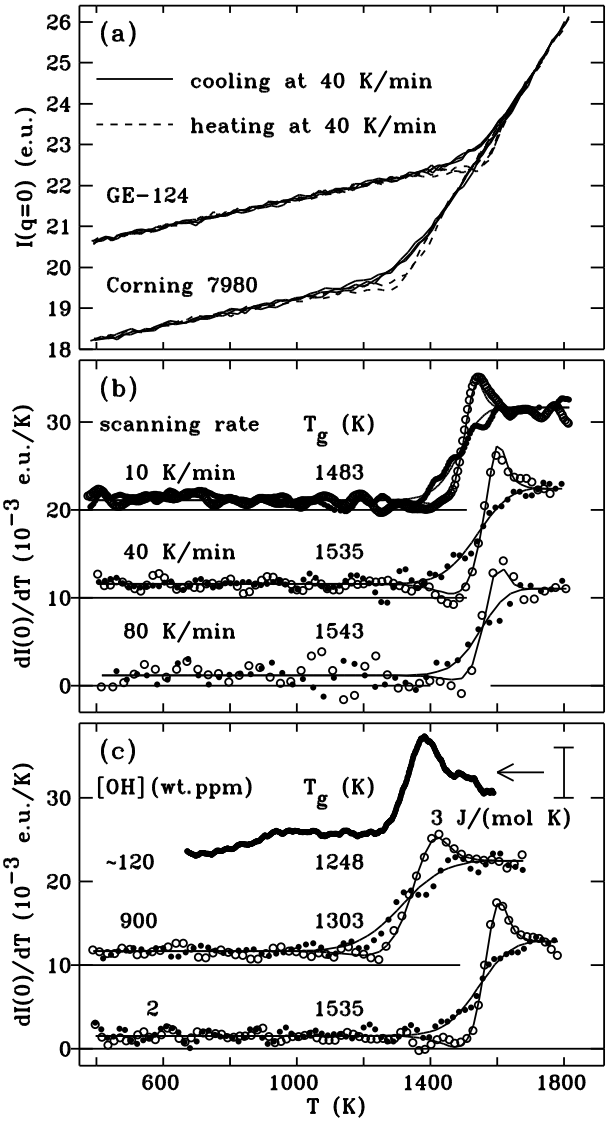
ESRF	Temperature scanning SAXS experiment in amorphous silica	**number:** 02-01-638
Beamline: BM02	**Date of experiment:** from: 15/07/2004 to: 19/07/2004	**Date of report:** 3/03/2005
Shifts: 12	**Local contact(s):** J.P. Simon/F. Bley	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
C. Levelut*, A. Faivre*, R. Leparc*, LCVN, Montpellier
R. Brüning*, Sackville, Canada
J.-L. Hazemann*, Lab. de Cristallographie, Grenoble

Report:

The aim of this proposal was to carry out temperature scanning SAXS measurements in silica samples, in order to study the glass transition and the relaxation state in silica. We wanted to compare the results to those obtained by scanning calorimetry which is rather difficult to perfom in silica due to its high glass transition temperature. We performed measurements on two kind of silica: GE-124 with 2ppm OH (6 samples) and Corning 7980 with 900 pmm OH (3 samples) (fig 1a). For each sample the cool-heat cycle was repeated at least three times to ensure a good signal to noise ratio. Some of the measurements have to be thrown away because it is important that no interruption (due to refilling or any experimental problem) occurs in the critical part of the temperature ramp because the samples would be modified due to relaxation phenomenon around the glass transition temperature. Thus the 4 allocated days were just enough to obtain high quality results at 3 different temperature scanning rates (10, 40 and 80 K/min) (fig 1b). The derivative of the SAXS intensity extrapolated to a scattering vector q=0 directly compare to scanning calorimetry results (fig 1c). The SAXS data are clearly superior both in terms of the stability and linearity of the background as well as in providing data upon heating as well as cooling. They allow a detailed analysis and provide tighter constraints on the fit with a Adam Gibbs Fulcher model. New results about the glass transition in silica were obtained, showing that the transition is broader in the silica with higher hydroxyl content. The results are currently accepted for publication in Europhysics Letters (abstract attached).

Figure 1



Characterization of the glass transition in vitreous silica by temperature scanning small-angle X-ray scattering

R. Brüning[1](*) and C. Levelut[2] and A. Faivre[2] and R. LeParc[2] and J.-P. Simon[3] and F. Bley[3] and J.-L. Hazemann[4]

[1] *Physics Department, Mount Allison University, 67 York Street, Sackville (NB), Canada E4L 1E6*

[2] *Laboratoire des Verres, UMR 5587 CNRS-Université Montpellier II, cc 69, 34095 Montpellier Cedex, France*

[3] *LTPCM-ENSEEG-INPG, UMR-CNRS 5614, B.P. 75, 38042 St. Martin d'Hères Cedex, France*

[4] *Laboratoire de Cristallographie, UPR 5031, CNRS, B.P. 166, 38042 Grenoble, France*

PACS. 64.70.Pf – Glass transition.
PACS. 42.70.Ce – Glasses, quartz.
PACS. 61.10.Eq – X-ray scattering (including small-angle scattering).

Abstract. – The temperature dependence of the x-ray scattering in the region below the first sharp diffraction peak was measured for silica glasses with low and high OH content (GE-124 and Corning 7980). Data were obtained upon scanning the temperature at 10, 40 and 80 K/min between 400 K and 1820 K. The measurements resolve, for the first time, the hysteresis between heating and cooling through the glass transition for silica glass, and the data have a better signal to noise ratio than previous light scattering and differential thermal analysis data. For the glass with the higher hydroxyl concentration the glass transition is broader and at a lower temperature. Fits of the data to the Adam-Gibbs-Fulcher equation provide updated kinetic parameters for this very strong glass. The temperature derivative of the observed X-ray scattering matches that of light scattering to within 14%.