	Experiment title: **Effect of fictive temperature on the nanostructure in high purity silica glass**	**Experiment number**:02-01-78
Beamline: BM02	**Date of experiment**: from: 08/11/00 to:11/11/00	**Date of report**: 28/02/01
Shifts: **5/2000**	**Local contact(s)**: J.P.Simon	*Received at ESRF:*

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REPORT

Low OH content silica glass are of great interest nowadays because their application in optical fibers. Silica samples having fictive temperatures ranging in 1000-1500°C have been prepared by long annealing to allow structural relaxation followed by a quenching which freeze the new structural order.

Those samples have already been investigated by Raman and Rayleigh scattering(1).The fictive temperature of the samples has been estimated using Raman scattering and Infra-Red absorption methods.

The small angle scattering measurement were performed on D2AM beamline at the ESRF (Grenoble) with an energy of E=15keV and 200s accumulation time.

In situ temperature measurement have been performed in a molybdenum furnace, using heating time of 5mn/100°C. Technical improvements have been achieved on the furnace and further modifications are planned for next experiments.

The spectrum were recorded so as to have a great accuracy on smaller angles, with a q range (0.0019-1.03 A^{-1}) for room temperature measurements and (0.0019-0.76 A^{-1})for measurements in furnace. The curve have been fitted to the usual empirical law $I(q)=I_0 \exp(bq^2)$, and I_0 is extracted; it represents the level of density fluctuations (Porai Koshit)

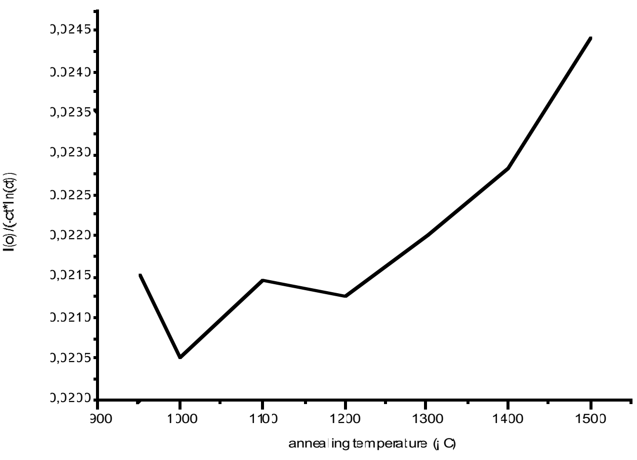

Fig. 1

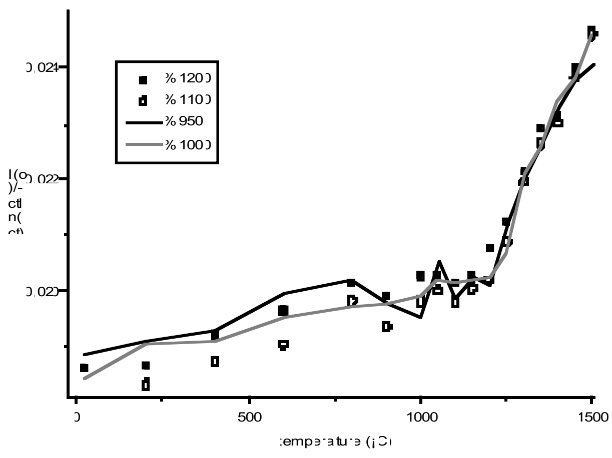

Fig. 2

Fig (1) shows room temperature measurement of I(q=o) versus annealing temperature. The density fluctuations measured here include frozen electronic density fluctuations and adiabatic electronic fluctuations. It appears obviously that electronic density fluctuations increase with annealing temperature. Light scattering measurements of Landau Placzec ratio (IRayleigh / I Brillouin) on the same sample reported in (1) lead to similar results.

Sample heat treated at 950°C has a particular behaviour, the fictive temperature of this sample has been estimated to be 1200 +/- 60°C. The electronic density fluctuation level revealed by SAXS raises at the level of the samples annealed at 1100 and 1200°C. This sample annealed during 10days did not relax completely from the point of view of the structures probed by spectroscopic methods (1) and also from the point of view of electronic density fluctuations.

Temperature dependence of SAXS intensity is common to many glasses : from room temperature to Tg, the intensity is slightly increasing, above Tg, a change in the slope appears, corresponding to the supercooled region .

Samples annealed at different fictive temperature under Tg (fig 2) present slightly different level of density fluctuations under Tg, the differences are clear for the sample annealed at 1200°C for which the SAXS intensity is bigger than the sample annealed at 1100°C. For the two other samples, the intensity oscillate in between the two other curves.

The SAXS intensity versus temperature for the high fictive temperature glasses obtained after quenching (fig. 3) is different: the density fluctuations level rises with fictive temperature, each curve having the same slope, from room temperature to 1050¡C and then the intensity falls down because of structural relaxation to a minimum value of the scattering intensity corresponding to the glass transition temperature. Finally, the last part of the curve corresponds to supercooled liquid in which the density fluctuations increases quickly with the temperature.

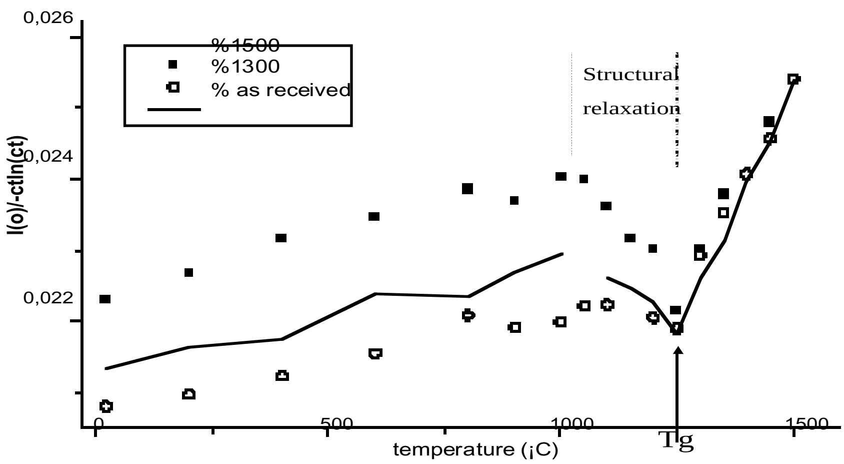

Fig. 3

CONCLUSION:

SAXS experiments on SiO2 glasses with different fictive temperature are very successful and complement the light scattering experiments. The X-Ray scattering evolves with temperature with two different slopes above and below Tg. For high fictive temperature samples *in situ* temperature measurements demonstrate, at our knowledge for the first time, a relaxation from 1050°C and a scattering minimum in the region of Tg. These results suggest ways to reduce light scattering of optical fibers and new promising experiments on Ge doped silica are planned

These results have already been presented in a conference (3) and will be soon discussed in an international Wokshop and published (2)

REFERENCES:

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