



Mesures par SAXS de l'effet de la diffusion de l'eau dans la silice vitreuse

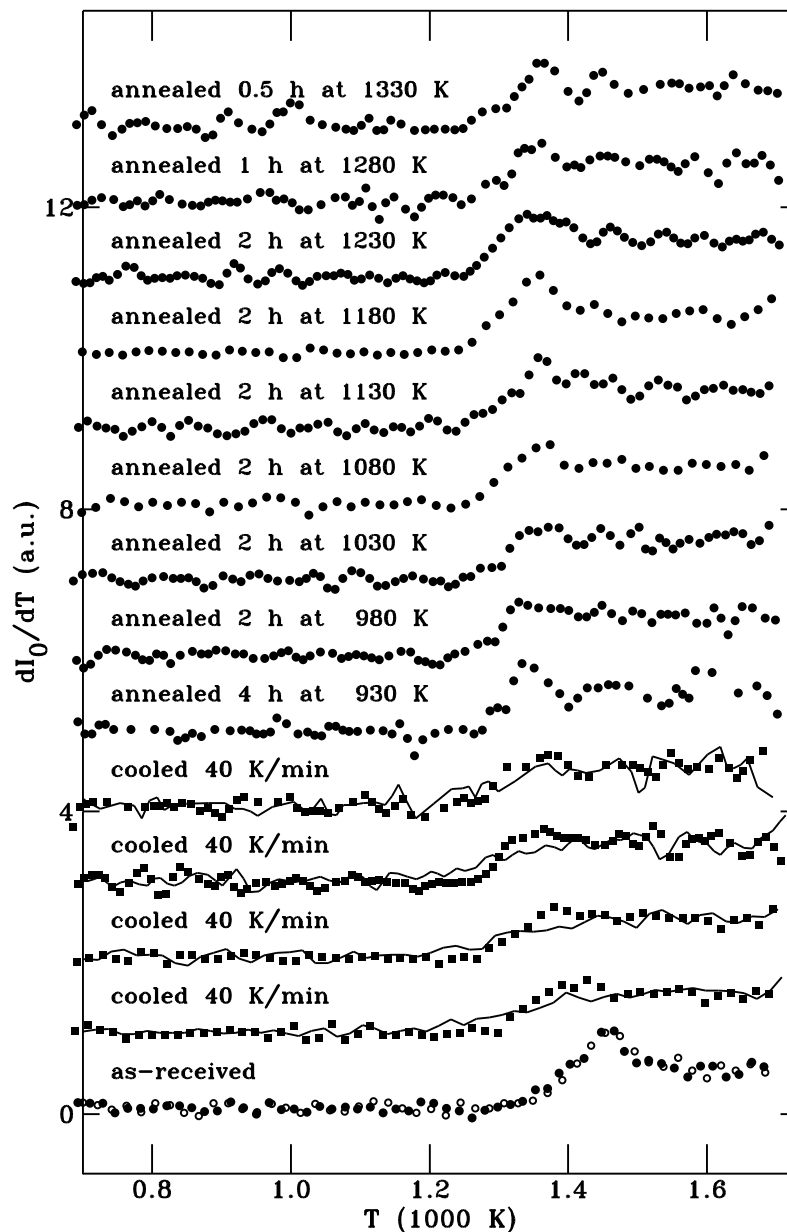
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Report:

We have recently performed SAXS measurements of the hysteresis associated with the glass transition in two different samples of vitreous silica [1,2]. Those measurements showed that SAXS signal and the enthalpy of a glass depend on the state of the glass in a similar way. In silica containing 120ppm OH, an apparent increase of enthalpy was observed before the usual lowering characteristic of the glass transition [3]. This feature is related to the water diffusion process. The experiment we report here concentrate on one silica sample with a higher OH content. We want to evidence the same phenomenon using SAXS. Measurements were performed on Corning 7960 silica samples, containing 900ppm OH. The sample was annealed at different temperature corresponding within to the temperature range where the enthalpy decreases was observed. Each measurement consists of three decreasing temperatures ramps (from 1723 K down to 373 K) and then increase up to 1773 K in order to prepare the sample in a "standard" state. Then the sample is cooled down to the heat-treatment temperature T_a ; after a waiting time ranging from 15 to 120 min it is cooled down to 373 K and the SAXS signal is measured upon heating.

Annealing were performed at $T_a=1380$ K for 15 min, 1330 K for 30 min, 1280 K for one hour, 1230 K, 1180 K, 1130 K, 1080 K, 1030 K and 980 K for two hours, 930 K for four hours. We have shown in our previous experiment that calorimetry signal can be compared to the derivative of the SAXS signal [1,2]. A very weak decrease of the derivatived SAXS signal (roughly analoguous to an enthalpie decrease) can be observed after the lowest temperature heat treatment (657 °C). However this decrease is very weak and cannot be confirmed by heat-treatment are others temperatures.



Derivative of the SAXS intensity extrapolated to zero scattering vector, $dI(0)/dT$, versus temperature T . The first (lower curve) is measured on the as received sample, the following four are measured upon heating after cooling down at 40K/min. The other curves are taken upon heating after the heat treatment indicated on the left of the curves. Curves have been shifted up for clarity.

- [1] R. Brüning, C. Levelut, A. Faivre, R. LeParc, J.-P. Simon, F. Bley and J.L. Hazemann, Characterization of the glass transition in vitreous silica by temperature scanning small-angle X-ray scattering, *Europhys. Lett.* 70 (2005) 211-217.
- [2] R. Brüning, C. Levelut, A. Faivre, R. LeParc, J.-P. Simon, F. Bley and J.L. Hazemann, Characterization of the glass transition in vitreous silica by temperature scanning SAXS, *ESRF Highlights* 2005 (2006) 24-25.
- [3] R. Brüning, on the glass transition in vitreous silica by differential thermal analysis measurements, *J. Non-Crystalline Solids* 330 (2003) 13-22.