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 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.

	Experiment title: In situ imaging of 3D damage evolution in polyamide at different stress triaxiality levels	**Experiment number**: MA-1003
Beamline: ID19	**Date of experiment**: from: 06/05/2010 to: 09/05/2010	**Date of report**: 1st Sept. 2010
Shifts: 9	**Local contact(s)**: W. Ludwig	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Thilo Morgeneyer*

Dr. Henry Proudhon*

Dr. Eric Maire**

Dr. Lucien Laiarinandrasana*

*MINES ParisTech, Centre des matériaux, CNRS UMR 7633, BP 87 91003 Evry Cedex (France)

**MATEIS, CNRS UMR 5510 LyonTech Campus, 25 Av. Jean Capelle, F-69621 Villeurbanne Cedex

Report:

The first observation of the necked area in a tensile specimen, removed before failure was carried out in January 2009 at ID19 and showed for the first time a very particular structure of elongated axi-symmetric columns of voids separated by thin ligaments of material (Fig. 1). This was rapidly analysed and published in the journal of polymer science, part B [1]. Based on the insight of this first observation, a beam time proposal was submitted and accepted to carry out a systematic study of the damage in polymer materials with two main aspects:

1. observe the damaged zone in many interrupted tests undergoing very different mechanical conditions (different stress triaxiality ratios due to different notch radius);
2. observe in situ the growth of voids inside polymer samples during straining.

Quite some time was devoted to setup the beam for optimum resolution. This was indeed a rather important points since some of the materials contained very fine initial voids (around one micrometer) which we wanted to characterize.

A second important aspect was the scanning time since the absorption of the beam by the sample is possibly altering it. This was thus of primary importance for in situ examinations. In the end we ended up using two different tomography setups. In the first case, we used the 0.35 pixel size in binning mode to speed up scanning time (so 0.7 micrometer pixel size) to 3 minutes per scan. We spend 3 shifts in **ultra high resolution** mode on a second tomography setup with 0.12 micrometer pixel size again in binning mode (so 0.24 micrometer pixel size) with 40 minutes per scan. In this case, one has to take a reference scan with a small needle to

correct for wobble errors during the rotation, and enable wobble correction during the reconstruction process.
All tomographic scans used local tomography which worked remarkably well (even on non axi-symmetric samples).

1. OBSERVATIONS OF INTERRUPTED TESTS

   More than 20 samples were examined in this way, resulting in tremendous amount of data available for several materials and lots of different loading conditions (more than 100 scans). In several case, many scan were carried out on one sample to record the spatial evolution of the void growth during the necking. From each scan, the voids are obtained by applying a threshold to the 8 bit raw image and the volume fraction of the porosity is computed. To treat the large amount of data obtained, a python routine was written to automatically load the image, apply the threshold, remove objects smaller than 4 pixels, compute 2D and 3D views and finally compute the void volume fraction. An example for a particular sample is shown below.

   Example for the sample "Pa6_lg_a-s_": 19 scans

   - Polyamide 6
   - Tensile-test interrupted (57.4Mpa):

