Polymeric foam behaviour under impact tests: deformation study by micro tomography

P. Viot¹ & D. Bernard²
¹LAMEFIP, ENSAM de Bordeaux, Talence, France
²ICMCB, CNRS, Université PESSAC, France

Abstract

The mechanical behaviour of polymeric foams depends on several parameters such as temperature, material density and strain rate. This last point implies that compression tests on conventional testing machines are not sufficient. The behaviour characterisation requires special apparatus such as the fly wheel, drop tower or Hopkinson bar, allowing high speeds of compression.

The studied polypropylene foams are multi-scale materials; agglomerated grains (diameter from 2–3 mm) are visible to the naked eye and, at the microscopic scale, each grain is composed of smallest closed cells (diameter of a few tens of microns). The response of a sample to a shock presents three regimes; an elastic phase followed by a plastic phase and finally the densification. The plastic phase is of prime interest since a great part of the shock energy is dissipated there.

Micro tomography was used in order to better understand the damage mechanisms during the plastic plateau. The final objective of this work is to determine the 3D-strain field of porous materials at several levels of shock. As tomography is not fast enough to directly follow the impact deformation, interrupted impact tests were carried out by controlling the levels of the sample deformation. Between each impact step, a micro tomographic analysis will enable the analysing of the progressive deformation of the sample. The results of these impact tests completed by the micro tomographic visualisation are presented and commented in this paper.

Keywords: foam, cellular material, dynamic loading, impact, micro tomography.
1 Introduction

Polypropylene foam is largely studied for a few years [1]. This multi-scale cellular material is composed of agglomerated polygonal grains (diameter of 2 millimetres) having a wall thickness about ten micrometers. Each grain is composed of small closed cells which are random distributed (figure 1). Cell diameters are variable, from 10 micrometers for the smaller ones up to 60 micrometers for the larger ones. The cell wall thickness seems constant and smaller than 1 micrometer. If a large number of works currently concerns the identification of the material elastic response in relation to the microstructure, the impact engineering objective is more particularly focused in the plastic behaviour characterisation since, during this phase, the material is able to dissipate a significant part of the shock energy with a constant stress (figure 2). The densification final stage of the material behaviour is less interesting because the stress increases exponentially.

Previous works on this material have shown that the plastic plateau corresponds to the cell wall buckling and also to those much thicker (and rigid) of the cellular grains [2]. To complete these studies, it is necessary to show the influence of grain buckling on the material global response; the grain wall thicknesses are significant and, consequently, their deformations dissipate an important energy. The main objective of this work is thus the identification of the expanded grain deformation during an impact. Then, this first step will allow the porous material structure modelling – at the grain scale - in order to reproduce its real behaviour by the use of a new phenomenological law taking into account the structure dynamic buckling.

Figure 1: SEM picture of the foam mesostructure.

Until now, it was impossible to measure, in the heart of the sample, the buckling phenomena of foam structure without a preliminary cutting, often cause of damage as significant as those to be estimated. Some new non-destructive measurement methods permitting the 3D reconstruction of inhomogeneous material structure are now available. Micro tomography is one of them. This technique proved to be well adapted to multi-materials and porous materials that evolve according to external conditions; examples are glass sintering [3], pressure solution in rocks [4], dissolution by reactive percolation [5], etc.

However, this technique imposes that the sample structure is not perceptibly varying during the acquisition. Real time characterization during dynamic
compression is then impossible. The project of characterizing in 3D the foam deformation and its damage propagation by buckling was sufficiently interesting and original to develop a specific dynamic test methodology compatible with micro tomography requirements. This article presents this procedure and some preliminary results.

![Diagram](image)

Figure 2: Typical evolution of the stress–strain response of a polymeric foam under compression.

## 2 Experimental method

The adopted experimental method consists in carrying out interrupted impact tests followed by micro tomography measurements. First, a micro tomographic record of the intact foam structure (not impacted) is done (point A, figure 2). This sample is then impacted using a drop tower. During this loading, the deformation amplitude is limited to a determined value (2 mm for a sample height of 10 mm). The sample is kept in compression and placed once again on the micro tomography set-up to carry out a second record (point B, figure 2). These operations (impacts and X rays scans) are repeated until the complete densification of the foam (points C, D, E and F). The cellular material deformation can then be evaluated from the 3D reconstructions at the different steps of the dynamic test.

