ESRF	**Experiment title:** Tomographic/acoustic inspection of polymer foams during in situ tensile test	**Experiment number**: ME 799
Beamline: BM05	**Date of experiment**: from: 20/07/2004 to: 23/07/2004	**Date of report**: 02/2005
Shifts: 9	**Local contact(s)**: Dr Joanna HOSZOWSKA	*Received at ESRF:*

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Report:

Damage mechanisms and growth have recently received a lot of attention in the physics community [1]. In order to predict failure time of materials, a new approach has been considered : fracture can be viewed as a critical phenomenon. Interestingly, for some materials fracture can be described as a clustering of microcracks [2-4]. The main prediction of this theoretical description is that the damage rate presents a critical divergency close to the failure time [5-6].

We focus on heterogeneous materials whose fracture can be described as a phase transition [7]. Indeed, one important assumption of this new model is that macroscopic cracks are the result of a nucleation and percolation process of microcracks. Therefore, such phenomenon is better observed with materials that have numerous points where microcracks can stop so that global rupture is not controlled by a single event (nucleation and growth of a single crack and no appreciable precursors). These termination points are composed of heterogeneities.

Solid polymer foams are appropriate for such study: the degree of heterogeneity can be adjusted, their mechanical properties change with temperature and they are more and more used in different technological uses. We use polyurethane (PU) foams which can be considered as controlled model materials [8].

Our main experimental tool is the monitoring of the acoustic emission signatures of the microfractures, which occur during mechanical tests. We record both the spatial and time distributions of acoustic emission emitted by a sample: each microcrack nucleation corresponds to a burst of energy that can be localized on the widest face of the specimen by considering the difference in propagation time to two different detectors. So far, we have been able to evidence diffuse microcrack nucleation and the progressive localization into a dominating crack, with a good fit of the amplitude and time intervals between two damaging events by a power law.

Now, by combining the monitoring of the acoustic emission and a X-ray tomography technique during a mechanical test, we are able to correlate the acoustic emission signal with the microscopic cracking event. Thus, we analyze the deformation mechanisms during in situ tensile tests. Specimens of PU foams as in Fig.1. are loaded with a special rig designed for this purpose [9]. We studied PU foams of different densities : from 10% to 62% porosity.

A PMMA tube ensures transmission of the load between the upper mobile grip and the lower fixed one. This tube was carefully polished to ensure that it does not affect X-ray absorption. Force and displacement are recorded on a computer and monitored during the test. The rig is directly mounted on a rotating stage between the beam and the detector.

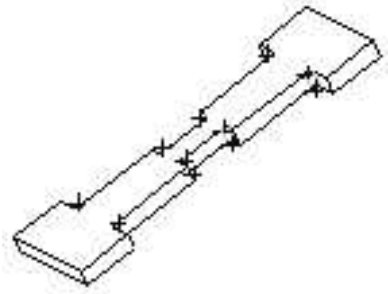

Figure 1 Specimens for the in situ tensile tests

Using the different images obtained by X-ray tomography we managed to do a particle size analysis of the PU foams of different densities (Fig.2). We notice that the mean diameter increases with the porosity and the more the density is high the more the distribution is sharp.

