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MTL TR 92-6

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Mg-Si-Al OXYNITRIDE GLASSES

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CERAMICS RESEARCH BRANCH

February 1992

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1. REPORT NUMBER: MTL TR 92-6

2. GOVT ACCESSION NO.:

3. RECIPIENT'S CATALOG NUMBER:

4. TITLE (and Subtitle): Mg-Si-Al OXYNITRIDE GLASSES

5. TYPE OF REPORT & PERIOD COVERED: Final Report

6. PERFORMING ORG. REPORT NUMBER:

7. AUTHOR(s): Donald R. Messier and Robert P. Gleisner*

8. CONTRACT OR GRANT NUMBER(s):

9. PERFORMING ORGANIZATION NAME AND ADDRESS:
U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
SLCMT-

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS:
D/A Project: IL161102AH42

11. CONTROLLING OFFICE NAME AND ADDRESS:
U.S. Army Laboratory Command
2800 Powder Mill Road
Adelphi, Maryland 20783-1145

12. REPORT DATE: February 1992

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

*Presently at OI-NEG TV Products, Inc., Pittston, PA 18640.

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

Oxynitride glasses
Glasses
Physical properties
Mechanical properties
Metallic precipitates
Thermal decomposition

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

(SEE REVERSE SIDE)

Block No. 20

ABSTRACT

The glass-forming region at 1650°C was investigated for Mg-Si-Al-O-N compositions containing up to 20 equivalent % N (16 atomic %), and glass batches of up to 30 g were prepared and characterized. Glasses containing up to 8 atomic % N were grey colored, but transparent, while crystal-free glasses with higher N contents were unattainable. Little, if any, N loss occurred during melting; glass density, microhardness, and elastic modulus all increased with N content. The origins and consequences of the metallic precipitates that cause the grey color are discussed in the context of the literature on glasses prepared under reducing conditions.

INTRODUCTION

While a growing body of literature has evolved on oxynitride glasses,[1-6] relatively little has been reported on the preparation of such glasses in quantity. In order to evaluate the usefulness of oxynitride glasses for applications such as windows and high modulus fibers, significant quantities of glass (tens to hundreds of grams) must be prepared and characterized. By analogy with the oxide system,[7] the Mg-Si-Al-O-N glass system appeared to have great potential for the fabrication of high modulus glasses and glass fibers, and this investigation was begun to evaluate that potential.

The first synthesis of Mg-Si-Al-O-N glasses as separate phases was reported by Jack[2] who gave no property data. Loehman[3] subsequently investigated the glass-forming region in the system and reported property data on selected glass compositions noting that density, glass transition temperature, and microhardness increased and thermal expansion coefficient decreased with increasing N content. Verdier, et al.,[8] determined the glass-forming region in the ternary MgO-SiO_2-AlN at 1400°C and reported microhardness and density data for several glass compositions. Homeny and McGarry[9] synthesized and measured mechanical properties of a number of Mg-Si-Al-O-N glasses, and the glass-forming region and certain glass properties were further discussed by Hampshire, et al.,[10] in a paper that dealt with oxynitride glasses in general. Hayashi and Tien[11] presented data on the properties and crystallization of glasses melted at 1550°C with emphasis on compositions close to cordierite.

None of the aforementioned papers dealt with the preparation of Mg-Si-Al-O-N glasses in quantities greater than a few grams. Furthermore, none addressed what has become a critical issue with regard to the transparency of the glasses, and with regard to critical defects in Mg-Si-Al-O-N glass fibers; namely, the presence of very fine (1 μm or less) metallic precipitates that apparently arise from thermal decomposition of the glass during melting. The present investigation was intended to yield further information on both of these issues, as well as to provide additional data on the glass-forming region in (and properties of) glasses in the system Mg-Si-Al-O-N.

