

Formation and properties of nitrogen-rich strontium silicon oxynitride glasses

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Abstract Glass formation in the system Sr–Si–O–N was investigated and properties of obtained glasses evaluated. The glass-forming region was determined for glasses prepared by melting mixtures of Sr metal, SiO₂ and Si₃N₄ powders in Nb crucibles at 1600–1750 °C in nitrogen atmosphere using a radio frequency furnace. The glasses were found to be homogenous, translucent gray to opaque black, and to contain high contents of N (up to 45 e/o) and Sr (up to 36 e/o). The glass transition temperature (790–973 °C), density (2.99–4.07 g/cm³), microhardness (8.10–10.50 GPa), and refractive index (1.65–1.93) are strongly correlated with the amounts of Sr and N. The properties are compared with findings in other oxynitride silicate glass systems.

Introduction

Oxynitride glasses have received considerable attention, from both scientific and technological points of view, since the late 70s due to their properties compared with their oxide glass counterparts. Recent studies have shown that La–Si–O–N [1] and Ca–Si–O–N [2] glasses can be formed over wide compositional ranges by using the electropositive elements in their metallic state as starting materials. The glasses retain high amounts of the electropositive elements and nitrogen during formation and exhibit very

high values of glass transition temperature, microhardness, and refractive index.

Oxynitride glasses in M–Si–O–N and M–Si–Al–O–N systems (with M a modifying cation such as Li, Na, K, Mg, Ca, Ba, Y, La, Pr) [1–15] have been prepared with a wide variety of compositions. It has been found that the glass-forming capability increases initially as nitrogen is added in the form of Si₃N₄, but then reaches a maximum and decreases upon further addition [3, 4, 16]. Glasses in RE-sialon and RE–Si–O–N systems (with RE = Ln or Y) [12–15] have been studied in more detail than those in alkaline earth metal systems. An exception is Mg–Si–Al–O–N [3, 4, 16–20] glasses, which have been investigated extensively due to that a combination of MgO and Al₂O₃ can be used as a sintering aid for Si₃N₄ or sialon ceramics, for obtaining dense ceramics at relatively low temperatures where no decomposition of Si₃N₄ occurs.

Very little research has been carried out so far in the system Mg–Si–O–N [4, 9, 21]. The reported glass formation range is relatively narrow, with Mg contents from 15 to 28 e/o and N contents from 10 to 12 e/o. For compositions outside of these ranges, phase-separated glasses were obtained. Addition of Al₂O₃ was found to suppress the phase separation and considerably broaden the glass-forming region [3, 4, 22].

In the Ca–Si–Al–O–N [3, 4, 23–25] and Ca–Si–O–N [3, 16] systems, only a few glass compositions have been synthesized by traditional synthesis routes, in which mixtures of SiO₂, Si₃N₄, and metal oxides are melted. The incorporated amounts of Ca and N are rather small. The properties of Ca-sialon glasses are quite similar to those of Mg-sialon glasses. Ca–Si–O–N glasses have a propensity for phase separation, resulting in opaque, milky appearances that closely resemble those of Mg–Si–O–N glasses. However, a newly adopted synthesis method has enabled

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the formation of glasses in a wider compositional range in the system Ca–Si–O–N [2], extending considerably glass contents of nitrogen as well as calcium. In this new synthesis route, CaH₂ is used as a precursor instead of CaO. The reaction kinetics is enhanced as CaH₂ decomposes to fine-grained and nominally oxygen-free Ca metal at a relative low temperature, ca. 250 °C. It subsequently reacts with nitrogen gas and forms a nitride, which reacts with SiO₂ and Si₃N₄ to a melt. The glasses obtained in the Ca–Si–O–N system by this route are homogenous and with high concentrations of Ca (42 e/o) and N (58 e/o).

A number of investigators have outlined the limits of the glass-forming region in the Ba–Si–Al–O–N system [10, 26]. Tredway [10] reported glass transition temperatures, thermal expansion coefficients, and indentation hardness values for Ba–Si–Al–O–N glasses in a narrow compositional range and discussed general property trends as a function of nitrogen content. The glasses were microscopically homogeneous, although macroscopic segregation of metallic particles (Si and Fe) of approximately 1 mm in diameter was observed.

