



	<b>Experiment title:</b> LONG-RANGE LENGTH SCALE STRUCTURAL STUDY OF SINTERED SILICA AEROGELS	<b>Experiment number:</b> SC-539
<b>Beamline:</b> ID2	<b>Date of experiment:</b> from: 99/06/18                      to: 99/06/20	<b>Date of report:</b> August 1 <sup>st</sup> 99
<b>Shifts:</b> 9	<b>Local contact(s):</b> <b>Peter BÖSECKE – T. NARAYANAN</b>	<i>Received at ESRF:</i>
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### Report :

The aim of our experiments performed at ESRF last mid-June was to study in real time the changes in the morphology of silica aerogels (highly porous materials issued from sol-gel processes) during the densification process (mainly by sintering). We were essentially interested by the domain of long ranges of length scale (above  $10^2$  Å) where only few and contradictory information is available. That is why we planned to use the Bonse-Hart optics on line ID2 for studying the kinetics of structural evolution during the sintering especially in the final stages of densification.

Unfortunately a major experimental problem occurred (the needed furnace broke down at the early beginning of our shifts) making our original experimental project impossible.

Instead we focus our attention on sets of samples previously prepared in our laboratory: we mainly studied partially densified aerogels. Densification was performed by heat treatment (sintering) or by combination of isostatic compaction followed by sintering. Typical spectra are plotted in Figure 1 in normalized intensities: the full line corresponds to a sintered aerogel sample with a final density of  $d = 1.05$ . For comparison the “background” spectrum

means without any sample - is showed in full squares. Then this spurious signal was systematically subtracted from the original one in order to get the pure diffusion spectrum ( $\Delta I$ ) of the sample.

In Figure 2 are plotted the results obtained for partially densified aerogels. The three upper curves correspond to sintered samples with increasing value of density ( $d$ ). One remarkable point is the increasing in intensity for decreasing  $q$  values (below few  $10^{-3} \text{ \AA}^{-1}$ ) according to a power law the exponent of which is denoted by  $s$ . It is worth noticing that the kink between this linear behavior (in our log-log plot) corresponds to a  $q$  value ( $q_K$ ) which increases with the density. These first results may put the light on important features on the morphology of aerogels at very long range of length. Indeed our observation may be interpreted as the signature of a *macroporosity* the typical length of which (around few hundreds of Angström) decreases during the sintering processes. Another confirmation of this hypothesis is visible in Figure 2 (full square): For an aerogel compressed in a first step and then sintered,  $q_K$  has a higher value than that for analogous bulk density of an only sintered aerogel. We had previously shown that isostatic compression mainly affects the broadest pores of aerogels. These pores decrease in size as the pressure increases. Obviously the sintering causes too a decrease in the pore size. This feature confirms that the observed regime change must be related to macropores' size.

Other sets of samples were also studied and spectra analyses with quantitative analysis are now in progress.

Figure 1

Figure 2 (the curves are arbitrary shifted along the Y axis)