EXPERIMENTAL PROCEDURES

Batch Preparation

The compositions of the glasses used in the investigation of the glass-forming region are given in Figure 1 and other compositions are specified further on in this report. The batch components for all of the glasses investigated were Al_2O_3,* MgO,† Si_3N_4,‡ and SiO_2.◇ Ten gram batches were mixed to make the 1 g specimens used to determine the glass-forming region, and 100 g batches were mixed for larger specimens. After weighing, the components were wet-mixed for 4 h in acetone. Mixing was done in a paint mixer with the batches in polyethylene bottles containing a few Al_2O_3 balls. The batch was subsequently dried and compacted by isostatic pressing at 140 MPa in a latex bag. Some of the 1 g specimens were formed by cold pressing in a steel die. The isostatically-pressed material was broken into chunks before melting, while the cold-pressed pellets were used as formed.

*99.995% α-Al_2O_3, Gallard Schlessinger Corp., Carle Place, NY.
†Johnson-Matthey high-purity MgO.
‡Kawecki-Berylco Industries, Inc., Boyertown, PA (not currently a Si_3N_4 supplier).
◇99.5% high-purity fused SiO_2, Thermo Materials Corp., Atlanta, GA.

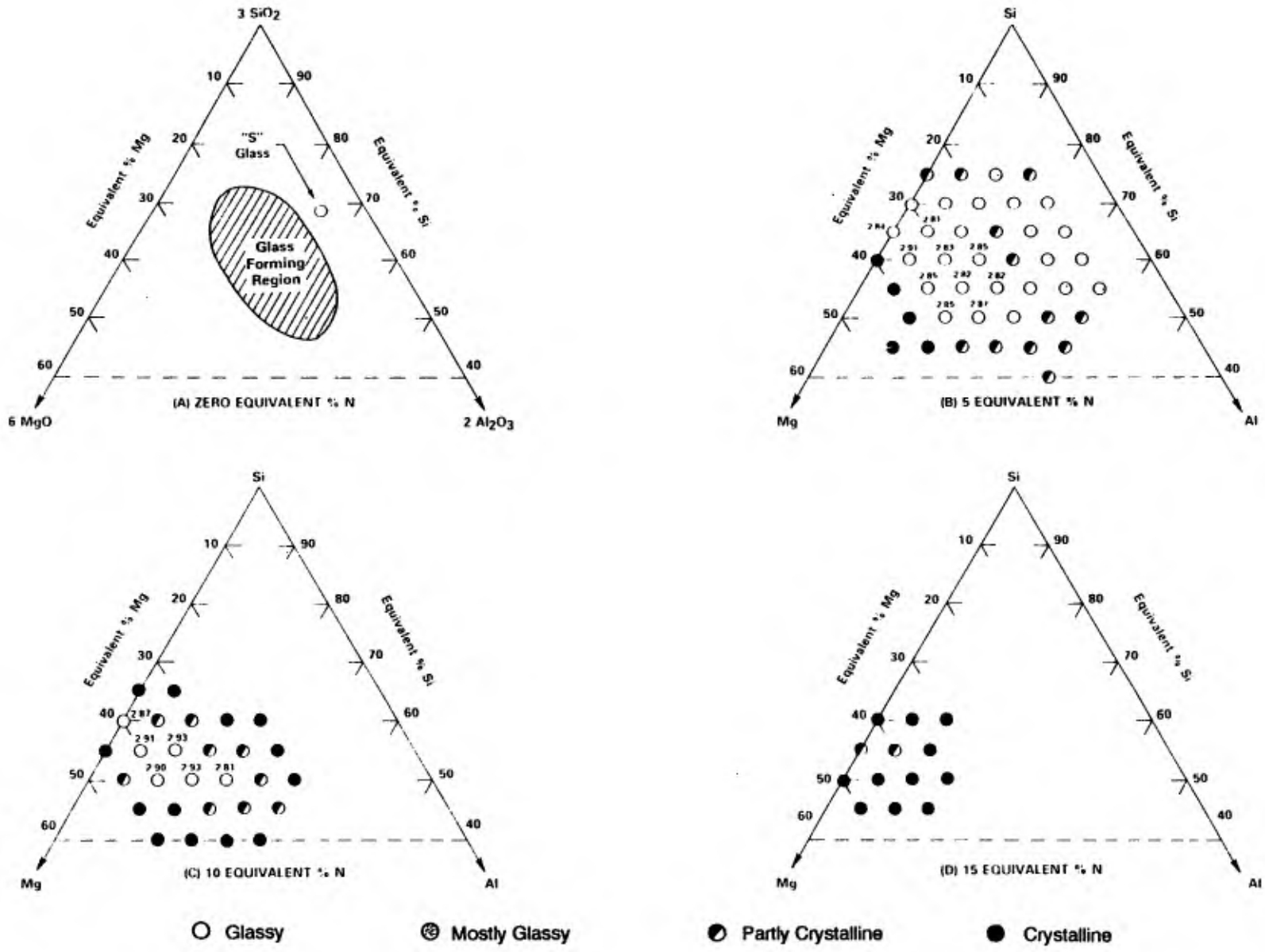

Figure 1. Glass-forming region at 1650°C in the system Mg-Si-Al-O-N. (A) was adapted from McMillan,[1] and (B) through (D) are from this work. The numbers are densities of glasses whose compositions correspond to the adjacent data points.

Glass Preparation

Experiments to determine the glass-forming region in the system Mg-Si-Al-O-N were done with 1 g specimens contained in individual compartments in BN crucibles that held three specimens each. Up to three such compartmented crucibles were loaded into large, covered, Al_2O_3 or BN crucibles for heating in a water-cooled resistance furnace with a W-mesh heating element.