EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcosme.do

Reports supporting requests for additional beam time

Reports can 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 determination of dual scale flow for glass & carbon fiber woven fabrics by laminography	**Experiment number**: MA/3491
Beamline: ID19	**Date of experiment**: from: 21/07/2017 to: 24/07/2017	**Date of report**: *Received at ESRF:*
Shifts: 7	**Local contact(s)**: Lukas Helfen	

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Report:

This experiment was dedicted to the investigation of the dual scale flow during the infiltration of epoxy resins in glass fibres. During the beamtime MA3491 (July 2017), one single liquid (an epoxy resin with high viscosity) and on type of fibers (E-glass) were systematically tested, i.e. applying different infiltration velocities, due to the limited time granted for this proposal (6 out of 9 shifts requested). In situ resin infiltration experiments were performed at 26keV, FOV of 5.3x5.3mm and a resolution of 2.2µm using a PCO.edge detector. The projections scan time was 1.5min (50msec exp. And 1799 projections) and the SDD 295mm which allows for Paganing reconstruction. The inclination table angle was set to 32.5º.

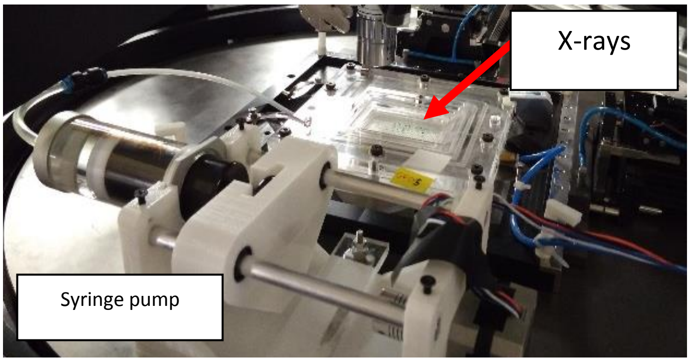

Figure 1: a) Infiltration before laminograms were taken. b) Shows the observation point at different times during the experiment showing the evolution of the flow front

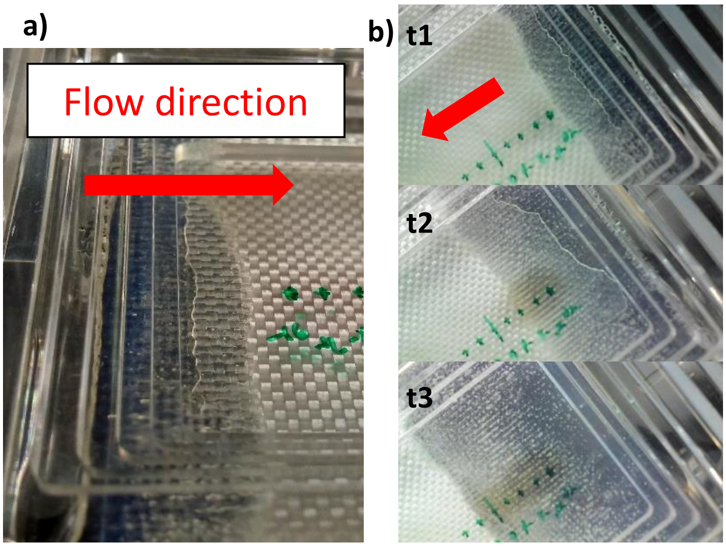

Figure 2: The experimental set-up at ID19-ESRF

During the experiments, different regimes of resin flow were achivied by adjusting the resin injection velocity for each infiltration experiment. This velocity is controlled by a syringe pump, Fig 1, developed specially for this experiment in order to comply with the beamline requests (weight, size, etc). The syringe containing the resin is connected to the inlet of the a special transparent mould developed for this experiement. The PMMA mould (low absorption coefficient) allow observing roughly the flow with naked eye to locate the flow front region and scan it with X-rays. The x-rays go through the thickness of the mould, Fig 1.

The laminograms were acquired in the middle of the sample (thus avoiding any edge effect) at

several distances from the inlet to observe the flow front at different positions as well as the transport processes behind the flow front. These observation points were marked prior to experiments on the mould, Fig 2.
Each experiment was divided in rounds (different infiltration times), in which between 1-4 laminograms were taken. The number of laminograms per round depends on the injection velocity. The observed positions were selected to acquire the position ahead of the flow front, at the flow front and behind it. This sequence allows to obtain the infiltration evolution in the same area of fibres. During the experiments, photograph centered in the observation points were taken from above the mould, Fig 2b.

Already the radiographies at an inclined angle, Fig. 3, allows for the observation of an inhomogenous viscous flow front (different grades of gray shades), through the different interply faces. The generation of porosity is also visble from this simple inspection, which is only posible with the use of X-rays. The reconstruction of some volumes is still been performed and the 3D quantitative information of the already reconstred volumes is been processed. Fig. 4 shows a reconstructed slice at the flow front. Capturing the flow front with different injection velocities will provide valuable information about the dual-scale flow mechanisms in these materials. Besides, the image quality allows to obtain the meso-porosity (intertow) as well as the relevant micro-porosity (intratow), resin meniscus shape, impact of the fibre orientation, pore transportation, etc.