To our knowledge, there are so far no reports on Sr–Si–O–N or Sr–Si–Al–O–N glasses and their properties. This article presents the results of a determination of the glass-forming region in the Sr–Si–O–N system, as well as values of glass transition temperature, density, microhardness, and refractive index. The glass-forming region and the measured properties are compared with those of previously reported glasses in alkaline earth–Si–O–N systems. In addition, property variations are discussed with respect to the effect of nitrogen and strontium contents in the glasses.

Experimental

Glasses were prepared in 5-g batches using Sr metal (99.9% in mineral oil, ABCR GmbH & Co.), SiO₂ (99.9%, ABCR GmbH & Co.), and Si₃N₄ (ChemPur GmbH) powders. After thorough dry mixing in a glove box in an argon atmosphere, they were melted in niobium crucibles in nitrogen atmosphere at temperatures between 1600 and 1750 °C using a radio frequency furnace. The mixtures were kept fused for 2–4 h and then cooled by turning off the furnace at the end of the run.

X-ray powder diffraction (XRPD) analysis, using a Guinier–Hägg camera and CuK α ₁ radiation, was used to confirm that the glasses were amorphous. Cation concentrations were determined by energy dispersive X-ray (EDX) point analysis on polished and carbon-coated surfaces, using a JSM 7000F scanning electron microscope (SEM) equipped with a Si detector and a LINK INCA program system. Oxygen and nitrogen contents were determined by combustion analysis using a LECO equipment.

The densities were measured according to the Archimedes principle, in distilled water. Measured glass densities were reproducible to ± 0.005 g/cm³. The glass transition temperature (T_g) and crystallization temperature (T_c) for crushed pieces of glass were measured using differential thermal analysis (DTA) up to 1350 °C in flowing nitrogen by means of a SETARAM TG-DTA 1600 instrument, using Pt cups and a heating rate of 25 °C/min. The onset point of an endothermic drift on the DTA curve was taken as representing T_g , and the onset of an exothermic peak as corresponding to T_c . The precision in the T_g and T_c measurement was estimated to be ± 5 °C. Vickers hardness values were determined at room temperature using a Matsuzawa microhardness tester, Model MXT- α 1, with a pyramid-shaped diamond indenter. A load of 300 g was applied on polished glass samples with at least 12 indentations were made on each sample. The indentation dimension for each sample was measured with a light microscope (Olympus PMG3, Japan) under a magnification of 200 \times and hardness values were calculated in GPa using the expression $H_V = (18.42 P)/d^2$, where $P(N)$ is the load applied on the indenter and d (μ m) is the average indentation diagonal length. The refractive index (RI) of polished glasses was estimated by measuring the Brewster's angle θ_B , for a laser light source operating at $\lambda = 640$ nm. The refractive index was calculated by using the equation $n = \tan(\theta_B)$. Errors in the measurement of refractive index were estimated to be on the average 0.05, corresponding to an error of ca. 2° in $2\theta_B$.

Results and discussion

Glass-forming region

Samples with different compositions and high contents of strontium and nitrogen were heat-treated at 1600–1750 °C in order to demarcate the glass-forming region. Figure 1 shows the initial compositions; with circles representing compositions that formed glasses and circles with an inserted C representing those that were found partially crystalline. The observed glass-forming region, for samples with determined cation and anion concentrations, is shown in Fig. 2, together with previously reported glass-forming regions in the systems Mg–Si–O–N [4, 9] and Ca–Si–O–N [2, 3]. It extends to Sr contents up to 36 e/o and N contents up to 45 e/o. While glass forming in the system Sr–Si–O–N has not been studied before, the found glass-forming region extends to significantly higher N contents than those reported for the other alkaline earth containing systems with the exception of our findings for the Ca–Si–O–N [2] system. We believe that the extension can be attributed to the use of elemental Sr as starting material, since studies of