* The specimens were heat treated at 1650°C in N_2 at 200 kPa for 15 to 30 minutes and subsequently furnace-quenched and removed for examination. The specified heating times were sufficiently long for the complete melting of these small specimens. Removal was generally easy with most of the specimens having melted into spheres about 5 mm in diameter.

The larger (15 to 30 g) glass batches were melted in covered Al_2O_3 or graphite crucibles. In either case, the insides of the crucibles were slurry-coated with high-purity BN powder that served as a mold release. Melting times of one to four hours were required for homogenization of these batches, and melting temperatures of 1675°C were required for some compositions. These specimens were also furnace-quenched and readily removed from the crucibles in which they were melted.

Characterization

The glasses were characterized by several analytical techniques. The main tool used to determine the presence of crystalline phases was optical microscopy; this proved to be a more sensitive technique than X-ray diffraction done on some of the glasses. Chemical analyses were as indicated in Table 1, and densities were measured on polished sections using a Knoop indenter and a 0.98 N load. Elastic modulus measurements were done on machined glass disks by the same ultrasonic technique discussed in an earlier paper.[12]

Table 1. CHEMICAL ANALYSIS OF Mg-Si-Al-O-N GLASS
GLASS FORMULA: $Mg_8Si_{24}Al_8O_{57}N_5$

Element	Nominal Starting Composition (wt%)	Analyzed Glass Composition (wt%)
Si	33.5	35.69
Mg	9.5	9.02
Al	8.0	8.98
Ca	-	0.04
Fe	-	0.01
Y	-	0.02
Na	-	0.04
C	-	0.03
S	-	0.012
O	45.6	42.29
N	3.5	3.92

*Centorr Associates, Suncook, NH.

RESULTS

Figure 2 shows polished disks of Mg-Si-Al-O-N glasses containing 5 e/o N and 10 e/o N, respectively. Both disks are very transparent and light grey in color.