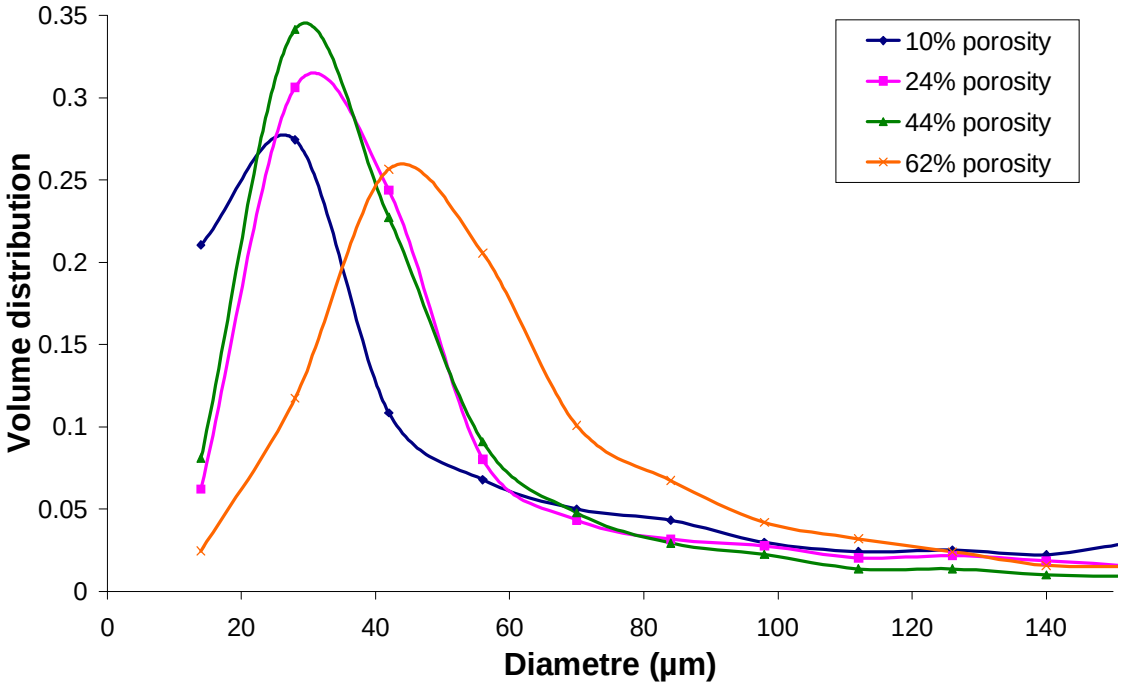

Figure 2 Particle size analysis of PU foams of different porosities

The different samples are imaged at the initial state and different levels of tensile strain. At the same time, we observe the mechanical stress versus strain and we try to correlate this evolution to the number of A.E events. In addition, the position of microcracks is recorded along the specimen length. Spatial localization of A.E is a technique which has been widely used in seismography, and to map the nucleation and development of fractures. In our set-up, as we use only two sensors, the knowledge of the wave speed in the material and the difference in arrival times at each sensor of a given wave is not sufficient to determine the precise location of the A.E. However, we can get an idea of the typical distance from the sensors for the A.E assuming it occurred on the median line joining the centers of the two sensors. Besides, only a certain amount of events can be situated along the specimen : only those whose intensity is big enough to reach both sensors can be localized.

Here we present the results for the PU foam of 10% porosity, the different states studied are : initial states, 8.8%, 12.6%, 16.6%, 18.5% and 21.5% (Fig.3). After each stage, the crosshead displacement is interrupted and the sample is relaxed for a time long enough to avoid blurring caused by displacement of the sample during the relaxation process. Our observations are shown as 2D slices numerically extracted from the 3D images.

We notice that there is a lack of A.E bursts at the beginning of the loading, this may be due to the deformation of the material coming without any wall breaking (case for the initial state and 8.8% strain in Fig.3). Then, above a certain degree of strain, walls between cavities begin to collapse i.e. the growth of a crack ends as soon as it encounters a pore : this can be seen in the 2D tomographic slice of 12.6% strain in Fig.3 In fact, AE signals represent wall breaking between adjacent pores (Fig. 3, 16.6% of strain). Afterwards, crack propagation is more likely to happen towards the end of the test (Fig. 3, 21.5% of strain). The separated events lead to a more important one : the microcracks coalesce and culminate in the catastrophic rise of a global crack implying the material's fracture. Actually, the concentration of microfractures may be a good indicator that the sample is approaching failure.