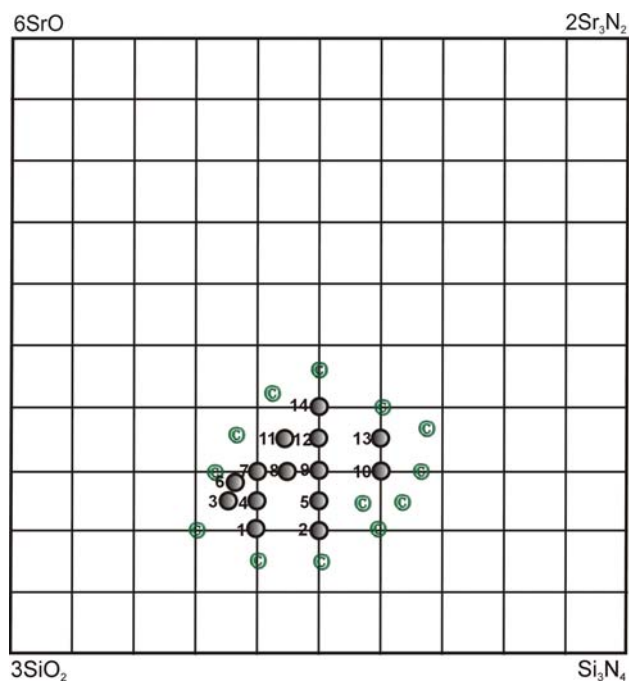


Fig. 1 Starting compositions heat-treated in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system. Filled circles represent glasses formed and circles inserted with “C” represent partially crystalline samples

the systems M–Si–O–N with M = La [1], Pr [27], and Ca [2] have shown that large glass-forming regions are achieved when using metal precursors for the electropositive elements. The determined glass-forming region is strongly elongated along a line with a constant Sr:N ratio of 1.37, implying that an increase in N content is accompanied roughly by a corresponding increase in Sr content. The X:Si (X = O, N) ratios, see Table 1 and Fig. 2, are found to vary between 2.2 and 2.8, and with the exception of two glasses to fall in the range of 2.5–2.7. Since the X:Si ratio is a measure of the average connectivity of the glass network, the similar X:Si ratios indicate that the structures of the glasses are similar and do not change much with N content.

The element analyses of the glasses showed that considerable shifts in composition had occurred during the heat treatment, as seen in Table 1. Nitrogen loss was found to be dominant, about 10–30 e/o, along with loss of Si and a smaller amount of Sr. The element losses increased with increasing temperature and are attributable to decompositions of the melt at the comparatively high preparation temperatures. During heating, a strong exothermic reaction was observed at temperatures 650–800 °C, associated with a rapid nitridation of the Sr metal, resulting in the formation of both crystalline and amorphous phases, which then melted upon a further increase of the temperature. The temperature at which a melt formed was found to increase with increasing amount of Si₃N₄ and decrease with

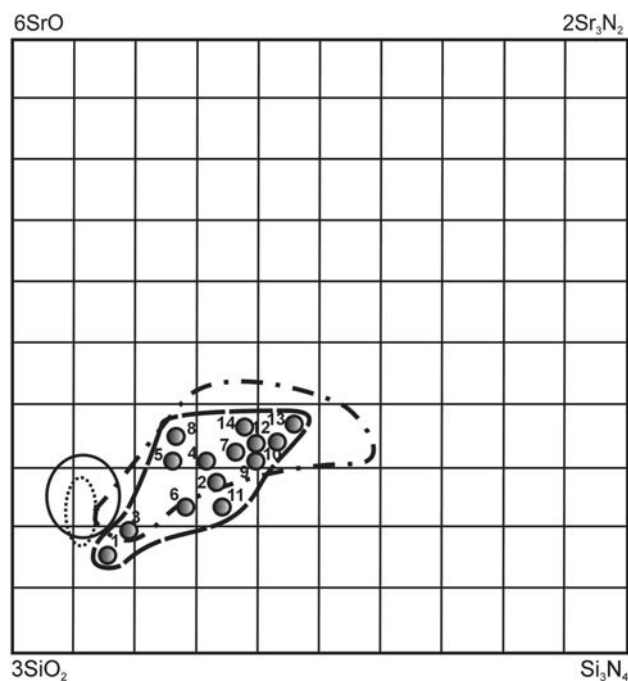


Fig. 2 Quaternary diagram showing glasses obtained in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system together with glass-forming regions in other alkaline earth silicon oxynitride systems. Dashed line shows glass-forming region in Sr–Si–O–N system at 1600–1750 °C. Dashed-dotted line shows glass-forming region in Ca–Si–O–N [2] system at 1550–1650 °C. Solid circle shows glass-forming region in Ca–Si–O–N [3] and Mg–Si–O–N [4] system at 1700 °C (the glass-forming region in both systems reported by Hampshire and Drew are almost identical). Dotted ellipse shows glass-forming region in Mg–Si–O–N [4, 9] system at 1600–1700 °C

increasing Sr content. The time for melt formation was also found to decrease with increasing Sr content.