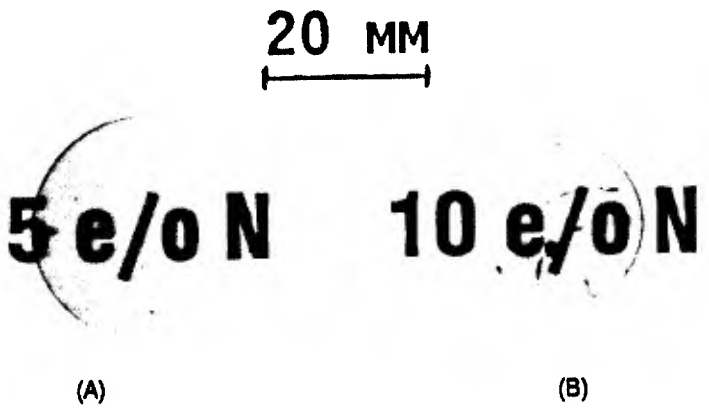

Figure 2. Disks of Mg-Si-Al-O-N glass containing (A) 5 e/o N and (B) 10 e/o N, respectively. Each disk is 5 mm thick. The disk in (B) contains a few cracks and crystalline inclusions.

The criteria that were used in determining the glass-forming region are illustrated by Figures 3A through 3D which show optical photomicrographs of various test specimens. Figure 3A is a polished, unetched section of a glass specimen of the same composition as the transparent disk shown in Figure 2B. The only observable inclusions are a few scattered white specks that represent metallic precipitates in the glass. Figures 3B and 3C are polished, unetched sections of specimens that were designated, **partly crystalline** and **crystalline**, respectively. Figure 3D is a transmitted light optical photomicrograph of a fragment of glass from a specimen of the type that was designated **mostly glassy**, showing the μm-sized precipitates found in such specimens. As discussed below, it is likely that those precipitates are metallic Si, or at least Si-rich. Such **mostly glassy** specimens contained no crystalline phases (oxides or oxynitrides) of the type evident in Figures 3B and 3C.

The results of experiments to determine the glass-forming region in the system Mg-Si-Al-O-N at 1650°C are summarized in Figure 1. The representation of compositions in equivalents, as shown in Figure 1, was proposed by Jack[2] to be particularly convenient for oxynitride systems. Figure 1 consists of ternary sections representing compositions containing 0, 5, 10, and 15 e/o N, respectively. Compositions containing higher N contents (20 e/o) were prepared but not formed glasses. The equivalent percentages reported are based upon 100 total equivalents of cations and anions; i.e., 50 e/o of O + N, and 50 e/o of cations. The data on the glass-forming region in the oxide system are from McMillan.[13] The numbers adjacent to the data points representing **glassy** oxynitride compositions are density values (in g/cm^3) for those glass compositions.