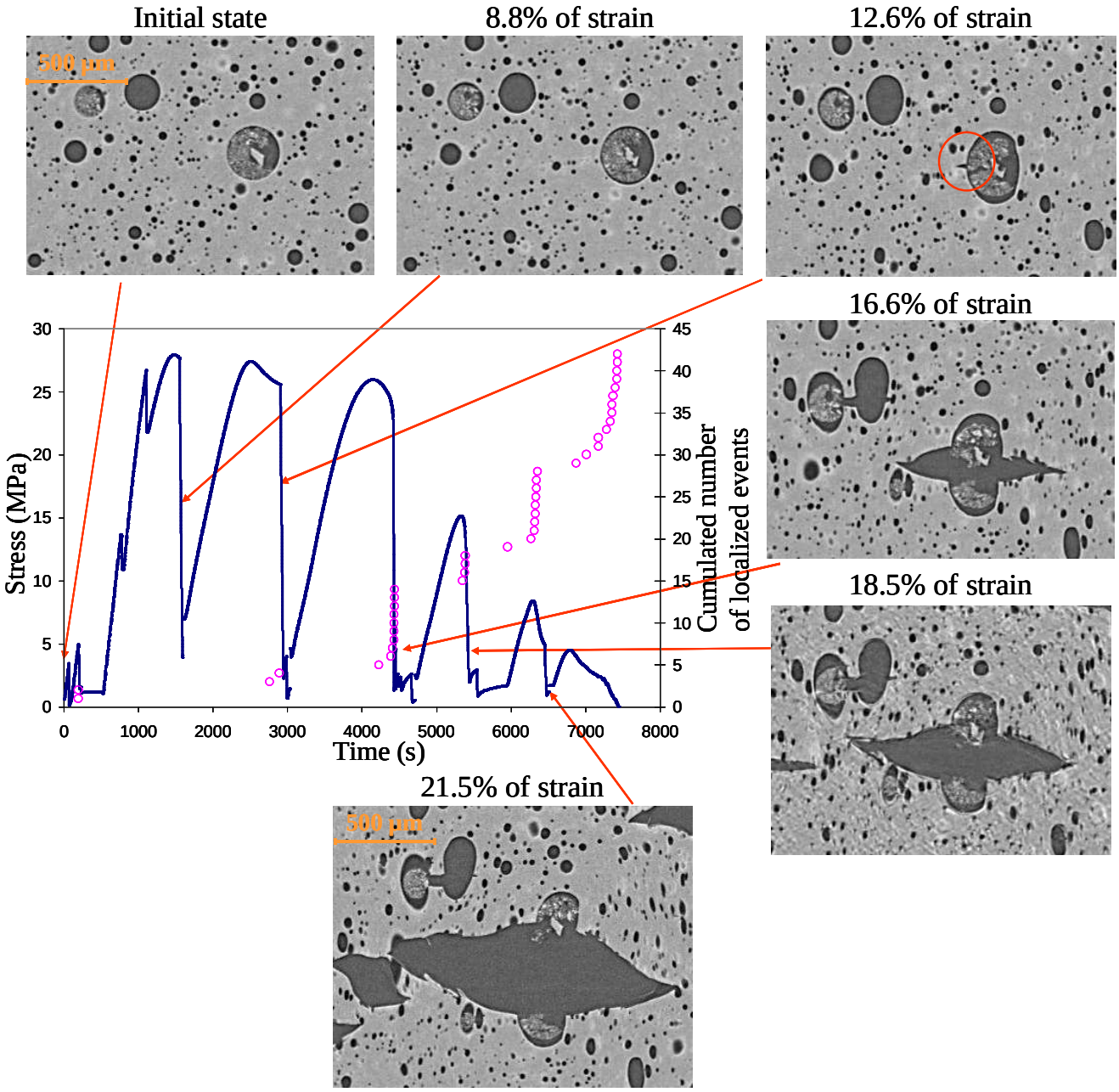

Figure 3 Stress/strain curve with acoustic activity and 2D extracted tomographic slices at different strain levels

The location of the different microcracks is recorded along the specimen length in Fig.4. During the in situ tensile tests, tomography is performed only in the center of the sample. We managed to correlate the acoustic emission bursts to the observations of internal microcracks. The acoustic emission bursts correspond first to wall breaking between adjacent pores then to different crack growth.