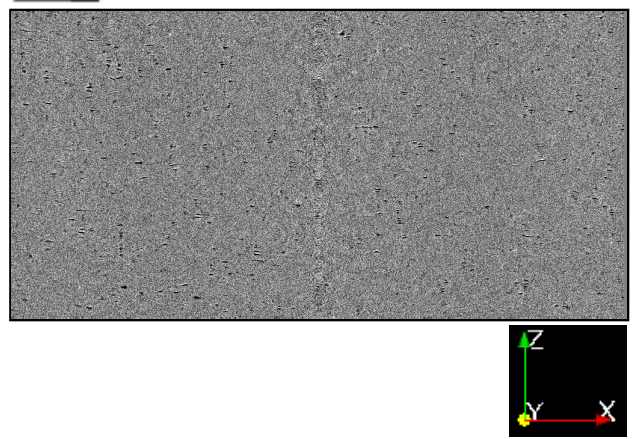   Positions of scans   The Sample
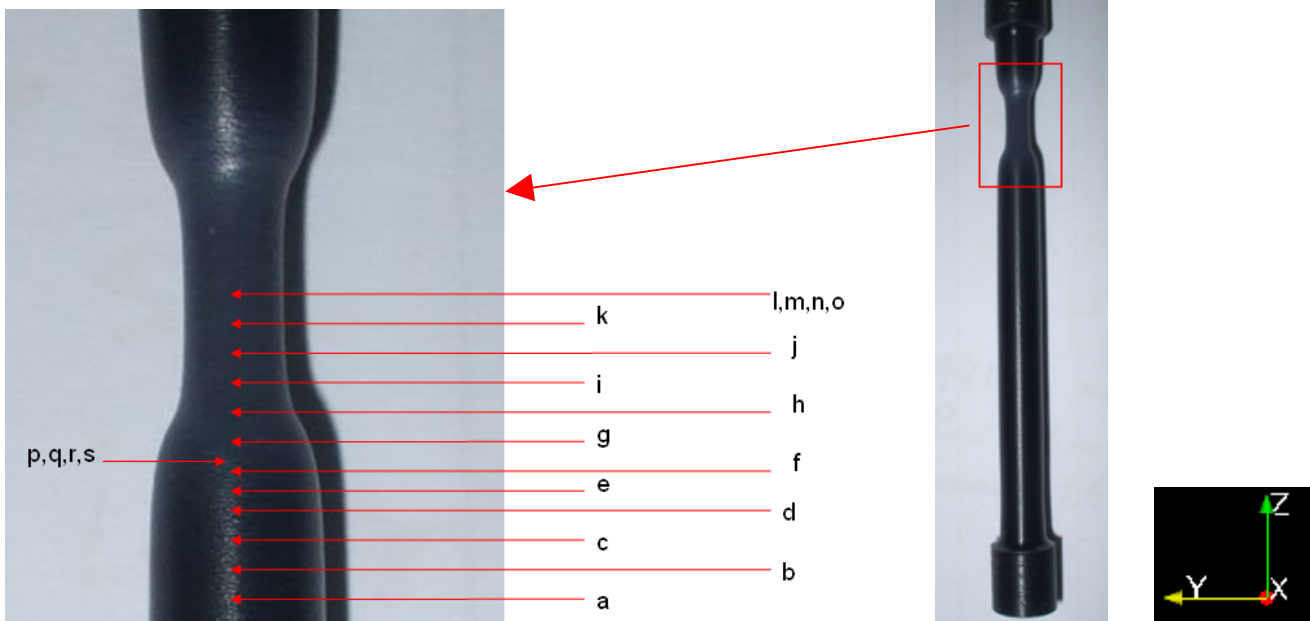
   - Scan "Pa6_lg_a_": "Unstrained material". Only some small voids (which may correspond to initial porosity). Void volume fraction : 1.18%