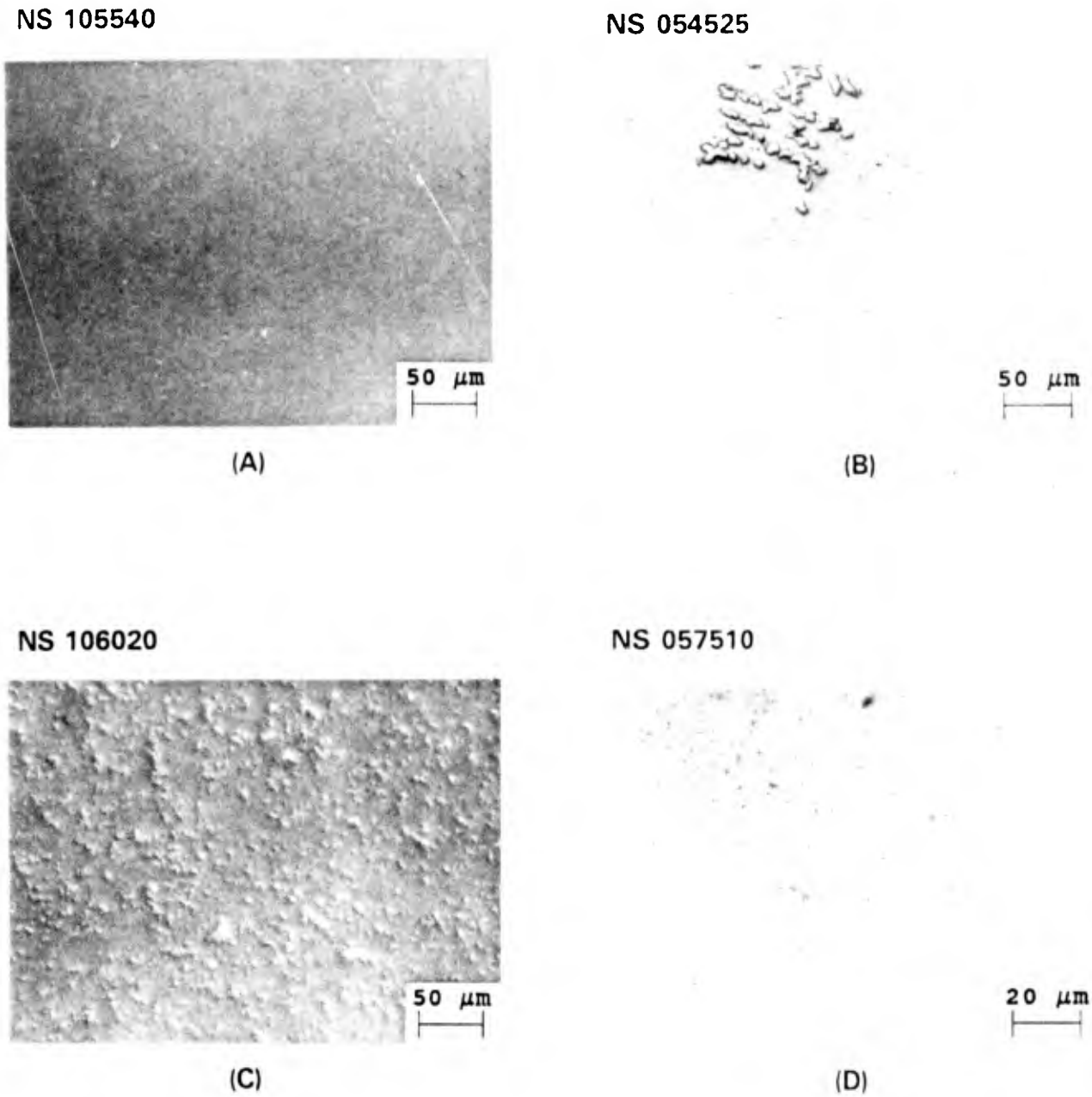

Figure 3. Optical photomicrographs of Mg-Si-Al-O-N glasses. (A) through (C) are polished sections photographed with reflected light, and (D) is a glass fragment taken with transmitted light. (A) and (D) are specimens characterized as glassy and mostly glassy, respectively, and (B) and (C) as partly crystalline and crystalline, respectively

Table 1 compares nominal starting compositions with chemical analysis data for glasses containing 6 e/o N and 10 e/o N, respectively; the first of the two is a high Al_2O_3 composition suitable for fiber-drawing while the second, a high MgO, composition is the same as the glass shown in Figures 2B and 3A.

Figure 4 is a plot of microhardness versus N content for 23 glasses of varying cation composition. While such measurements were done for virtually all of the compositions evaluated, the microhardness data in Figure 4 are limited to glasses containing no detectable oxide or oxynitride crystalline phases.