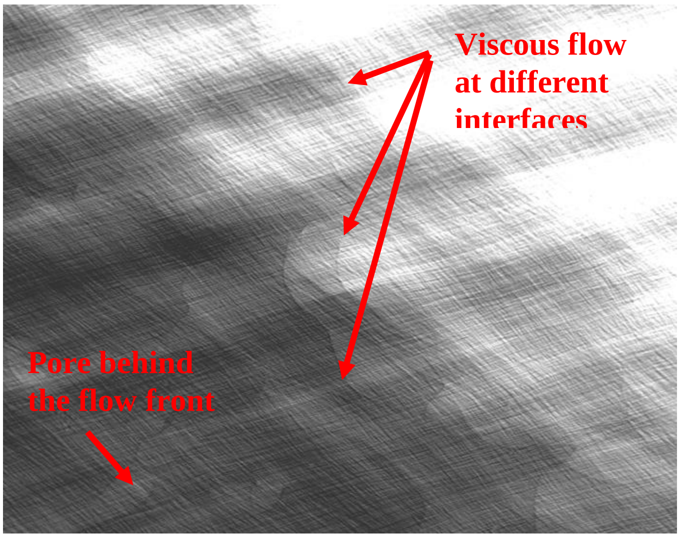

Figure 3: Single radiography during the laminogram acquisition at the flow front. Flow direction from bottom left to top right.

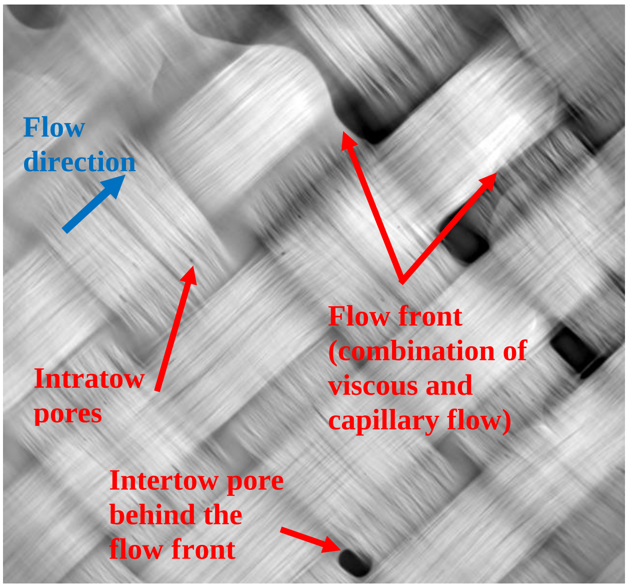

Figure 4: SXL reconstruction of single slice showing intertow and intratow porosiy. The inhomogenewous flow front is also observed.

For instance, a qualitative inspection of the volumes shows already the difference between low and high injection velocities. In the case of low injection velocities capilary forces prevail, Fig. 3(a-b), leaving intertow pores trapped between the fiber tows. At high injection velocities, however, the viscous flow moves faster than the capillar flow and small intratow pores are left inside the tows. The influence of high and low injection velocities on the viscous flow front and, therefore, on the generation of porosity on the final part can be observed in Fig. 4(c-d). A faster viscous flow is more homogeneous in the ply interface (Fig.4c)than a slowler flow (Fig. 4d). In the later case, the heterogeneous flow front generates more voids. All this information will be used to develop appropriate simulations to infer the optimum infiltration conditions for these type of materials.

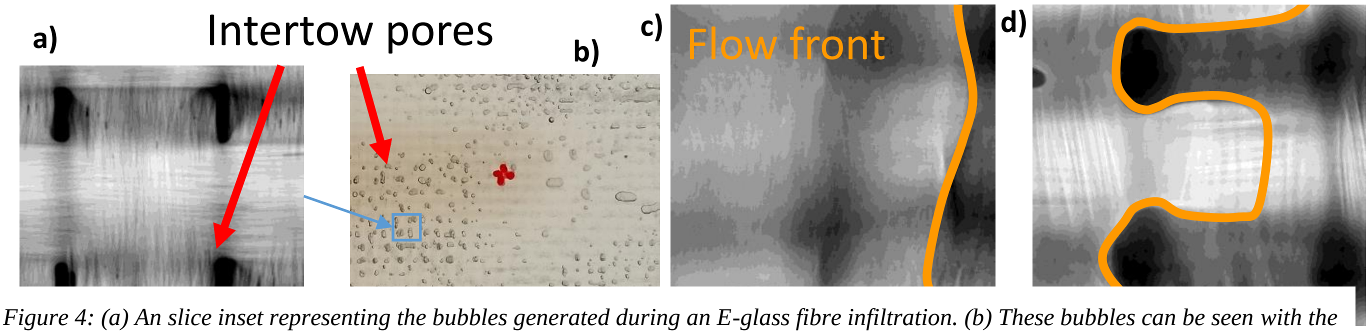

Figure 4: (a) An slice inset representing the bubbles generated during an E-glass fibre infiltration. (b) These bubbles can be seen with the naked eye. (c) The flow front with high injection velocity and with low injection velocity (d). Flow direction from left to right.