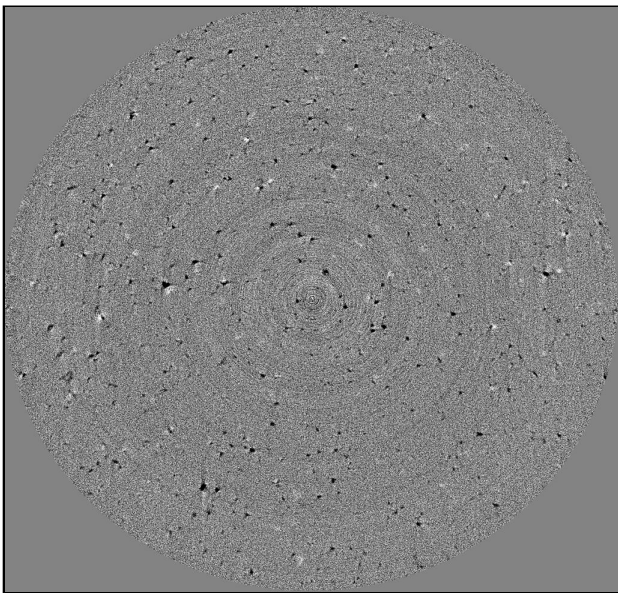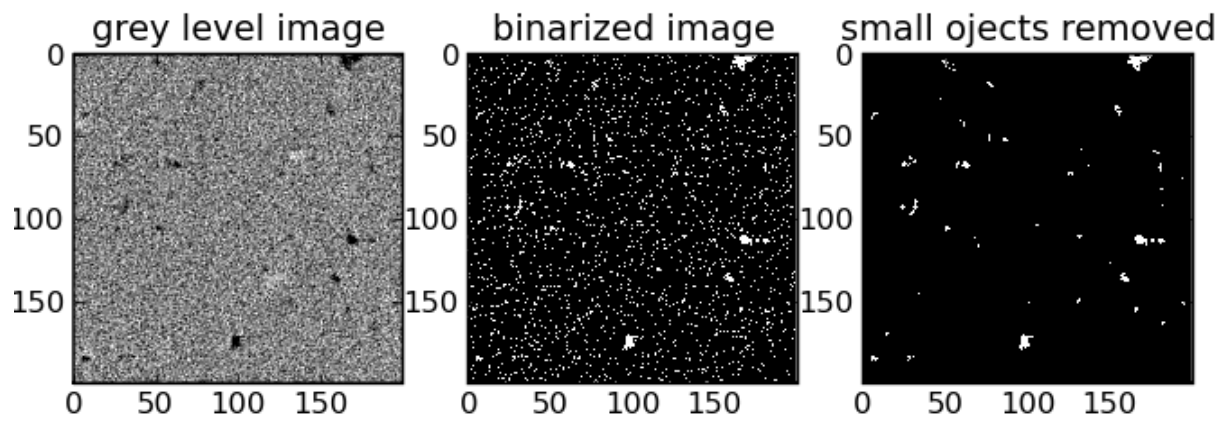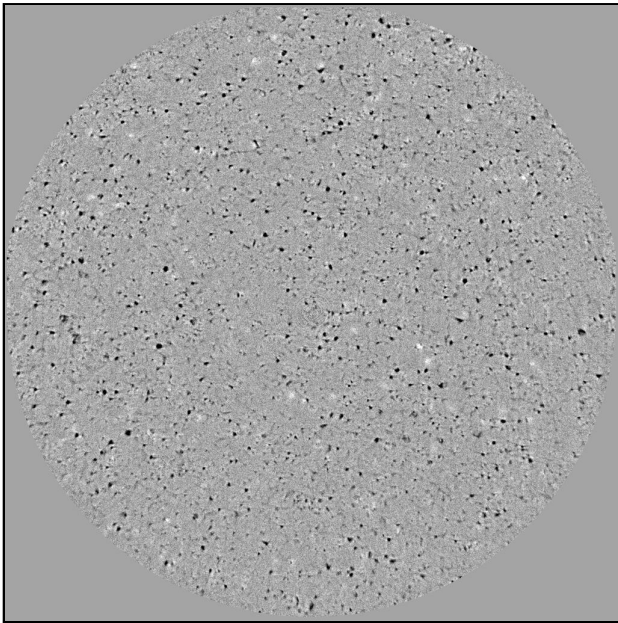
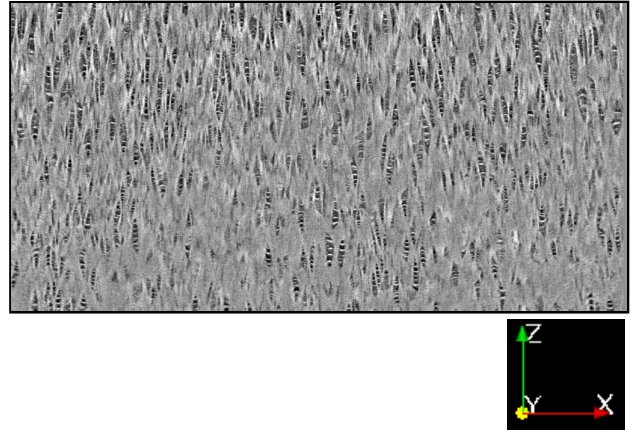Stages of binarisation (to calcul the void volume fraction):

