	Experiment title: ASAXS in-situ study of concentration fluctuations induced by germanium dopant in optical fiber glass	**Experiment number:** 02-01-123
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Report: ASAXS in-situ study of concentration fluctuations induced by germanium dopant in optical fiber glass

This experiment is the third part of a study on optical fibers preforms. Silica glass is used for optical fibers elaboration, and the fiber core where the signal propagates is often made of germanium doped silica glass. Density fluctuations are frozen in the glass at a temperature called « fictive temperature » and give rise to Rayleigh scattering inducing signal losses in the glass. As density fluctuations also give rise to a signal in small angles scattering, it is possible to extrapolate the frozen-in density fluctuations magnitude at zero angle : I(q=0). In the first part of the study we measured I(q=0) versus fictive temperature in silica glass, then in a second part, we measured I(q=0) in germanium weekly doped silica glasses.

In germanium doped silica glass, concentration fluctuations add to density fluctuations, and germanium clustering in hydrogen atmosphere has also been oberved. In order to know if the samples are inhomogeneous, an anomalous small angle scattering experiment has been done close to the edge of germanium.

The first step of this experiment has consisted in measuring absorption edge of Germanium, Germanium oxyde, and germanium doped silica having three increasing concentrations c1, c2, c3. This measurement shows an increasing absorption edge when doping increases.

Then SAXS signal has been measured for the three doped samples at 10.8, 11, 11.04, 11.075, 11.09keV. Room temperature measurements have shown an increase of signal as the energy comes closer to the edge, for the three compositions. This increasing signal give absolutely homothetic curves and is due to sample fluorescence. Then as no anomalous feature can be seen, germanium doped silica as far as c3 doping is homogeneous in composition.

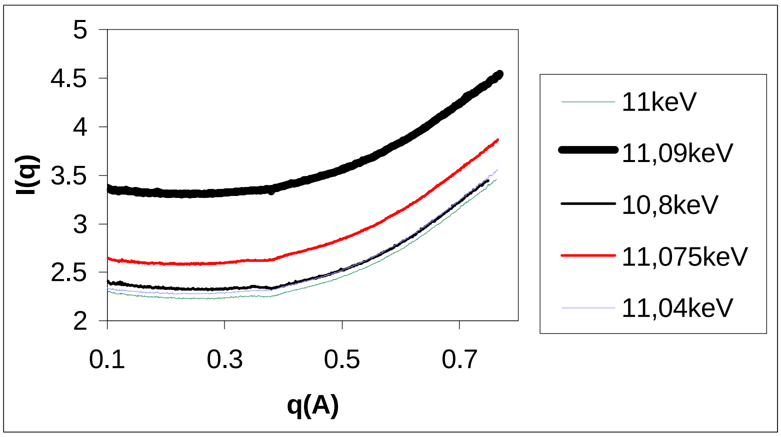

fig.1 : c3doped glass Signal for different incident beam energies.

In-situ temperature SAXS experiment has also been performed in this anomalous setting. No anomalous effect can be demonstrated as the temperature increases. Extrapolation of I(q=0) versus heating temperature for the samples c1, c2, c3, has been done. At first, I(q=0) increases as germanium concentration increases. Then the low and high temperature asymptotes slopes decrease in accordance with material « softening »; the junction between the two asymptote happens at a temperature corresponding to the fictive temperature, it is then possible to determine decreasing fictive temperatures 1300, 1000 and 900°C respectively for samples c1, c2, c3.

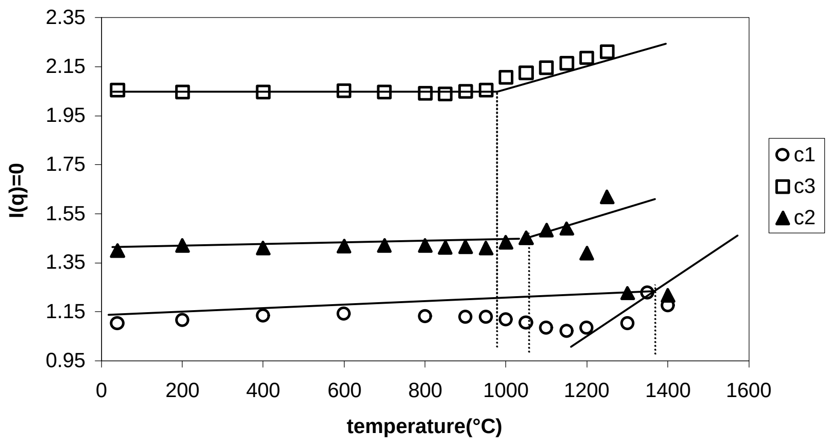

fig.2 : Temperature dependance of the extrapolated signal I(q=0) for different germanium concentrations. The intersection of the two asymptotes give an estimation of the fitive temperature : respectively 1300, 1000 and 900°C for the samples c1, c2, c3.

Thus, when germanium concentration increases, fictive temperature and density fluctuations decrease but in the same time concentrations fluctuations increase strongerly leading to a higher zero anlge SAXS signal.

The results of this experiment are very important for this study, and also give rise to new experimental projects : c3 concentration is lower than 1/4, then it would be interesting to study anomalous signal over this concentration to check that Germanium speading in the samples keeps homogeneous.