ESRF	**Experiment title:** The Dynamics of Titania-Nanoparticle Gels	**Experiment number**: SC-2689
Beamline: ID10A	**Date of experiment**: from: 06/05/2009 to: 12/05/2009	**Date of report**: 05/09/2009
Shifts: 18	**Local contact(s)**: Dr Yuriy Chushkin	*Received at ESRF:*

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Report:

With this experiment we aimed to study the dynamics of dense colloidal suspensions of titanium dioxide (titania) as they were heated and dried. The heating protocols adopted were chosen to match those most often used to prepare electrodes for dye sensitised solar cells in the literature [Barbe97]. Through this research we hope to inform electrode preparation techniques and our own research into novel solar cells.

We studied two different titania pastes. One, “Ti-Nanoxide T”, is a commercial paste for the construction of solar cell electrodes from Solaronix (www.solaronix.com). The other was produced in the lab from P25 titania available from Evonik (formerly Degussa, www.degussa.com). Quartz capillaries (Hilgenberg) were coated in a thin film of paste and placed into a heating stage. Scattering from the samples was then studied as the films were heated from room temperature to a final temperature between 200 °C and 500 °C at heating rates of 1 °C/min, 2 °C/min and 20 °C/min.

From these data the dynamics of the suspensions can be studied through the intensity-intensity correlation function $g^{(2)}(q)$ and two time correlation function $g^{(2)}(q, \tau)$. Data were analysed using the PyXPCS software provided by Dr Yuriy Chushkin. The availability of this software at the beamline allowed concurrent analysis of the data as they were collected which was very useful for refining experimental protocol.

Samples demonstrated ageing behaviour at room temperature (fig. 1) followed by steadily more rapid decorrelation at higher temperatures (fig. 2), suggesting faster dynamics. At even higher

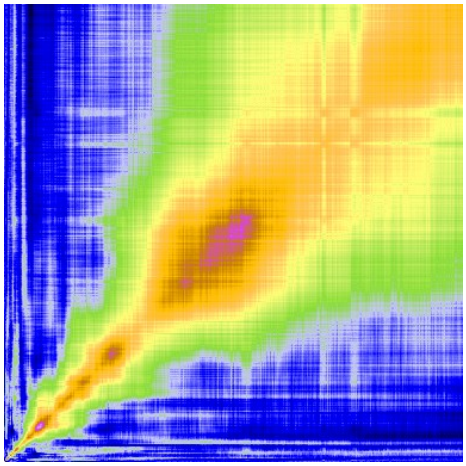

Figure 1 - two time correlation function for a P25 sample at room temperature.

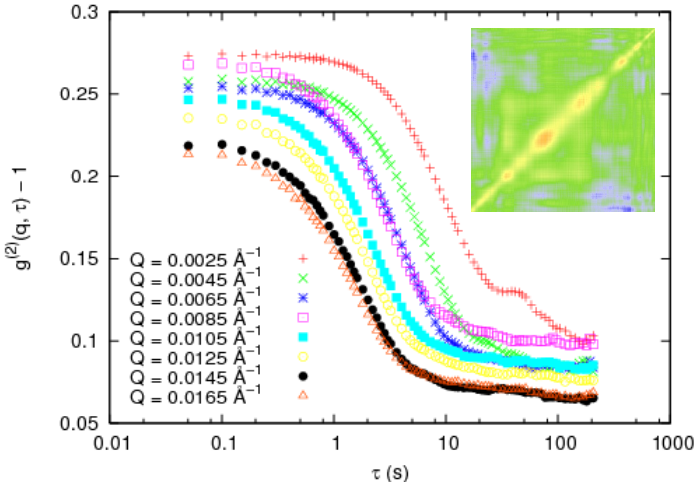

Figure 2 - intensity-intensity correlation function for a Solaronix sample at 100 °C. *(inset)* - two time intensity-intensity correlation function.

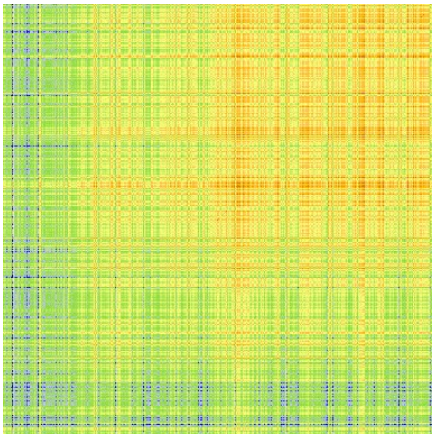

Figure 3 - an essentially static two time correlation function for a Solaronix sample at 125 °C.

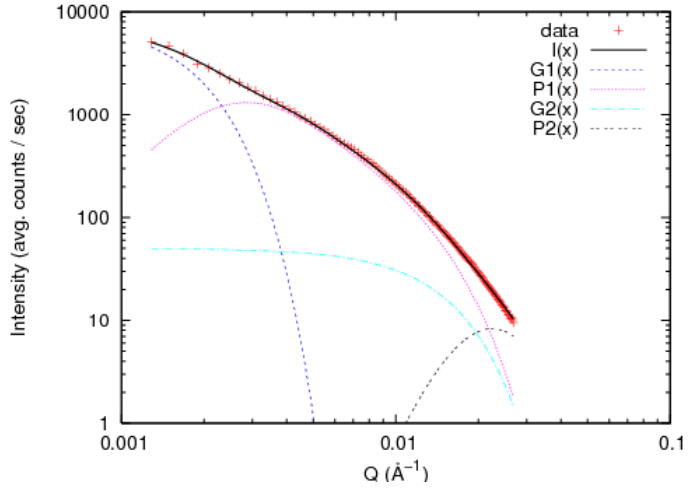

Figure 4 - fitting of preliminary static scattering data for a P25 sample at room temperature.

temperatures long time correlations begin to appear (fig. 3), indicating the onset of drying. Even at room temperature the pastes studied appear to demonstrate non-ergodic behaviour, indicating a degree of gelation. The analysis of this data is ongoing.

It has also been possible to extract static scattering information from the data through radial averaging of the scattering through rings of equal q. This was also performed using PyXPCS. Analysis of the scattering data using the Beaucage universal scattering function [Beaucage95] has indicated that useful data can be obtained (fig. 4) and it is hoped this will form the basis of a future dedicated SAXS study of this system.

We would like to acknowledge the invaluable advice of Yuriy Chushkin during our experiments and his kind provision of the PyXPCS software for our analysis both at the beamline and after the experiment.

References

[Barbe97] Barbé *et al* (1997), *Amer. Ceramic Soc.* **80** (12), 3157-3171.

[Beaucage95] Beaucage (1995), *J. Appl. Crystallogr.* **28** (6), 717-728.