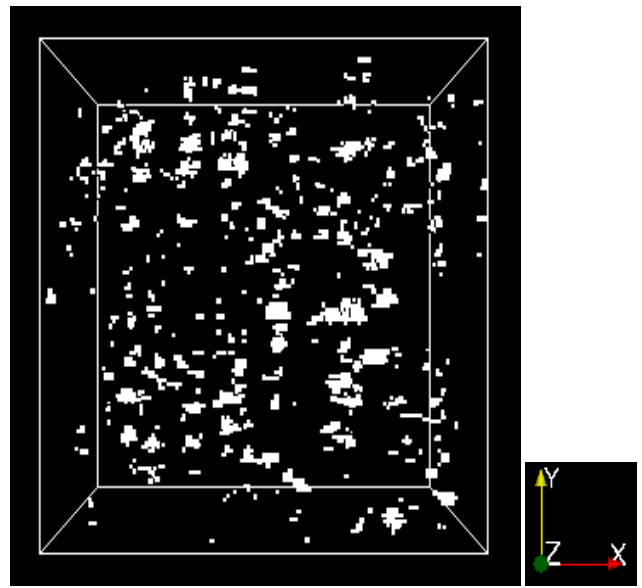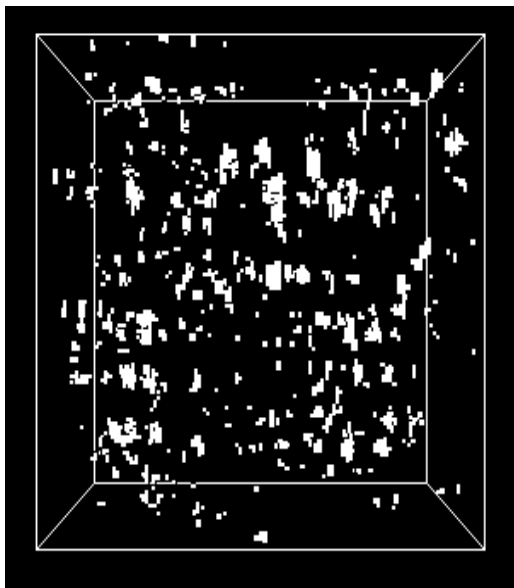- Scan "Pa6_lg_g_": "Begining of the necking-zone": Gradient of damage along the Z axis. Void volume fraction: 2.64%