The obtained glasses were homogeneous, with colors varying from translucent gray to opaque black. They were found to contain impurities in the form of 0.1–2 μm large spherical particles of Sr silicide, together with smaller amounts of elemental Si, which we hold probable to have primarily formed from a decomposition of the silicon nitride during heating. The amounts of these impurities were found to be typically around 1–2 vol% and less than 5 vol% for all prepared glasses as shown in Fig. 3, for a sample (no. 10 in Table 1). The presence of them is evidently unwanted and is assumed to be the cause of the low glass transparency. They may furthermore act as nucleation sites for crystallization of the glasses.

Density

Measured physical properties of the Sr–Si–O–N glasses are given in Table 1. A discrimination of the separate effects of the Sr and N contents on physical properties is hindered by the strong correlation of the Sr and N contents. For

Table 1 Compositions of starting mixture, determined glass composition, Sr content in e/o, N content in e/o, N content in wt%, X:Si ratio = [O, N]/[Si], density (ρ), glass transition temperatures (T_g) and crystallization temperatures (T_c), microhardness (H_V), and refractive index (RI)

Glass ID	Starting composition	Glass composition	Sr (e/o)	N (e/o)	N (wt%)	X:Si	ρ (g/cm ³)	T_g (°C)	T_c (°C)	H_V (GPa)	RI
Sr1	Sr ₅ Si ₁₀ O ₁₅ N _{6.67}	Sr _{3.33} Si ₁₀ O _{19.92} N _{2.27}	14.5	14.6	3.45	2.22	2.99	790	920	8.1(4)	–
Sr2	Sr ₅ Si ₁₀ O _{12.5} N _{8.33}	Sr _{8.02} Si ₁₀ O _{19.29} N _{5.82}	28.6	31.1	5.94	2.51	3.80	940	1080	9.3(2)	1.68
Sr3	Sr _{6.66} Si ₁₀ O _{17.33} N _{6.22}	Sr _{4.94} Si ₁₀ O _{20.30} N _{3.09}	19.8	18.6	4.01	2.34	3.23	–	–	8.2(4)	–
Sr4	Sr _{6.66} Si ₁₀ O ₁₆ N _{7.11}	Sr _{8.52} Si ₁₀ O _{19.55} N _{5.98}	29.8	31.0	5.88	2.55	3.72	920	1105	9.1(2)	1.66
Sr5	Sr _{6.66} Si ₁₀ O _{13.33} N _{8.89}	Sr _{9.41} Si ₁₀ O _{22.21} N _{4.80}	32.0	25.0	4.40	2.70	3.69	875	970	8.5(2)	1.73
Sr6	Sr _{7.40} Si ₁₀ O _{18.08} N _{6.21}	Sr _{5.98} Si ₁₀ O _{18.96} N _{4.68}	23.0	27.0	5.59	2.55	3.51	–	–	–	–
Sr7	Sr _{8.57} Si ₁₀ O _{17.14} N _{7.61}	Sr _{9.23} Si ₁₀ O _{19.50} N _{6.48}	31.6	33.3	6.08	2.60	3.86	915	1060	9.9(5)	1.68
Sr8	Sr _{8.57} Si ₁₀ O _{15.71} N _{8.57}	Sr _{9.99} Si ₁₀ O _{22.74} N _{4.84}	33.5	24.1	4.27	2.76	3.90	845	970	8.2(3)	1.74
Sr9	Sr _{8.57} Si ₁₀ O _{14.29} N _{9.52}	Sr _{8.67} Si ₁₀ O _{17.67} N _{7.33}	30.3	38.4	7.20	2.50	3.83	973	1120	10.1(6)	1.77
Sr10	Sr _{8.57} Si ₁₀ O _{11.43} N _{11.43}	Sr _{10.41} Si ₁₀ O _{18.06} N _{8.23}	34.3	40.6	7.22	2.63	4.01	960	1106	9.5(5)	1.88
Sr11	Sr _{10.77} Si ₁₀ O _{13.5} N ₁₁	Sr _{7.66} Si ₁₀ O _{20.27} N _{4.93}	27.6	26.3	5.14	2.52	3.59	883	1020	8.7(3)	1.65
Sr12	Sr _{10.08} Si ₁₀ O _{15.38} N _{11.28}	Sr _{9.50} Si ₁₀ O _{18.26} N _{7.49}	32.2	38.1	6.95	2.58	3.90	970	1100	9.4(3)	1.76
Sr13	Sr _{10.08} Si ₁₀ O _{12.31} N _{12.31}	Sr _{11.23} Si ₁₀ O _{17.19} N _{9.36}	36.0	45.0	7.85	2.66	4.00	965	1124	9.9(4)	1.93
Sr14	Sr _{13.33} Si ₁₀ O _{16.67} N _{11.11}	Sr _{11.05} Si ₁₀ O _{19.08} N _{7.98}	35.6	38.6	6.71	2.71	4.07	930	1100	10.5(6)	1.87

