Devitrified phases in a heat-treated $92SiO_2-8Al_2O_3$ glass

Refractory glass-ceramics based on the crystalline-phase mullite are known to form by devitrification of aluminosilicate glasses [1]. One of the limitations on the usefulness of mullite glass-ceramics is the formation of the undesirable cristobalite phase in heat-treated glasses.

In this communication we describe some of the results of phases developed during devitrification, by heat treatment, of a 92 mol% SiO_2–8 mol% Al_2O_3 glass. The glass was prepared by melting an appropriately mixed batch in molybdenum crucibles (2.5 cm diameter x 3.5 cm height) at a temperature of $\approx 1975^\circ$ C for ≈ 2 h and cooling in flowing helium. X-ray amorphous glasses were obtained by this procedure as described previously [2].

Several samples of the glass were heat treated at temperatures ranging from ≈ 1020 to $\approx 1200^\circ$ C for 1 to 76 h to cause devitrification. All heat treatments were conducted in a Globar resistance furnace in air. X-ray diffraction was used to characterize the phases obtained in the devitrified glass samples. Table I summarizes the results of the phases formed by various heat treatments. Lengthy heat treatment (76 h) at a temperature of 1020° C and relatively short (1 to 5 h) heat treatments at 1100° C resulted in the formation of mullite as the only devitrified phase. Heat treatment at 1100° C for 10 and 24 h resulted in development of small amounts of cristobalite in addition to mullite. At 1200° C, cristobalite was observed to be the major crystalline phase in the devitrified glass. The onset of opacity with cristobalite formation was also observed in the 1200° C samples.

In addition to the formation of phases, microscopic observations also indicated negligible crystal growth of mullite during the heat treatments. Thus while mullite nucleates readily at temperatures just above $\approx 1000^\circ$ C extensive growth was not observed even after 76 h at 1020° C. This lack of secondary grain growth at 1020° C in mullite glass-ceramics is significant, since secondary grain growth is known to degrade the physical properties of glass-ceramics [3] and is, therefore, an important stability criterion. In summary, our results suggest that in order to form mullite glass-ceramics without cristobalite precipitation or secondary mullite grain growth, lengthy heat treatments at temperatures $> 1000^\circ$ C but $< 1100^\circ$ C should be most suitable.

References

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TABLE I

Temperature of heat treatment (° C)	Time (h)	Major phase	Minor phase
1020	76	Mullite	None
1100	1	Mullite	None
1100	5	Mullite	None
1100	10	Mullite	Cristobalite
1100	24	Mullite	Cristobalite
1200	27	Cristobalite	Mullite
1200	55	Cristobalite	Mullite

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