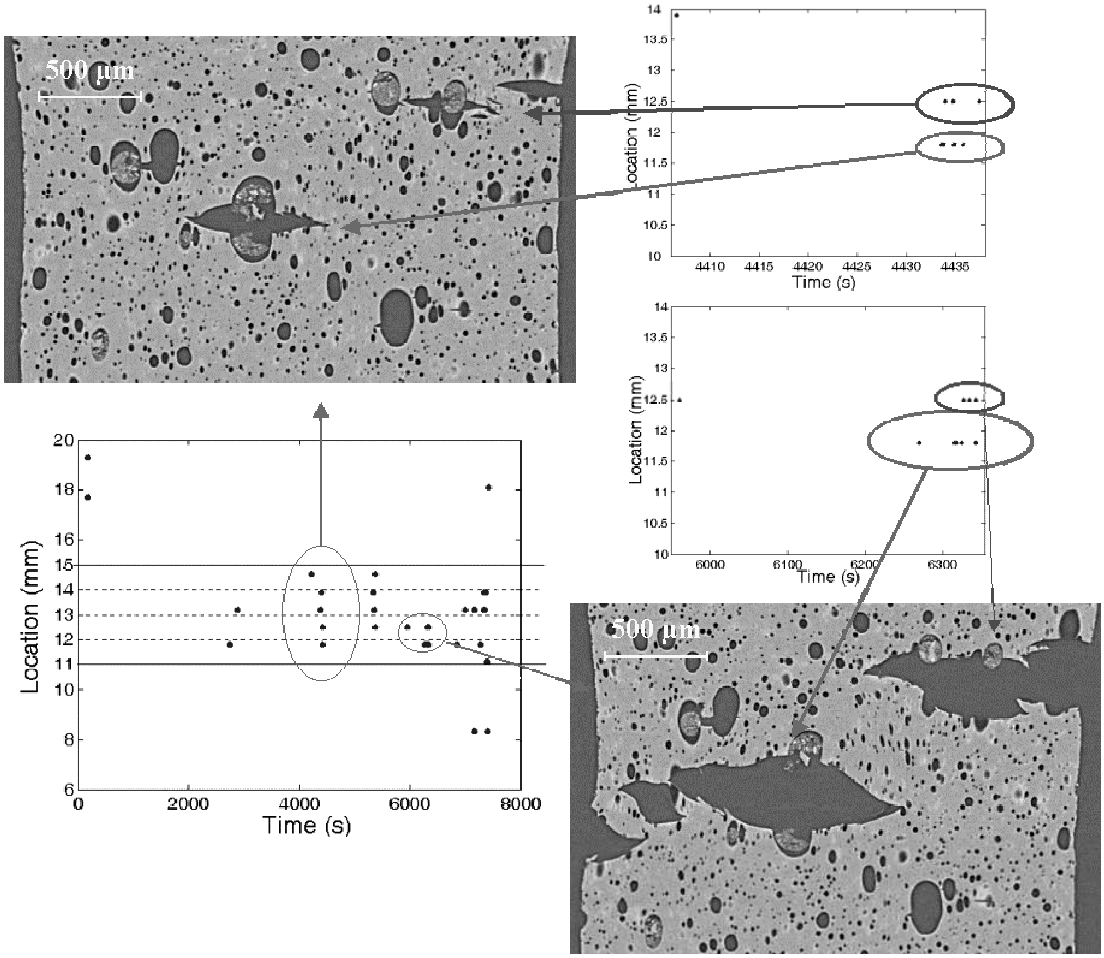

Figure 4 Correlation between the acoustic emission bursts and the observa tions of internal microcracks

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