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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** In-situ foaming of silicone based nanocomposites		**Experiment number**: MA-641
Beamline: ID19	**Date of experiment**: from: 03-10-2008	to: 07-10-2008	**Date of report**: 14/04/2009
Shifts: 12	**Local contact(s)**: Dr. L Helfen		*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists): Raquel Verdejo* (Inst. Ciencia y Tecnología de Polímeros, CSIC)			

Report:

Aim and Objectives

The purpose of the experiments was to monitor the evolution of the cellular structure during foaming of polymer matrices by in situ micro-radiography and tomography. Foaming can me divided into three stages which involve the formation of gas bubbles in a liquid system, and the growth and stabilisation of those bubbles as the matrix increases its viscosity and solidifies. In particular, the system under study is foamed by the condensation reaction between silanols (SiOH) on hydroxyl-terminated polydimethylsiloxane and silanes (SiH) on polymethylhydrogensilane in presence of a catalyst with the evolution of hydrogen gas. Hence, gas is produced as a by-product of the reaction, leading to the nucleation and growth of bubbles within a fluid whose rheology evolves from a low viscosity liquid to a viscoelastic gel.

The aim was to study the effect of nanofillers on the morphology and dynamics of polymer foams and to understand the effect of the nanofiller shape by adding two morphologically different carbon-based nanofillers, in particular multiwall carbon nanotubes (CNT) and functionalised graphene sheets (FGS).

Results

The experiments were carried out on beamline ID19 using two resolutions with the effective pixel sizes of 15 μm and 2.8 μm. A full analysis has not yet been completed. Preliminary observations are reported below.

a) Tomography

Tomography experiments were not successful enough to provide 3D data sets of the initial stages of the foaming process. These initial stages were too fast for the detector and rotation stage used and we observed motion artefacts which are preventing the proper reconstruction and analysis of the geometrical evolution of the samples (Figure 1). Although we tried to slowdown the reaction by cooling the reactants prior to mixing in a fridge and by directing cool air to the sample container during the tomography recording, the motion artifacts were still visible.

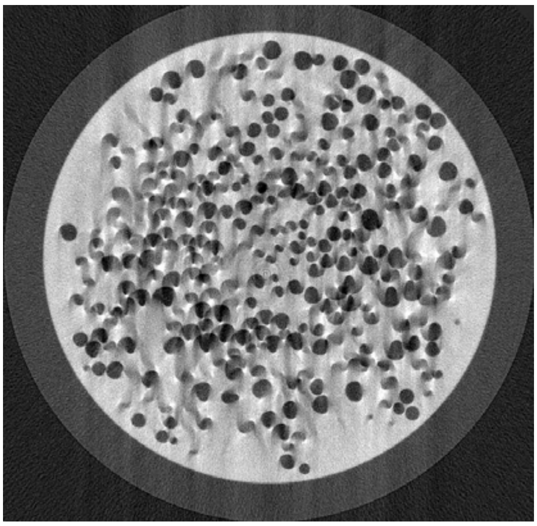

Figure 1: Reconstructed slice from the tomography of the foaming process showing the observed motion artefacts.

Nevertheless, at later stages of the foaming process motion artifacts were no longer visible and provided full 3D reconstruction of the samples (Figure 2). At this later stages, foams evolve through internal processes, such as gas diffusion, drainage and cell coalescence, leading to comparatively little changes in the bubble structure. Initial analysis of the data shows clear differences on cell size, roundnes and connectivity. The full analysis of the generated data is still under way.