Numbers in parentheses are estimated standard deviations

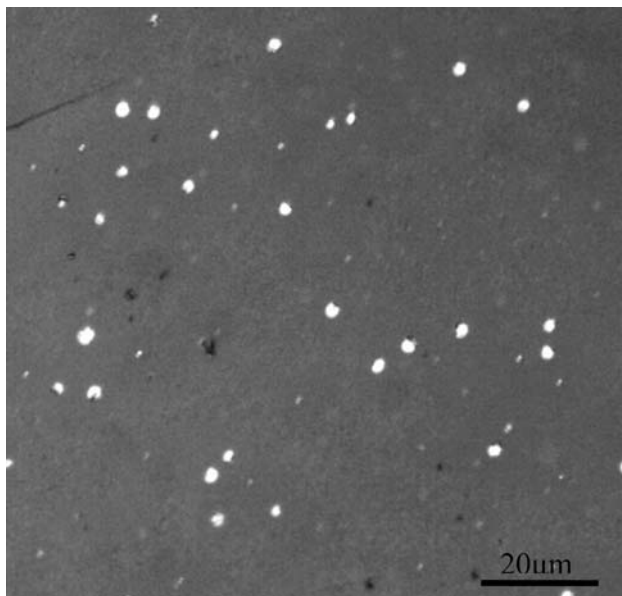


Fig. 3 Light microscope image showing the presence of Sr silicide in a glass (no. 10 in Table 1)

visualization of the variations in physical properties, they are illustrated in three-dimensional plots in Fig. 4. Further analysis is made under the assumption of a linear and independent dependence of the properties on Sr and N content, as has been shown to be valid for other oxynitride silicate glasses [28, 29].

The density of the Sr–Si–O–N glasses varies from 2.99 to 4.07 g/cm³ and increases with increasing Sr content. The highest density of 4.07 g/cm³ is found for a glass with composition Sr_{11.05}Si₁₀O_{19.08}N_{7.98}. Regression analysis

yielded $\rho = 2.34(8) + 0.039(4) \cdot [\text{Sr}] + 0.008(3) \cdot [\text{N}]$ g/cm³, with Sr and N concentrations in e/o, demonstrating that the density variations depend primarily on the Sr content. The density as a function of Sr content is shown in Fig. 5. The increase in density with Sr content can be attributed to the large atomic weight of Sr, while the smaller increase with N content may be due to a significant fraction of three-coordinated N in the glasses.

Glass transition and crystallization temperatures

Glass transition temperatures (T_g) and crystallization temperatures (T_c) are listed in Table 1 and a DTA recording for the glass no. 10 shown in Fig. 6. The glass transition temperatures range from 790 to 970 °C and crystallization temperatures from 920 to 1124 °C. The average temperature difference between T_g and T_c is approximately 145 °C.

Regression analyses yielded $T_g = 739(26) - 2(1) \cdot [\text{Sr}] + 7(1) \cdot [\text{N}]$ °C and $T_c = 851(27) - 3(1) \cdot [\text{Sr}] + 9(1) \cdot [\text{N}]$ °C. The results indicate that T_g , as well as T_c , depends primarily on the N content, as shown in Fig. 7, but that there is also a small negative dependence on the Sr content. The in general observed increase in T_g with N content for oxynitride silicate glasses has been attributed to an increased crosslinking of the glass network by three-coordinated nitrogen.

