRT-TY-ASV, W. Swink
	Commander, U.S. Army Aviation Systems Command, 4300 Goodfellow Boulevard, St. Louis, MO 63120-1798
1	ATTN: AMSAV-GTD
1	AMSAV-E
1	AMCPEO-AV

No. of Copies	To
	Naval Research Laboratory, Washington, DC 20375
1	ATTN: Dr. G. R. Yoder - Code 6384
1	Dr. D. Lewis
	Chief of Naval Research, Arlington, VA 22217
1	ATTN: Code 471
1	Dr. R. C. Pohanka
	Director, Structural Mechanics Research, Office of Naval Research, 800 North Quincy Street, Arlington, VA 22203
1	ATTN: Dr. N. Perrone
1	Bruce MacDonald
1	Ralph Lud
	Commander, David Taylor Naval Ship Research and Development Center, Bethesda, MD 20084
1	ATTN: Rod Peterson - Code 1240
1	Herb Wolk - Code 1740.1
1	Richard Swanek - Code 1240
	Naval Air System Command, Department of the Navy, Washington, DC 20360
1	ATTN: AIR-C3PAF
1	AIR-5164J, LT COL J. Sebolka
1	SEA-05M, Dr. Alex Kaznoff
1	SEA-05MB, LCDR W. M. Elger
1	SEA-05R 25, C. Zanis
	Naval Material Command, Washington, DC 20360
1	ATTN: MAT-0331
	Naval Post Graduate School, Monterey, CA 93948
1	ATTN: Code 57BP, R. E. Ball
	Naval Research Laboratory, 455 Overlook Avenue, Washington, DC 20375
1	ATTN: T. W. Crooker
	Commander, U.S. Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, OH 45433
1	ATTN: AFWAL/MLC
1	AFWAL/MLLP, D. M. Forney, Jr.
1	AFWAL/MLBC, Mr. Stanley Schulman
1	AFWAL/FIBC, Mr. L. G. Kelly
1	AFWAL/FIES, Mr. A. G. Kurtz
1	AFWAL/FIESD, Mr. A. Kurtz
1	AFWAL/MLLM, Dr. A. Katz
1	AFWAL/MLLM, Dr. R. Ruh
	National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, AL 35812
1	ATTN: R. J. Schwinghammer, EH01, Dir, M&P Lab
1	Mr. W. A. Wilson, EH41, Bldg. 4612
	Aeronautical Systems Division (AFSC), Wright-Patterson Air Force Base, OH 45433
1	ATTN: ASD/ENFEF, D. C. Wight
1	ASD/ENFTV, D. J. Wallick
1	ASD/XRHD, G. B. Bennett
	NASA - Ames Research Center, Army Air Mobility Research and Development Laboratory, Mail Stop 207-5, Moffett Field, CA 94035
1	ATTN: SAVDL-AS-X, F. H. Immen
	U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899
1	ATTN: Stephen M. Hsu, Chief, Ceramics Division, Institute for Materials Science and Engineering
	Director, Central Intelligence Agency, P.O. Box 1925, Washington, DC 20505
1	ATTN: OSWR-OSD, Ward Waltman
1	R. Gomez
1	J. Backofen
	United States Steel Corporation, Research Laboratory, Monroeville, PA 15146
1	ATTN: Dr. John M. Barsom