100µm

100µm

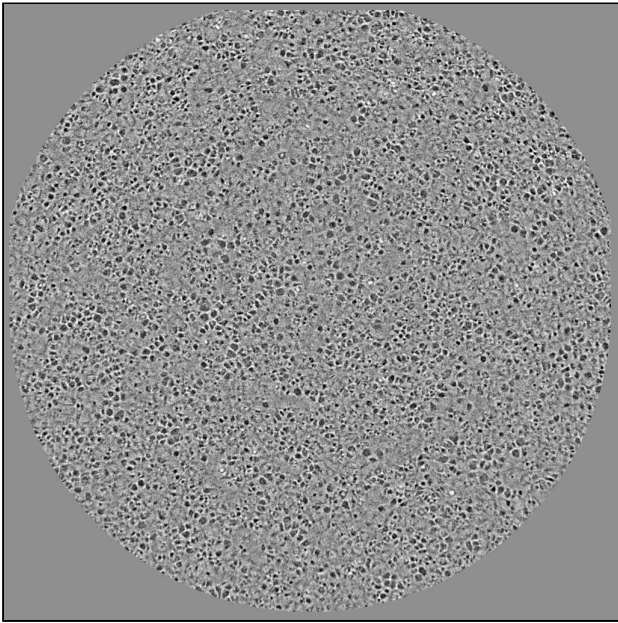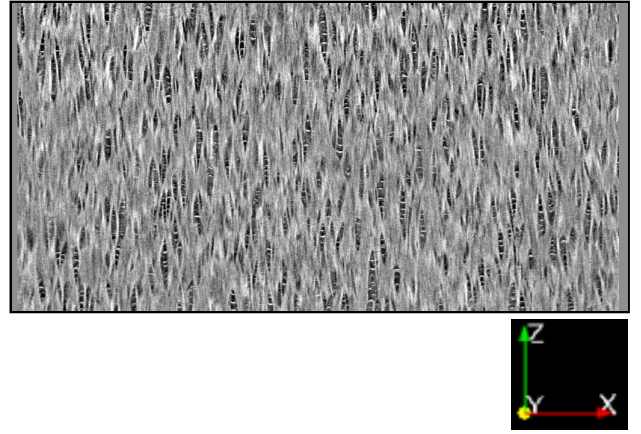
Two 3D pictures:

- Scan "Pa6_lg_i_": Necking zone: very large voids. Void volume fraction : 7.85%

Evolution of the void volume fraction according to the scan:
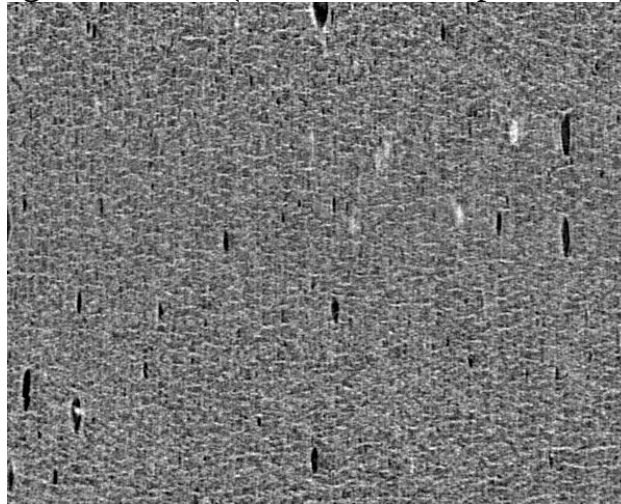