Vickers hardness

The microhardness shows a small variation between 8.10 and 10.51 GPa. Regression analysis yielded $H_V = 6.7(4) - 0.02(2) \cdot [\text{Sr}] + 0.09(1) \cdot [\text{N}]$ GPa, indicating a

Fig. 4 Three-dimensional graphs for properties in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system; **a** X:Si ratio, **b** density, **c** glass transition temperature, **d** glass crystallization temperature, **e** hardness, and **f** refractive index

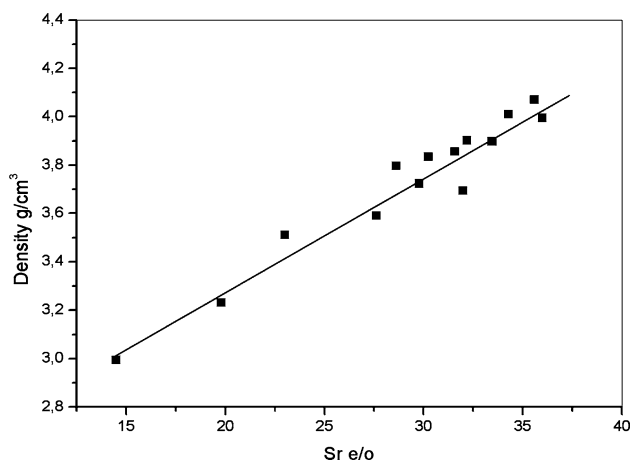
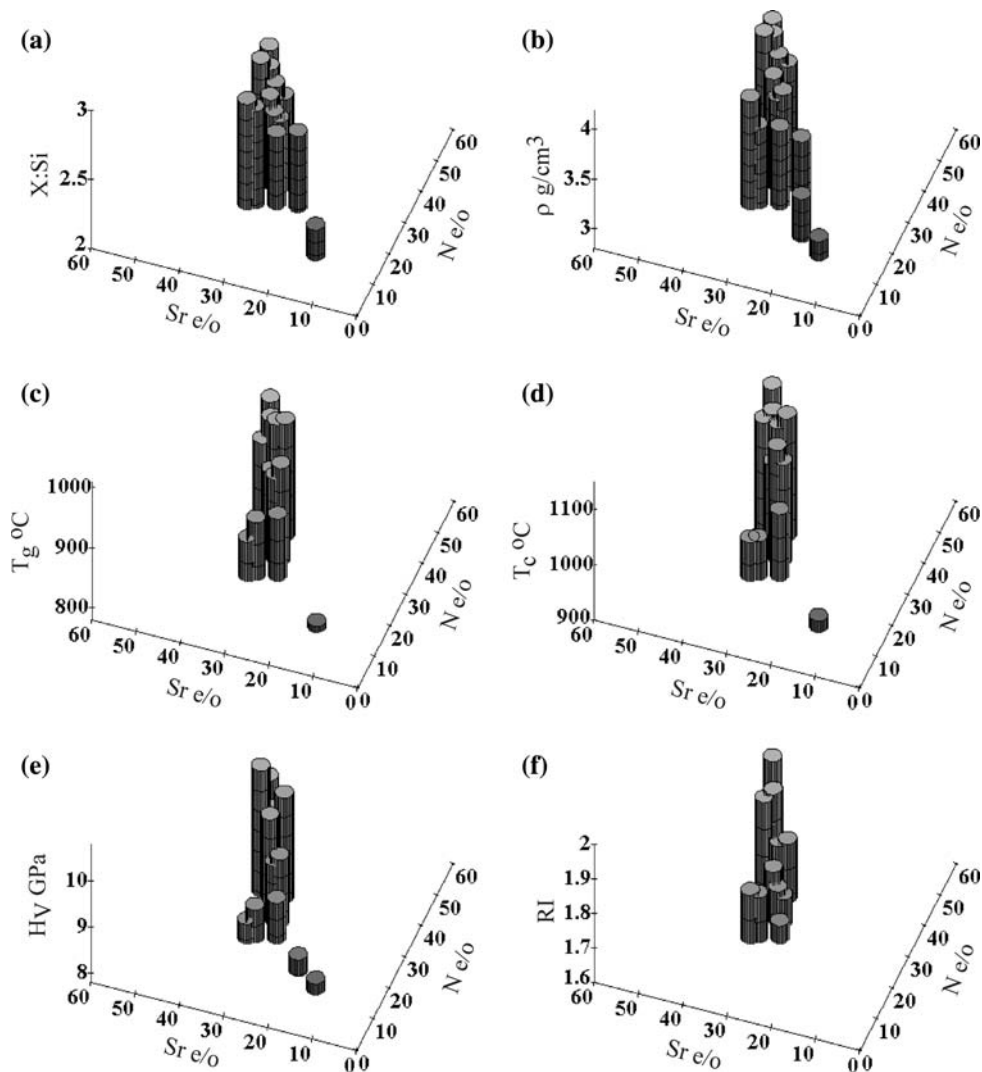