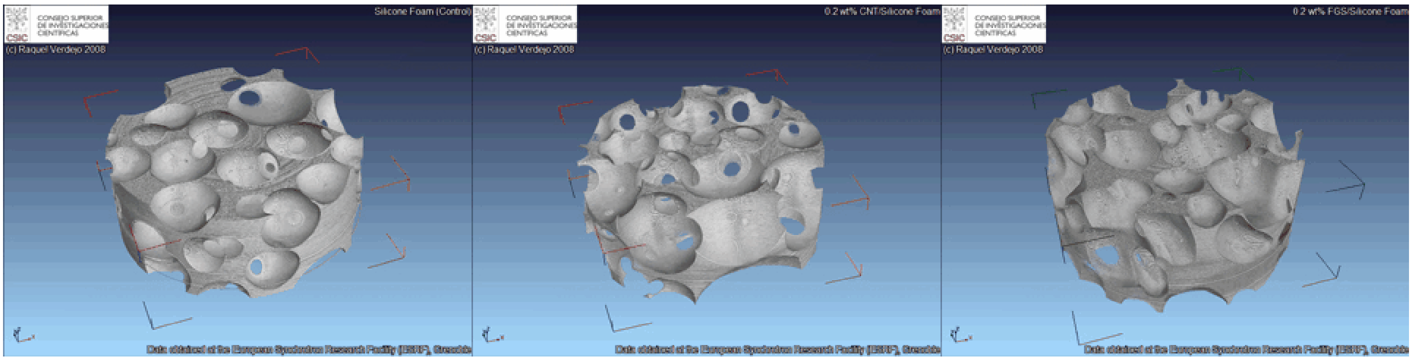

Figure 2: 3D reconstrucion of foam samples after gellation. From left to right: control sample, CNT filled sample and FGS filled sample. Pixel size is 2.8 μm.

b) Radiography

Radiography experiments were successfully applied to the analysis of the foaming process of the samples. Motion artefacts were not visible on radiograph images and provided more detailed information on the foaming dynamics of the system.
Initial inspection of the radiographs shows clear differences on the temporal evolution of the foaming process. Filled samples evolved more slowly than unfilled samples and it is possible to observe the early stages of the foam (Figure 3). Several physical phenomena of foam formation and stabilisation can be observed, from the diffusion of gas from small bubbles to large bubbles (Figure 4) to film ruptures (Figure 5).

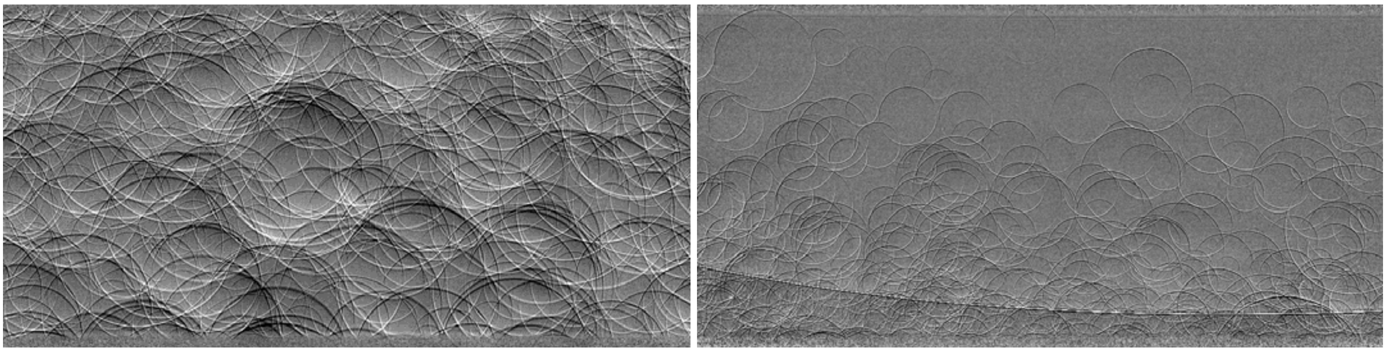
Figure 3. Radiograph taken 1 min after mixing the reactants of control (left) and FGS sample.

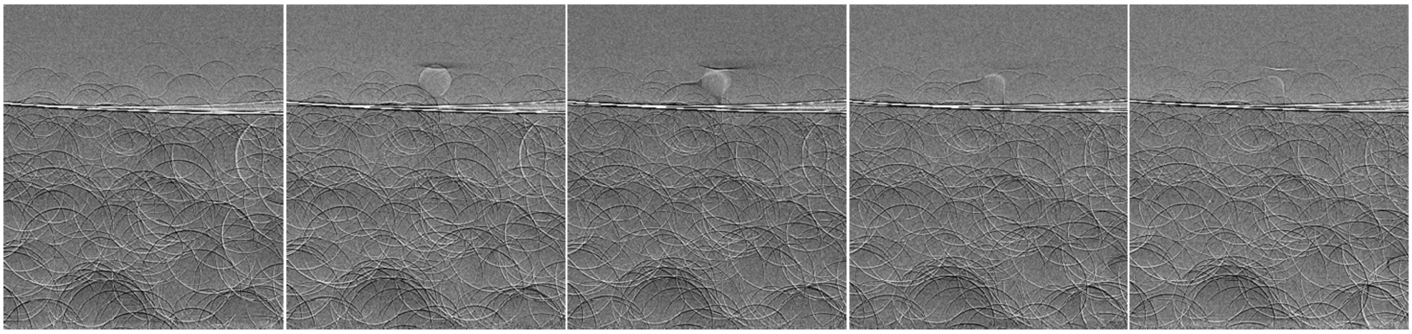
Figure 4. Difference images of succesive frames of the FGS sample showing the diffusion of gas from a small bubble to a large bubble.