Pourcentage de porosité en fonction des scans pour l'éprouvette pa6_lg_a-s_ size 200 Off 250
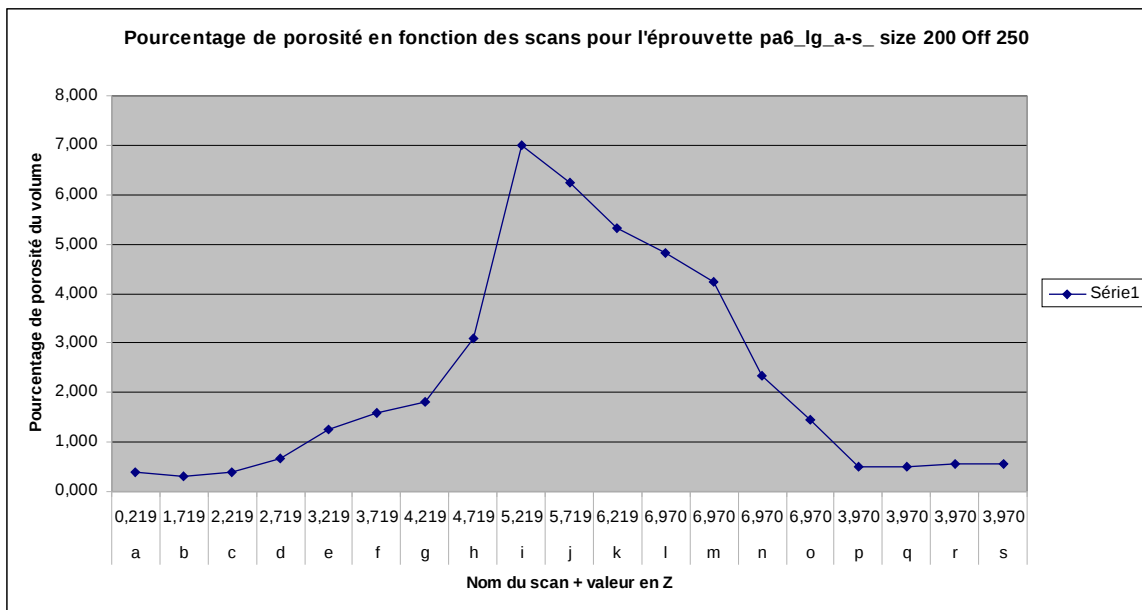
PE examinations with ultra high resolution (0.12 micrometer pixel size)
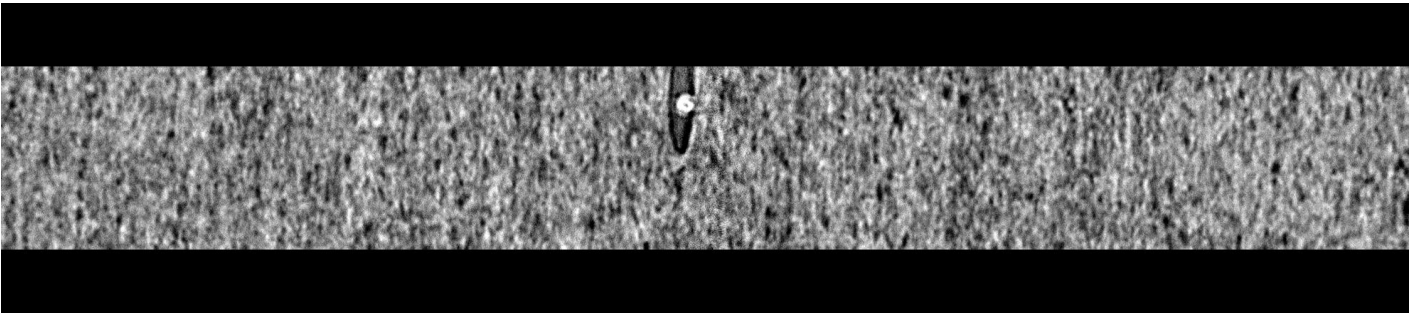
PA11 examinations with high resolution (0.7 micrometer pixel size)

2. IN SITU OBSERVATIONS OF VOID GROWTH

For in situ examination, notched samples were placed within a specific loading frame developed at INSA Lyon, which was used many times before with metallic materials. A sample was then strained step by step and a scan recorded each time.

Even with an optimised setup with a 3 minutes scan duration, it appeared that the samples were damaged by the repeated scans and failed prematurely.

Given the fact that images were less good due to the presence of the loading rig, not too much time was devoted on solving those problems as very good data were obtained on the ex-situ examinations.

Some complementary analyses were carried out a few rubber based samples after fatigue cycling. Large holes were uncovered and images at different loads.

REFERENCES

[1] Damage of semicrystalline polyamide 6 assessed by 3D X-ray tomography: From microstructural evolution to constitutive modeling, *Journal of Polymer Science Part B: Polymer Physics*. Special Issue: Deformation, Yield and Fracture of Polymers, Volume 48, Issue 13, pages 1516–1525, 1 July 2010, DOI: 10.1002/polb.22043