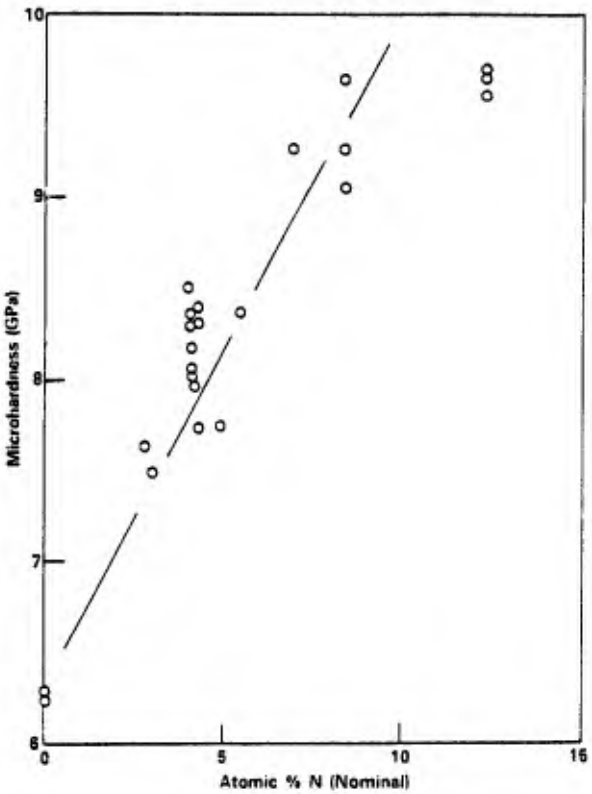

Figure 4. Microhardness versus nitrogen content for Mg-Si-Al-O-N glasses. All measurements were made with a Knoop indenter and a 0.98 N load.

Elastic property data from ultrasonic measurements on machined glass disks are listed in Table 2. Also included in the table are data for a composition similar to "S" glass[7] but melted in N_2 under the same conditions as the oxynitride glasses. The density values listed in Table 2 were determined on the same specimens used to obtain the modulus data.

Table 2. ELASTIC MODULUS MEASUREMENTS ON GLASS DISKS

Glass Composition (Atom %)	Density (g/cm^3)	E (GPa)	Poisson's Ratio	E/Density
$Mg_5Si_{22}Al_{10}O_{56}N_4$	2.58	111.1	0.266	43.1
$Mg_5Si_{22}Al_{10}O_{60}N_3$	2.56	105.8	0.265	43.1
$Mg_5Si_{22}Al_{10}O_{63}$ ("S" Glass)	2.49	89.7	0.194	36.0
$Mg_{21}Si_{17}Al_4O_{54}N_4$	2.88	125.3	0.296	43.5
$Mg_{24}Si_{17}Al_2O_{49}N_8$	2.94	135.3	0.293	46.0

DISCUSSION OF RESULTS

Any plot, such as the ones given in Figure 1 outlining a glass-forming region, has a certain arbitrariness. It is likely that heating at higher temperatures, holding for longer times, or quenching at faster rates all could expand the region shown. Nevertheless, the data presented can reasonably be assumed to define a minimum region of compositions that form glasses in the system Mg-Si-Al-O-N. Comparison with previous results on the glass-forming region[3-5,8-11] is inexact because of differences in experimental procedures and in how the data were reported. Nevertheless, the glass-forming region found in this study is slightly less extensive than, but otherwise in good agreement with, the results of Drew, Hampshire, and Jack[4,5,10] for melts of similar equivalent concentrations of N at a slightly higher temperature (1700°C) than the 1650°C in this study. Loehman[3] reported a slightly larger glass-forming region, also at 1700°C, but comparison is difficult because of the way his data were obtained; his compositions all contained 10 wt% Si_3N_4 and, thus, had varying equivalent concentrations of N. The data of Verdier, et al.,[8] were for compositions in the ternary, MgO-SiO_2-AlN at 1400°C and not directly comparable to any of the other studies, including the present one.

Figure 1 shows clearly that the glass-forming region gets smaller and moves toward the Si-Mg and join as the N concentration increases. Furthermore, it was impossible to prepare glasses from compositions containing 20 e/o N under our experimental conditions, and Figure 1 shows that crystal-free glasses were unattainable at 15 e/o N. Considering the foregoing, it is likely that the N solubility limit for glasses in the system Mg-Si-Al-O-N is around the 15 e/o N level.

As has been commonly observed for many oxynitride glass systems,[3,6] the data given in Figure 1 indicate that glass density increases with N content. Not apparent from the figure is that, as observed in this study, density also increases with MgO content. In recent work, we have made low MgO-content Mg-Si-Al-O-N glasses containing 5 e/o N with densities as low as 2.58 g/cm^3, some elastic properties of which are given in Table 2.

