ESRF	SAXS measurement of the glass transition at extreme temperatures in Al2O3-SiO2 glasses	**Experiment number:** 02-01-711
Beamline: BM02	**Date of experiment:** from: 15-11-2006 to: 18-11-2006	**Date of report:** 22/10/2007
Shifts: 9	**Local contact(s):** Jean-paul Simon	*Received at ESRF:*

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Report:

The aim of this proposal was to use temperature-scanning small angle X-ray scattering measurements as a tool to fully resolve and characterize the glass of binary aluminosilicate glasses with composition $(Al_2O_3)_x(SiO_2)_{1-x}$. Those glasses are of significant technological importance in the areas of high-temperature ceramics and rare earth doped fiber optic. To our knowledge there exist no direct measurements of the glass transition in vitreous aluminosilicate and this method has given us excellent results for vitreous silica.

The samples were home-made binary glasses prepared by ball-milling of an initial silica glass, the powder was sieved and mixed with alumina powder, then the material was sintered in air at 1100° C, followed by repeated melting in an oxygen-acetylen flamme. Temperature scanning SAXS measurements were taken on 10 different compositions ranging from 14 ppm Al_2O_3 to 10 % Al_2O_3. Each sample was heated at least twice from the as-prepared state at 300 K to between 1800 K and 1900 K. All heating and cooling scans were carried out at a rate of 40 K/min. Homogeneity of the sample was checked by measuring the SAXS signal in several positions at room temperature. The amorphous character of the sample was also checked. The glass transition temperature of aluminosilicate glasses was found to exhibit a non monotoneous variation with composition, showing a maximum around 100 ppm Al_2O_3. However, the overall variation is rather weak, from 1400 K with no alumina to 1480 for 100 ppm, and 1400 K for 5000 ppm.

We also performed additionnal measurements related to our previous proposal about SAXS measurements in silica glass. Indeed, an unexpected aging was observed during the first heating ramp of our previous measurements. The silica sample was then mounted into the furnace using a high temperature cement; new measurement were performed without the cement. The result showed that aging is much limited in that case [Temperature Scanning Small Angle X-Ray Scattering Measurements of Structural Relaxation in Type-III Vitreous Silica R. Brning, C. Levelut, R. Le Parc, A. Faivre, L. Semple, M. Vallee, J.-P. Simon and J.-L. Hazemann Journal of Applied Physics, to be published (2007).]. We concluded that atomic diffusion of minor elements from the cement to the scattering volume could be a possible reason for the apparent aging of the sample.