EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 real-time tomography of fuel cell diffusion layers under hydrodynamic flow conditions	**Experiment number**: MA411
Beamline: Id 19	**Date of experiment**: from: Nov 18, 2007 to: Nov 19, 2007	**Date of report**: 27.8.2008
Shifts: 3	**Local contact(s)**: Dr. Lukas Helfen	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

This experiment consisted of two logical parts which were investigated independently. Since the experiment is non-standard, it was in the first place required to optimize the experimental parameter set in order to find the best trade-off between spatial and temporal resolution. The occurrence of artifacts reduces (and hence the spatial resolution increases) with the number of projections, while the temporal resolution decreases. Furthermore, the distance between detector and sample needs to be optimized with regard to the image contrast. The use of potassium iodide (KJ) salts as a tracer was tested also to increase the contrast. In the second part of the experiment, water was pumped through the sample and the tomograms where taken as a time series.

The Optimization Experiment included the variation of the beam energy (20.5 keV and 33.4 keV), the use of KJ as a contrast agent to enhance the absorption contrast and the detector distance (10 mm, 20 mm and 40 mm) for optimized edge enhancement due to phase contrast. The test specimen was Toray 060 with 7% PTFE, pre-saturated. The sample diameter was 3 mm in all cases.

The Comparison of the Different Detector to Sample Distances are shown in Figure 1. The contrast between fibers and fluid-phase is fairly poor for a distance of 10 mm, it improves at a distance of 20 mm. At 40 mm, the transition between fiber and fluid phase is over pronounced and the number of artifacts increases significantly. For further experiments, the distance 20 mm was chosen.

The Energy and Tracer Variation is shown in Figure 2. Two energies (20.5 keV and 33.4 keV) had been tested with and without KJ as a tracer. It turned out that the contrast is reduced at the higher energy. The reason is the reduced flux density of the beam at this high photon energy and decreased detector efficiency. The use of contrast agent did not improve the contrast significantly, therefore it was abandoned.

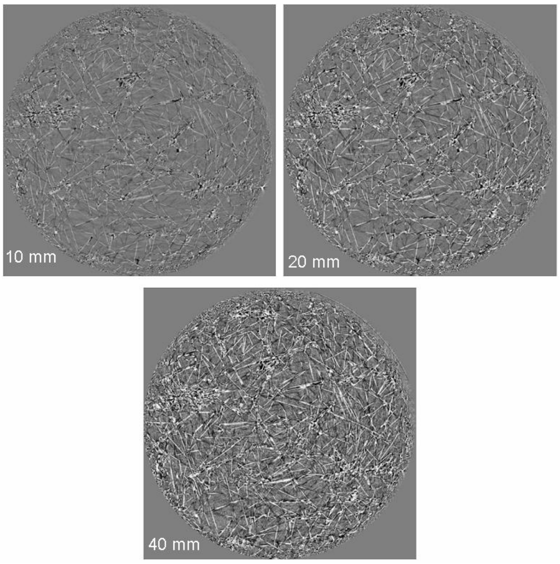

Figure 1: Reconstructed cross sections (3D synchrotron tomography) of pre-saturated Toray 060 with 7% PTFE. Conditions: no tracer, 20.5 keV beam energy, detector – sample distance see images.

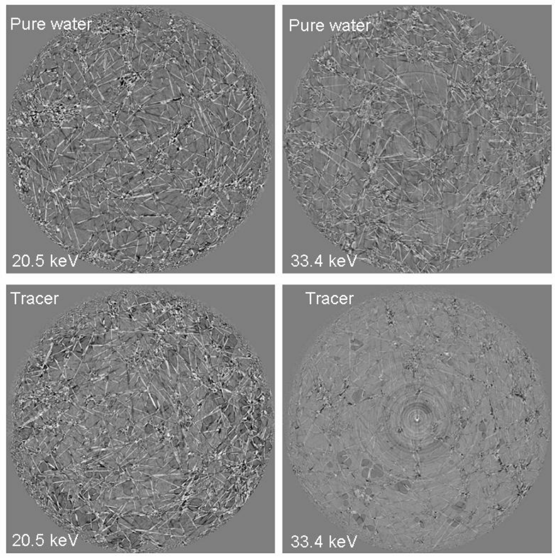

Figure 2: Reconstructed cross sections (3D synchrotron tomography) of pre-saturated Toray 060 with 7% PTFE. Conditions: pure water and tracer, 20.5 keV and 33.4 keV beam energy, detector – sample distance 20 mm.

The Flow Experiment was performed under conditions shown in Table 1.

Parameter	Value	Unit
Beam Energy	20.5	KeV
Distance detector –sample	20	Mm
Number of projections	800	
Time resolution	90	S
Spatial Resolution	2.8	µm
Water flow rate:	3	µl/min
Corresp. current density	17.7	A/cm²
Water pressure	4 - 12	kPa
Sample diameter	3	mm

The experimental setup is shown in Figure 3.