### 2.1 Micro tomography principle

Consider a homogeneous material characterized by $\mu$, its linear attenuation coefficient, and illuminated by a monochromatic beam of energy $E$, the ratio between $N_0$, the number of incident photons, and $N$, the number of photons transmitted through the material thickness $L$ is given by Beer-Lambert’s law [6].

$$N = N_0 \cdot e^{-\mu L}$$

(1)
For a heterogeneous material, $\mu_L$ must be replaced by the integral of $\mu$ along the photons path. The number of transmitted photon $N'$ is then:

$$N' = N_0 e^{-\int_{\text{path } L} \mu(l) \, dl}$$

(2)

By measuring $N_0$ and $N'$ the above integral of $\mu$ can be calculated:

$$\ln \frac{N_0}{N'} = \int_{\text{path } L} \mu(l) \, dl$$

(3)

Let us now consider an object characterized by its 3D mapping $\mu(x,y,z)$. We place it (Figure 3) on a rotation table ($z$ is the rotation axis) in the X-ray beam (direction $v$) and before the 2D detector (plane $(u,z)$, with $u$ perpendicular to $v$).

![Micro tomography apparatus scheme.](image)

Figure 3: Micro tomography apparatus scheme.

Each section perpendicular to $z$ can be considered individually because the synchrotron beam is parallel. For an angular position $\theta$ of the object, $N'$ can be measured at each point $u$ on the detector and relation (4) can be used to calculate the projection $P$:

$$P(\theta) = \left\{ P(u,\theta) ; \ u = \pm r \right\} = \left\{ \ln \left( \frac{N_0(u)}{N'(u)} \right) = \int_{\text{Ray } u} \mu(v) \, dv ; \ u = \pm r \right\}$$

(4)

where $(-r)$ and $(+r)$ represent the limits of $u$ on the detector, and $\text{Ray } u$ the line parallel to $v$ hitting the detector at $u$. The set of projections $P(\theta)$ for $\theta$ varying between $0^\circ$ and $180^\circ$ is Radon’s transform of $\mu(x,y)$. In 1917, Radon proved mathematically that it is possible to invert this transform and thus it is possible to reconstruct the 3D-map of $\mu$ from a set of projections ([7], [8]).

From the calculated 3D-map of the linear attenuation coefficient $\mu$ [cm$^{-1}$] it is possible to obtain a 3D image of the material thanks to the fact that, for a pure material, the mass attenuation coefficient $\mu/\rho$ is correlated to the photon energy $E$ and to the atomic number $Z$. For energies lower than 200 keV, the following relation can be used:
\[
\frac{\mu}{\rho} = K \frac{Z^4}{E^3} \quad \text{with } K \text{ constant} \quad (5)
\]

where \(\rho\) is the material volumic mass \([g/cm^3]\) and \(K\) a constant. Equation (5) implies that at a given energy \(E\) (an adjustable parameter in synchrotron tomography), the linear attenuation coefficient \(\mu\) is proportional to \(\rho\) and \(Z^4\).

The mass attenuation coefficient \(\mu/\rho\) of an heterogeneous material containing \(i\) elements (mass concentration \(w_i\) with specific mass attenuation coefficient \((\mu/\rho)_i\)) is obtained with the following relation:

\[
\frac{\mu}{\rho} = \sum_i w_i \left( \frac{\mu}{\rho} \right)_i
\]

X-ray computed micro tomography consists then in detecting the residual energy of a beam that crosses through a sample (this is a radiography giving \(N'\)) for a large number of different angles (between 900 and 1500 on 180\(^\circ\)). Some reference images are added to control the noise and the homogeneity of the incident beam (this gives \(N_0\)). The 3D representation of the X-ray absorption by the sample is then numerically reconstructed from all these 2D images. The most commonly used algorithm for reconstruction is the filtered back-projection method [6].

At a given energy, absorption of X-rays is function of several physical parameters, mainly the local density and the atomic number of the crossed material. In a porous material, if all the components of the solid phase have about the same X-ray absorption, the 3D-image of the absorption can be transformed into a 3D-image of the porosity. The use of micro tomography in material science thus requires that the studied material present differences of internal absorption, which can be measured within the studied volume elements (voxel).