No. of Copies	To
	L. Raymond & Associates, P.O. Box 7925, Newport Beach, CA 92658-7925
1	ATTN: Dr. L. Raymond
	Boeing Advanced Systems, P.O. Box 3707, Seattle, WA 98124-2207
1	ATTN: R. J. Bristow, MS 33-04
1	W. Herlin, MS 33-04
	Sikorsky Aircraft, A Division of United Aircraft Corporation, Main Street, Stratford, CT 06601
1	ATTN: George Karas
	Teledyne CAE, 1330 Laskey Road, Toledo, OH 43697
1	ATTN: Librarian, M. Dowdell
	Fairchild Industries, Inc., Fairchild Republic Company, Conklin Street, Farmingdale, Long Island, NY 11735
1	ATTN: Engineering Library, G. A. Mauter
	Gruman Aerospace Corporation, South Oyster Bay Road, Bethpage, NY 11714
1	ATTN: Technical Information Center, J. Davis
	McDonnell Douglas Helicopter Company, 5000 E. McDowell Road, Mesa, AZ 85205-9797
1	ATTN: Library, 2/T2124,D. K. Goss
1	Mr. A. Hirko
1	Mr. L. Soffa
	IIT Research Institute, 10 West 35th Street, Chicago, IL 60616
1	ATTN: K. McKee
	Kaman Aerospace Corporation, Old Winsor Road, Bloomfield, CT 06002
1	ATTN: H. E. Showalter
	Lockheed-California Company, A Division of Lockheed Aircraft Corporation, Burbank, CA 91503
1	ATTN: Technological Information Center, 84-40, U-35, A-1
	Vought Corporation, P.O. Box 5907, Dallas, TX 75232
1	ATTN: D. M. Reedy, 2-30110
	Martin Marietta Corporation, Orlando Division, P.O. Box 5837, Orlando, FL 32805
1	ATTN: Library, M. C. Griffith
	McDonnell Douglas Corporation, 3855 Lakewood Boulevard, Long Beach, CA 90846
1	ATTN: Technical Library, C1 290/36-84
	Northrop Corporation, Aircraft Division, 3901 W. Broadway, Hawthorne, CA 90250
1	ATTN: Mgr. Library Services, H. W. Jones
	Beech Aircraft Corporation, 9709 E. Central Avenue, Wichita, KS 67206
1	ATTN: Engineering Library
	Bell Helicopter Company, A Textron Company, P.O. Box 482, Fort Worth, TX 76101
1	ATTN: J. R. Johnson
	Boeing Helicopters, P.O. Box 16858, Philadelphia, PA 19142-0858
1	ATTN: N. Caravasos, M/S P30-27
	Cessna Military, P.O. Box 7704, Wichita, KS 67277-7704
1	ATTN: Wallace
	Los Alamos National Laboratory, Los Alamos, NM 87545
1	ATTN: R. Mah
1	D. Sandstrom
1	W. Blumenthal
1	R. Barks
	Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, CA 94550
1	ATTN: C. Cline
1	R. Landingham

No. of Copies	To
	Aluminum Company of America, Alcoa Technical Center, Alcoa Center, PA 15069
1	ATTN: A. Becker
1	P. V. Kelsey
	CERCOM, Inc., 1960 Watson Way, Vista, CA 92083
1	ATTN: R. Palicka
	Ceradyne, Inc., 3169 Redhill, Costa Mesa, CA 92626
1	ATTN: E. Conabee
	Norton Co., 1 New Bond Street, Worcester, MA 01606
1	ATTN: R. Bart
	Georgia Tech Research Institute, Georgia Institute of Technology, Atlanta, GA 30332
1	ATTN: K. V. Logan
	Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78248
1	ATTN: C. Anderson
	University of California-San Diego, Dept. of Applied Mechanics & Engineering Sciences, La Jolla, CA 92093
1	ATTN: M. Meyers
	Dow Chemical Co., 1776 Building, Midland, MI 48674
1	ATTN: A. Hart
	Lanxide Corp., Tralee Industrial Park, Newark, DE 19711
1	ATTN: M. Newkirk
1	Dr. B. R. Rossing
	University of Dayton Research Institute, 300 College Park, Dayton, OH 45469
1	ATTN: S. Bless
1	N. Hecht
	SRI International, 333 Ravenswood Ave., Menlo Park, CA 94025
1	ATTN: D. Shockey
1	D. Curren
1	Dr. S. M. Johnson
	PPG Industries, Inc., Fiber Glass Research Center, P.O. Box 2844, Pittsburgh, PA 15230
1	ATTN: Dr. A. Calabrese
1	Dr. D. H. Griffiths
	Technical Center, Owens/Corning Fiberglas Corp., Granville, OH 43023
1	ATTN: Dr. W. W. Wolf
	Massachusetts Institute of Technology, Cambridge, MA 02139
1	ATTN: Dr. J. S. Haggerty, 12-009
	United Technologies Corp., Research Center Silver Lane, E. Hartford, CT 06108
1	ATTN: Dr. W. H. Sutton
1	Dr. K. M. Prewo
1	Dr. J. J. Brennan
	University of Missouri, Rolla, Materials Research Center, Rolla, MO 65401
1	ATTN: Prof. D. E. Day
	Rensselaer Polytechnic Institute, Department of Materials Engineering, Troy, NY 12181
1	ATTN: Prof. M. Tomozawa
	Worcester Polytechnic Institute, 100 Institute Road, Worcester, MA 01609
1	ATTN: Prof. G. L. Leatherman
1	Prof. T. El-Korchi
	Dow Corning Corporation, Midland, MI 48686
1	ATTN: Mr. R. E. Jones
1	Mr. W. E. Hauth
1	Mr. W. H. Atwell