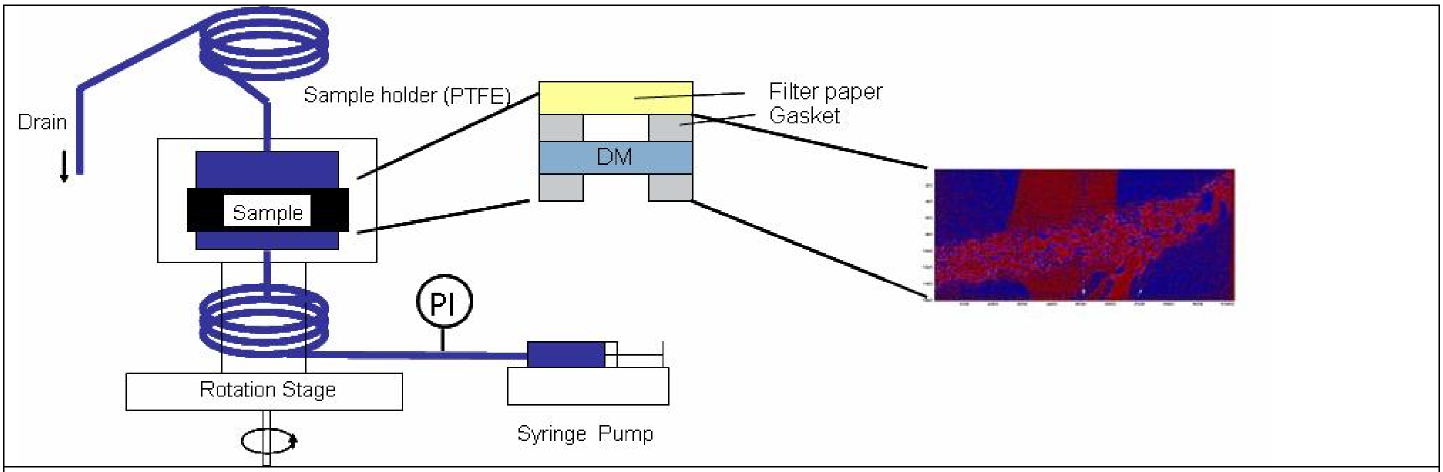

Figure 3: Experimental setup for the synchrotron experiment with water flow.

The sample was mounted in a PTFE tube with silicon foam sealing and a filter paper above the top sealing. The function of the filter paper was to avoid accumulation of water on the low pressure side. This is supposed to be a variable in a later experiment to study the influence of the boundary condition on the water inside the diffusion media. The flow rate was set to 6 µl/min and the sample diameter was 3 mm.

The data from the second experiment shows the same location within the 3D dataset at six successive time steps (see Figure 5). Each tomogram took 90 s (30 s for the image taking and 60 s for the data storage). 90 s elapsed between two consecutive images.

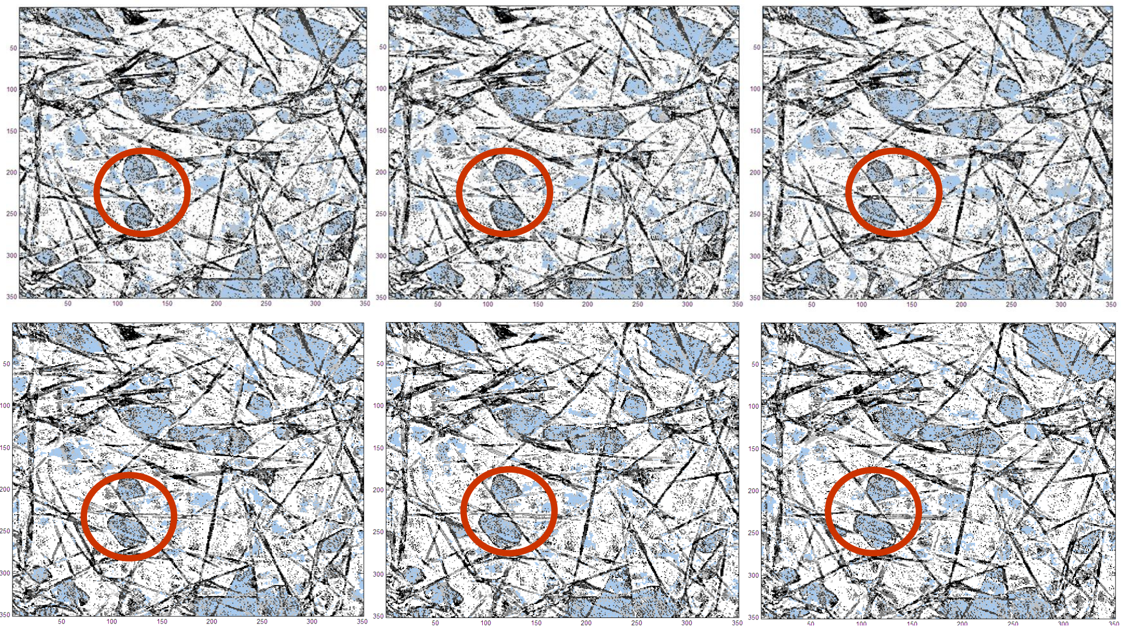
Figure 5: Six successive time steps for the same location within the 3D stack. The red circles mark the locations where the local water distribution changes.

It was found that the shape of the individual water "filaments" changes during the experiment. The data had been segmented automatically by threshold setting. The porosity of the dry fiber structure of the depictured section was 87.5% ± 1.62% (three standard deviations) and the saturation 23.8% ± 3.14 %.
The standard deviation for the porosity is a quality measure for the segmentation algorithm (based on the fibers). The larger standard deviation of the saturation is based on the fact that the water is flowing.

Result

The demonstrated spatial resolution allows us to resolve the fibers, as well as the interface between water and air due to the phase contrast. The time resolution demonstrated in this work, enables the study of the dynamic three dimensional evolution of liquid water structures inside the carbon fiber structure which is unique to this experiment. The contrast of the images obtained is sufficient to perform automated image analysis to separate the water and the fibers from the original image.
All these properties enable the visual investiagtion of slow flow processes in porous substrates, particularly usefull for investigation of fuel cells. The influence of different porous material treatments on distribution patterns under flow can be studied with the choosen setup. The results also indicate the clear prospect that in the future the time-dependent 3D water distribution of running fuel cells could be investigated *in situ*.