Fig. 5 Glass density as a function of Sr content in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system

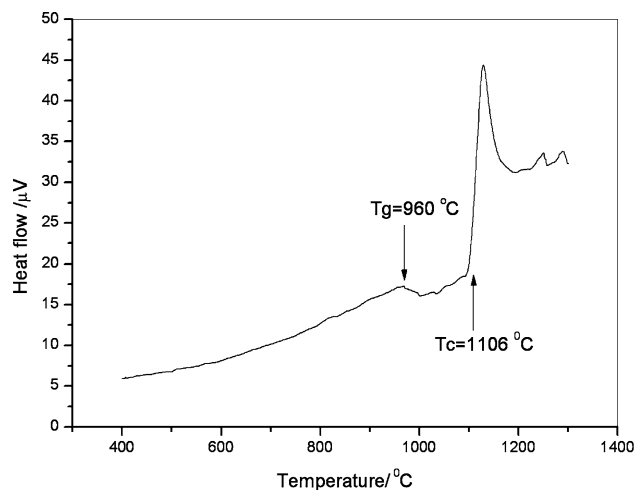


Fig. 6 DTA recording for Sr_{10.41}Si₁₀O_{18.06}N_{8.23} glass (sample no. 10)

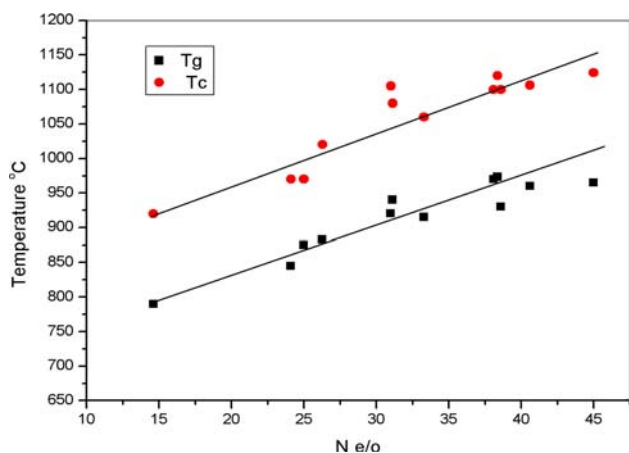


Fig. 7 Glass transition (T_g) and glass crystallization (T_c) temperatures as a function of N content in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system

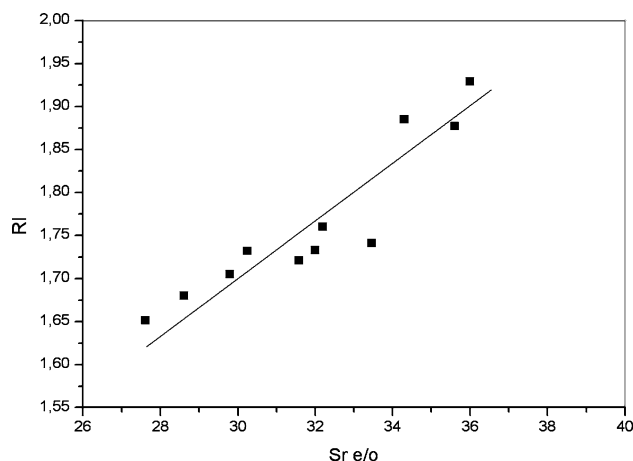


Fig. 9 Glass refractive index (RI) as a function of Sr content in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system