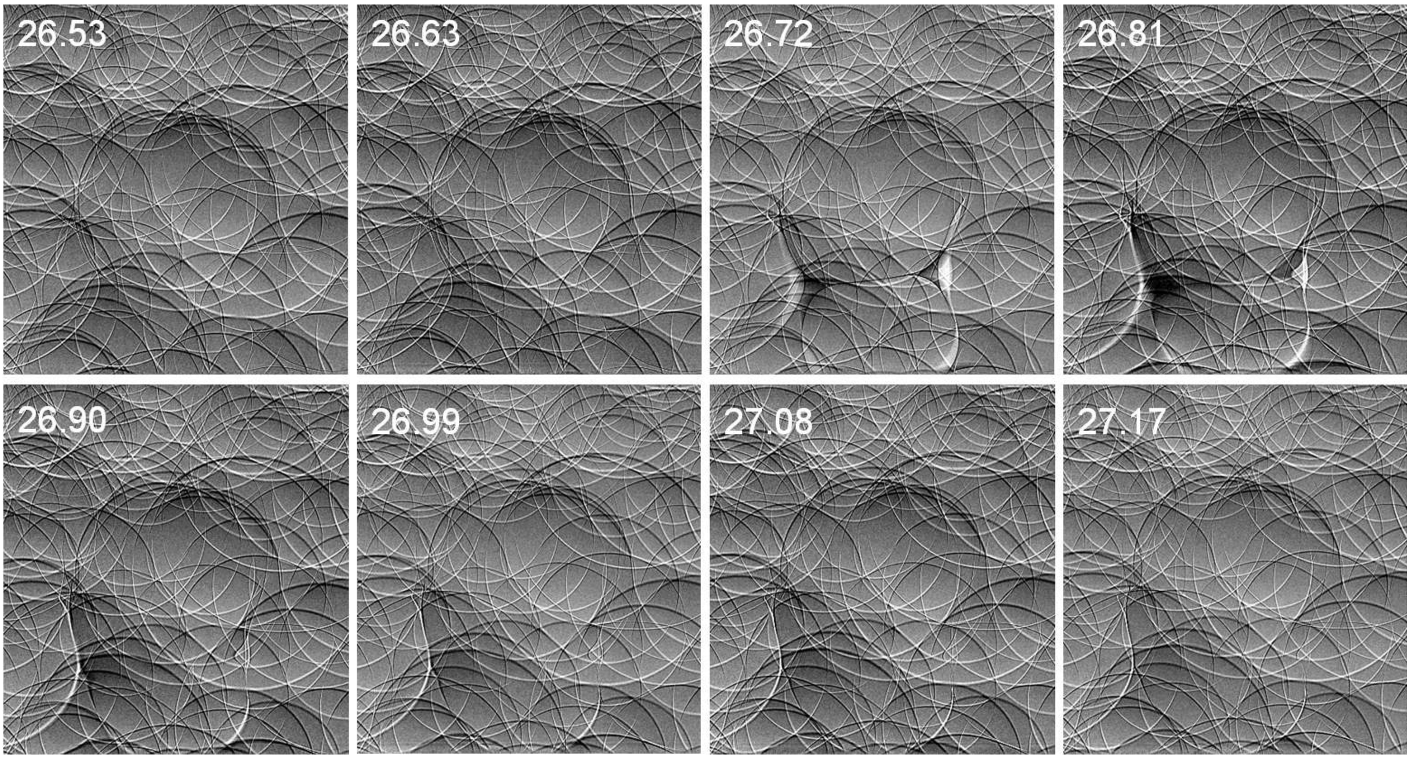

Figure 5. Difference images of succesive frames of the control sample showing a coalescence event and stabilisation of the system (Scale s)

Conclusions

Results from both tomography and radiography experiments have shown clearly that the inclusion of nanofillers affect the evolution of the foaming process. Furthermore, initial optical flow analysis shows differences due to nanofiller morphology on the foaming evolution of the system.

Full analysis of the data obtained is in progress and is expected to lead to the publication of several papers. The reported results suggest that faster tomography scans would provide further information on the material morphology evolution which will enable a deeper understanding on the foaming evolution and dynamics.