No. of Copies	To
	General Electric Company, Research and Development Center, Box 8, Schenectady, NY 12301
1	ATTN: Dr. W. B. Hillig
1	Dr. R. J. Charles
	Corning Glass Works, Corning, NY 14831
1	ATTN: Dr. J. R. Booth
1	Mr. D. E. Larsen
1	Dr. W. R. Prindle
	Sandia National Laboratories, Albuquerque, NM 87185
1	ATTN: Dr. R. E. Loehman
	3M Company, 3M Center, Building 201, St. Paul, MN 55144
1	ATTN: Dr. M. Leitheiser
	Allison Gas Turbine Division, General Motors Corporation, 4438 Clayburn Drive, Indianapolis, IN 46268
1	ATTN: Dr. P. Khandelwal
	University of Delaware, Spencer Laboratory, Newark, DE 19716
1	ATTN: Prof. A. P. Majidi
	EG&G Idaho, INEL, Idaho Falls, ID 83415
1	ATTN: Dr. R. M. Neilson, Jr.
	SUNY College of Ceramics, Alfred, NY 14802
1	ATTN: Prof. D. L. Pye
	FMC Corporation, Central Engineering Laboratories, 1205 Coleman Avenue, Box 580, Santa Clara, CA 95052
1	ATTN: Dr. A. Vasudev
	GTE Laboratories, Inc., 40 Sylvan Road, Waltham, MA 02154
1	ATTN: Dr. S. T. Buljan
1	Dr. A. E. Pasto
	NASA-Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135
1	ATTN: Dr. S. Dutta
	Norton Company, High Performance Ceramics, Goddard Road, Northboro, MA 01532-1545
1	ATTN: Mr. C. A. Abel
1	Mr. N. D. Corbin
1	Dr. S. D. Hartline
	University of California, Santa Barbara, CA 93106
1	ATTN: Prof. A. G. Evans
	Georgia Tech Research Institute, Atlanta, GA 30332
1	ATTN: Dr. W. J. Lackey

No. of Copies	To
	Universal Energy Systems, 4401 Dayton-Xenia Road, Dayton, OH 45432
1	ATTN: Dr. T. Mah
	Rockwell International, 1049 Camino Dos Rios, P.O. Box 1085, Thousand Oaks, CA 91360
1	ATTN: Dr. P. E. D. Morgan
	General Electric Company, P.O. Box 8555, MS: U-1219, Philadelphia, PA 19101
1	ATTN: Dr. S. Musikant
	W. R. Grace and Company, 7379 Route 32, Columbia, MD 21044
1	ATTN: Mr. R. W. Rice
	Ceramatec, Inc., 163 W. 1700 South, Salt Lake City, UT 84115
1	ATTN: Mr. D. W. Richardson
	Coors Ceramics Company, 17750 West 32nd Avenue, Golden, CO 80401
1	ATTN: Dr. J. Stephan
1	Dr. D. W. Roy
1	Dr. R. Paricio
	Center for Advanced Ceramic Technology, Alfred University, Alfred, NY 14802
1	ATTN: Dr. R. M. Spriggs
	Carborundum Company, P.O. Box 832, Niagra Falls, NY 14302
1	ATTN: Dr. M. Srinivasan
1	Dr. J. Hinton
	The American Ceramic Society, Inc., 757 Brooksedge Plaza Drive, Westerville, OH 43081-6136
1	ATTN: Ms. L. M. Sheppard
	University of Michigan, Dow Building, Ann Arbor, MI 48109-2136
1	ATTN: Prof. T. Y. Tien
	Pennsylvania State University, 201 Steidle Building, University Park, PA 16802
1	ATTN: Prof. R. E. Tressler
	TRW, Inc., Valve Division, 1455 E. 185th Street, Cleveland, OH 44110
1	ATTN: Dr. R. R. Wills
	Director, U.S. Army Materials Technology Laboratory, Watertown, MA 02172-0001
2	ATTN: SLCMT-TML
3	Authors