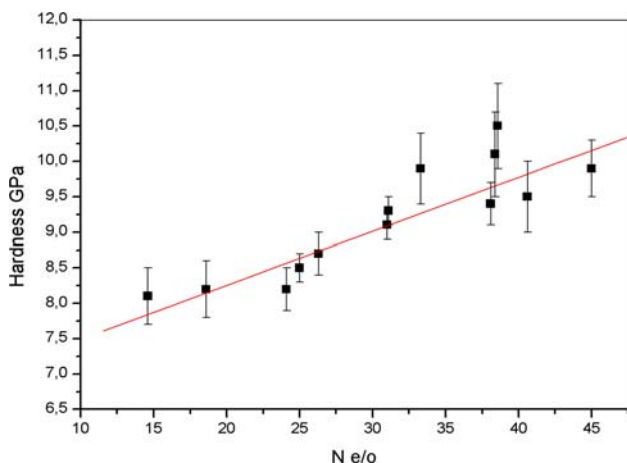


Fig. 8 Glass microhardness as a function of N content in the SrO–SiO₂–Sr₃N₂–Si₃N₄ system

positive dependence on N content, while a dependence on Sr content cannot be established from the data. The microhardness as a function of N content is shown in Fig. 8. The increase in microhardness with N content implies that the incorporation of nitrogen into the glass network strengthens the glass structure. As for other properties, this has been attributed to the presence of three-coordinated nitrogen. However, an increase in hardness with nitrogen content was observed also for high-nitrogen content La–Si–O–N [27] glasses with high X:Si ratios, thus implying very fragmented networks, suggesting that there might be other causes for the observed increase in hardness.

Refractive index

The refractive index at 640 nm varies from 1.65 to 1.93. Regression analysis yielded $n = 0.88(5) + 0.022(2) \cdot [Sr] + 0.006(1) \cdot [N]$. The refractive index is thus found to

increase with both Sr, predominantly, and N content. The refractive index as a function of Sr content is shown in Fig. 9.

Comparison with other alkaline earth Si–O–N and alkaline earth sialon systems

Fair comparisons of the results for the present Sr–Si–O–N glasses with those for other oxynitride silicate glasses are difficult due to differing compositions and synthesis techniques. The densities of the Sr glasses are higher than those for Ca–Si–O–N [2] glasses with similar compositions. Pastuzak [30] compared densities for M–Si–Al–O–N glasses with M = Mg, Ca, and Ba and verified that the density increases with increasing atomic number of M. At a constant N content of 6 e/o, densities of 2.70, 2.75, and 3.68 g/cm³ were found for Mg, Ca, and Ba glasses, respectively. A comparison of results of Homeny [20] for Mg-sialon glasses and Sakka [23] of Ca-sialon glasses similarly confirms that the Ca-sialon glasses have higher densities than Mg-sialon glasses.

The Sr glasses show higher T_g values than Mg-sialon [3, 4], Ca-sialon [3, 4], and Ca–Si–O–N glasses [2], but smaller than Ba-sialon [10] glasses. We find no reports in the literature of glass transition temperatures for Mg–Si–O–N and Ba–Si–O–N glasses.

The hardness value of the Sr glass no. 1, with a low nitrogen content, is slightly smaller than those reported for Mg-sialon [20] and Ca-sialon glasses [23], which have similar hardness values of 8.4 and 8.5 GPa, respectively, at a nitrogen content of 5 at%. Ba-sialon glasses show lower values of hardness [10], i.e. 6 GPa at 8 at% of nitrogen. No reports are found for hardness values for M–Si–O–N (M = Mg, Ca, Ba) glasses. These results accord with those for rare-earth doped oxynitride glasses [28], for which the hardness increases with increasing cation field strength.

The Sr glasses exhibit slightly higher values of refractive index in comparison with Mg and Ca-sialon glasses [4]; 1.64 and 1.69, respectively, at 18 e/o nitrogen. No values of refractive index are reported for the Ba–Si–O–N and Ba-sialon glasses.

Conclusions

A relatively large glass-forming region exists in the Sr–Si–O–N system. Glass formation is believed to be enhanced by the use of Sr in its metallic form as a modifier. The obtained glasses are homogenous with large amounts of Sr, 36 e/o, and N, 45 e/o. A high concentration of Sr in the glass network enhances reaction kinetics and lowers the glass melting temperature. According to elemental analyses, losses of Sr, Si, and N accompany formation of the glasses during the heat treatment. Sr–Si–O–N glasses are translucent gray to opaque black and elemental Si and Sr silicides are assumed to be responsible for the colors. The presence of silicide particles in the glass matrix is unwanted, as it is assumed to be responsible for degenerated glass properties. The results show that addition of nitrogen leads to a systematic increase in glass density, transition temperature, hardness, and refractive index. The strontium content also affects glass properties, in particular density and refractive index, but to a smaller degree.

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