While it is probable that loss of the volatile MgO constituent contributed significantly to the observed weight losses of 1% to 2% that typically occurred upon melting, the chemical analysis results in Table 1 show a net gain of Mg as compared to the nominal starting composition. The reason for this is not understood at present, but similar results were obtained in chemical analyses of Mg-Si-Al-O-N glass fibers. It is noted that the N contents of the glasses increased slightly, which is perhaps surprising, however, it is consistent with the findings of Verdier, et al.[8] who found that virtually all of the N in the starting batch was retained in the Mg-Si-Al-O-N glasses that they prepared.

The observed increases in microhardness with N content support previous findings on various oxynitride glasses,[3-6,8-12] as does the increases in elastic modulus over oxide glasses. Of special interest for glass fiber applications are the relative modulus (modulus/density) values in Table 2 which are significantly higher than the values for "S" glass. The listed modulus value of 89.7 GPa for our "S" glass (melted in N_2) is a little higher than the literature value of 84 GPA,[7] possibly due to incorporation of N into the glass during melting. Use of the lower modulus value for calculating relative modulus would make the oxynitride glass data even more impressive.

A key issue in the assessment of the usefulness of oxynitride glasses for engineering applications concerns the metallic precipitates commonly found in such glasses. As proposed by several workers,[1,14,15] such precipitates cause the characteristic grey color commonly observed in oxynitride glass fibers.[15]

The origins of metallic precipitates in oxynitride glasses were first discussed by Kelen and Mulfinger.[1] They stated that the fine Si particles in their oxynitride glasses arose from a process similar to that first proposed by earlier investigators[16,17] for other types of glasses. Zintl[16] stated that the darkening of silica glass heated in reducing atmospheres was due to finely divided Si that came from the disproportionation of SiO to Si and SiO_2. Geld and Esin[17] investigated in detail the behavior of slags heated in reducing atmospheres, and presented persuasive evidence that the Si particles observed in the quenched glasses resulted from the same type of disproportionation reaction. As illustrated in Figure 5, Geld and Esin proposed that the Si^{+2} in the molten slags came from the reduction of Si^{+4} during heating. They further suggested that Si precipitated during quenching via the second reaction shown in Figure 5.

1. REDUCTION OF Si^{+4} IN THE GLASS DURING MELTING:

$$Si^{+4} + O^{-2} = Si^{+2} + 1/2\ O_2\ (g)$$

2. OXIDATION/REDUCTION OF Si^{+2} DURING COOLING:

$$2\ Si^{+2} = Si^{+4} + Si\ (s)$$

Figure 5. Proposed mechanism for formation of fine Si precipitates in Mg-Si-Al-O-N glasses from Geld and Esin.[17]

An interesting consequence of the foregoing discussion is that the metallic precipitates found in oxynitride glasses are not so much a result of the presence of N in the glass as they are of the necessity of the use of a highly reducing N_2 atmosphere for glass-melting. It follows, therefore, that a potential means of lowering the content of metallic particles in such glasses would be to make the melting environment sufficiently oxidizing to prevent the reduction of Si^{+4} to Si^{+2} but not oxidizing enough to remove the N from the glass. Initial attempts to do this were unsuccessful, probably because of unfavorable kinetics of interaction between the molten glass and the furnace atmosphere. Additional experiments are planned, however, to test this approach further.

ACKNOWLEDGMENT

We appreciate the capable assistance of R. E. Rich with the experimental portion of this study.

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Physical properties

Technical Report MTL TR 92-6, February 1992 11 pp-
illus-tables, D/A Project: IL161102AH42

The glass-forming region at 1650°C was investigated for Mg-Si-Al-O-N compositions containing up to 20 equivalent % N (16 atomic %), and glass batches of up to 30 g were prepared and characterized. Glasses containing up to 8 atomic % N were grey colored, but transparent, while crystal-free glasses with higher N contents were unattainable. Little, if any, N loss occurred during melting; glass density, microhardness, and elastic modulus all increased with N content. The origins and consequences of the metallic precipitates that cause the grey color are discussed in the context of the literature on glasses prepared under reducing conditions.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
Mg-Si-Al OXYNITRIDE GLASSES -
Donald R. Messier and Robert P. Gleisner

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Key Words

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