	Experiment title: Optical transparency of high fictive pressure glasses: in situ high temperature SAXS experiments	**Experiment number**: HD425
Beamline: BM02	**Date of experiment**: from: 19.04.2010 to: 23.04.2010	**Date of report**: 15.09.2010
Shifts: 9	**Local contact(s)**: Jean Paul Simon	*Received at ESRF:*

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Report:

The aim of the experiment was to demonstrate that glasses elaborated under high pressure (high fictive pressure glasses) have actually lower fluctuations which are the origin of improved optical properties. Additionally the evolution of these fluctuations evolve temperature should be determined.

During the allocated beam time at the ESRF we investigated samples with two mainly different composition Na_2O-B_2O_3-SiO_2 (NBS) and mixed anionic sulfo-phosphate glasses (SP) as well as pure SiO_2 glass. These samples were compressed above T_g at pressures up to 500 MPa and quenched under pressure to room temperature.

We caried out two different type of experiments: The first three shifts were used for ambient SAXS measurements. During the beam shutdown we installed a molybdenum high temperature furnace which was provided by the beamline. For the further six shifts we followed during heating as well as during isothermal treatment in situ SAXS. We took at all over 3000 spectra.

We assume that the glass structure correlating with the different fictive pressures was frozen in. The resulting glasses then exhibit the same composition, the same short range structure but different mid and long range structure i.e. in a different position in the energy landscape of the glasses and therefore also different optical properties. High pressure densified glasses are expected to have a higher optical transparency than room pressure elaborated glasses what is ascribed to a decrease of the density fluctuations. The low q limit of the scattered intensity extrapolated at q=0 in the flat region is related to the amplitude of such density and concentration fluctuations. With the ambient measurements carried out we proved an increase in homogeneity and therefore decrease in fluctuations for the higher compressed samples as shown in Fig. 1.

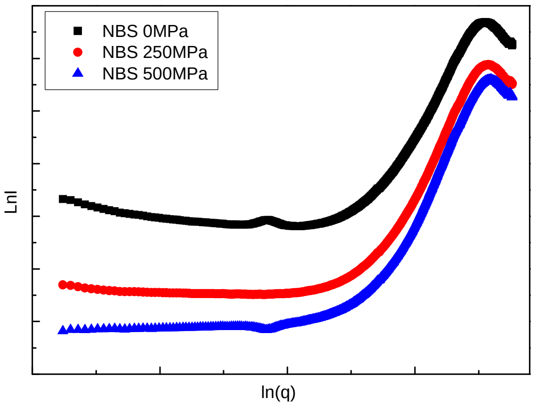

Fig.1 Ambient SAXS spectra of three different compressed NBS glasses

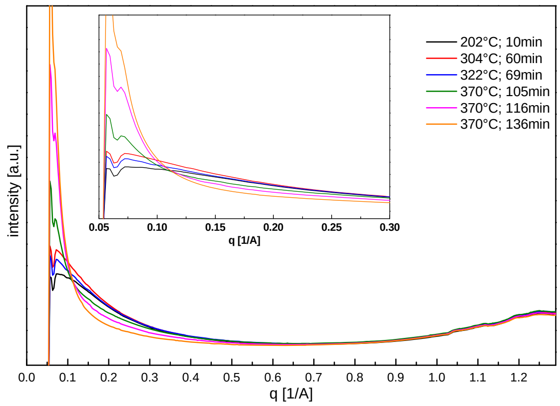

Fig.2 SAXS spectra on SP-glass in the high T molybdenum furnace heated with 2K/min

To understand kinetics and thermodynamic aspects of the glass transition and structural relaxation phenomena both path, temperature and pressure should be considered. Our experiments on different compressed samples during heating and isothermal treatment conform to this. We are right now evaluating the data of the NBS and SP-glasses. For compressed SiO_2-glass we can readily demonstrate the conversion from a very homogeneous structure to the regular quenched glass. Surprisingly it was observed that this structural relaxation during it´s heating from 400°C to 1450°C starts at about 700°C which is a very low temperature compared to the regular quartz glass T_g.

As shown in Fig. 2 for the SP-glasses we observed at least one peak evolving at very low q with temperature and time leading to the conclusion of the evolution of long ranged inhomogeneity. Unfortunately the spectra are cutted by the beam stop in that region. Therefore it would be highly appreciated to repeat and go on with these experiments under optimized conditions to fully observe lower q-ranges. Even because sulphophosphate glasses are practically unkonown in literature.

Summing up, the experiments done during HD-425 session confirmed the expected results and opened new perspectives. The beam focus and properties were well adapted to our samples. The time could be fully used with the installation of the furnace during beam shutdown. The furnace performance was highly satisfying with a full covering of temperature range and accuracy. More beamtime to investigate the evolution of long range sized inhomogeneities in sulphophosphate glasses is needed.

These results will be reported in at least two paper which will be submitted to the Journal of Applied Physics.