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
Y-Si-Al-O-N GLASS FIBERS -
Donald R. Messier, Robert P. Gleisner,
and Ronald E. Rich

Technical Report MTL TR 89-44, May 1989, 11 pp-
tables-illus, D/A Project: 1L16102.AH42

AD________________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Fiber-reinforced composites
Glass
Oxynitride glass

The excellent mechanical properties and outstanding water corrosion resistance of Y-Si-Al-O-N glasses indicate that they are attractive candidate materials for forming into high performance glass fibers. Fibers of glasses containing, respectively, 3.2 and 6.6 wt% N were drawn freehand in air, and from glass rods in N_2. Continuous fibers (100-m long) of the former glass were melt-drawn in N_2 while being wound in air outside of the glass-melting furnace. Some fibers had diameters as small as 10 μm, and characterization data show that the fibers retain all of the desirable properties of the bulk glass.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
Y-Si-Al-O-N GLASS FIBERS -
Donald R. Messier, Robert P. Gleisner,
and Ronald E. Rich

Technical Report MTL TR 89-44, May 1989, 11 pp-
tables-illus, D/A Project: 1L16102.AH42

AD________________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Fiber-reinforced composites
Glass
Oxynitride glass

The excellent mechanical properties and outstanding water corrosion resistance of Y-Si-Al-O-N glasses indicate that they are attractive candidate materials for forming into high performance glass fibers. Fibers of glasses containing, respectively, 3.2 and 6.6 wt% N were drawn freehand in air, and from glass rods in N_2. Continuous fibers (100-m long) of the former glass were melt-drawn in N_2 while being wound in air outside of the glass-melting furnace. Some fibers had diameters as small as 10 μm, and characterization data show that the fibers retain all of the desirable properties of the bulk glass.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
Y-Si-Al-O-N GLASS FIBERS -
Donald R. Messier, Robert P. Gleisner,
and Ronald E. Rich

Technical Report MTL TR 89-44, May 1989, 11 pp-
tables-illus, D/A Project: 1L16102.AH42

AD________________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Fiber-reinforced composites
Glass
Oxynitride glass

The excellent mechanical properties and outstanding water corrosion resistance of Y-Si-Al-O-N glasses indicate that they are attractive candidate materials for forming into high performance glass fibers. Fibers of glasses containing, respectively, 3.2 and 6.6 wt% N were drawn freehand in air, and from glass rods in N_2. Continuous fibers (100-m long) of the former glass were melt-drawn in N_2 while being wound in air outside of the glass-melting furnace. Some fibers had diameters as small as 10 μm, and characterization data show that the fibers retain all of the desirable properties of the bulk glass.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
Y-Si-Al-O-N GLASS FIBERS -
Donald R. Messier, Robert P. Gleisner,
and Ronald E. Rich

Technical Report MTL TR 89-44, May 1989, 11 pp-
tables-illus, D/A Project: 1L16102.AH42

AD________________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Fiber-reinforced composites
Glass
Oxynitride glass

The excellent mechanical properties and outstanding water corrosion resistance of Y-Si-Al-O-N glasses indicate that they are attractive candidate materials for forming into high performance glass fibers. Fibers of glasses containing, respectively, 3.2 and 6.6 wt% N were drawn freehand in air, and from glass rods in N_2. Continuous fibers (100-m long) of the former glass were melt-drawn in N_2 while being wound in air outside of the glass-melting furnace. Some fibers had diameters as small as 10 μm, and characterization data show that the fibers retain all of the desirable properties of the bulk glass.