![Figure 4: Sample sizes vs. precision of the 3D images. (a) The two samples: for images b (centre) and c (top). (b) Tomographic result for a pixel size of 0.3 µm. (c) Tomographic result for a pixel size of 4.9 µm.](image)

The data presented in this paper have been obtained on the BM05 beam line at the European Synchrotron Radiation Facility (ESRF) in Grenoble (France).
The tomographic apparatus available on BM05 allow the acquisition of 1024 x 1024 or 2028 x 2048 pixels radiographs with pixel sizes ranging from 40 µm to 2 µm. The selected optical set-up placed after the scintillator that converts, as efficiently as possible, X-rays to visible light fixes this pixel size. Those parameters also determine the size of the sample. Indeed, in classical tomography, samples must have a maximal lateral dimension such as the entire projections match the field of view of the digital camera. The maximal dimension is then equal to the pixel size multiplied by the number of pixels comprised on a row of the detector. For small foam samples which diameter is less than 0.8 mm, the tomographic device authorizes to visualize in 3D the cell morphology of the polypropylene foam (figure 4b). For our study, because impacts have to be done on samples representative of the foam structure, the selected sample diameter was 10 mm. With this size it is not possible to visualize the cells with a good accuracy but it is sufficient to measure the location and the dimensions of larger grain walls (figure 4c).

2.2 Impact apparatus

The foam microstructure characterization between each dynamic compression requires impact tests carried out near the micro tomography set-up. It was then necessary to develop a portative drop tower able to carry out interrupted impacts.

This drop tower was specifically designed for this study. It includes two subsets: a loading module and a measurement set (figure 5). The loading module consists of a cylindrical projectile (mass 0.37 kg) guided in a rectified metallic tube of height $H = 1.6$ m. This set is placed above the measurement module constituted of an aluminium base, a die and a punch. The sample is closed inside the die.

![Figure 5: Drop tower scheme.](image)
During the test, the projectile is dropped at the top of the tube with a null initial speed and freely accelerates. The projectile comes in contact with the punch which plunges into the die and compresses the sample. At the beginning of the impact, the measured speed of the punch is 5 m/s. The punch speed is constant during the compression since the projectile energy is clearly higher than the energy spent in the foam deformation.

The successive compressions are carried out by using several punches of different lengths (l = 16, 18, 20, 22 and 24 mm); a maximum deformation of 2 mm is imposed at each impact. This die – punch set was designed in order to apply a uniaxial compression on the foam sample and also in order to be removed of the drop tower and placed on the micro tomographic table. After an impact, the punch shoulder stays against the die, the sample is kept compressed during the tomographic measurement.

This compression device is instrumented to measure compression force and sample deformation during the test. The punch displacement is obtained from a laser sensor. The sample strain and strain rate can be deduced from this the laser sensor measurement in considering the hypothesis of infinitely rigid device. To complete (and verify) this measurement, impacts are filmed with a high-speed camera Phantom V4, at the frequency of 5000 frames per second and with a resolution of 128 * 256 pixels². One can obtain also the punch rate in analyzing point displacement on each image. A sensor (placed below the die) measures the impact force; it was made from an aluminium tube instrumented by 4 gages connected in Wheatstone Bridge.

3 Results

First results were carried out on polypropylene foam of several densities. Results presented in this paper concern a foam volumic mass of \( \rho = 80 \) kg/m³.

3.1 Impact results

Figure 6 shows the first tests obtained with the impact apparatus. These first results have to be confirmed by complementary experiments but they allow already to show the cellular material behaviour under impact.

Two test results are plotted. The black line concerns a first test that was not interrupted in order to show the three stages of the material behaviour. One finds firstly the foam elastic behaviour followed by a plastic plateau (characterized by the plateau stress \( \sigma_{pl} \)). Compression ends with the rise in stress during the foam densification.

The second test (large line) has been only done in two impacts in order to show the behaviour of the foam successively impacted. The first impact (curve AB, fine line) presents an elastic stage and the beginning of the plastic plateau. The loading was interrupted in point B. After this first impact the sample deformation (distance AC) was limited to 2.9 mm. The second impact began in point C. The elastic behaviour (zone CD) seems to be less rigid. After this stage,
the foam plastic response is similar to the one of the first sample (continuous test). The two curves are very close.

Hence, curves obtained from this drop tower display strong oscillations attenuating during impact. This phenomenon is due to the impact wave propagation through the sample and the compression device.

Figure 6: Evolution of the force as a function of displacement for a PPE foam dynamic compression.

3.2 Microstructure deformation analysis

Micro tomography measurements were carried out on a sample before the first test and after each impact. The complete modelling of sample structure requires a large computing time. The reconstruction optimization had to be done on a small cubic zone of the sample in order to obtain quickly the calculation parameters. A small cube of 2 mm side was then extracted in order to visualize, for the first time, the deformation mechanisms at the expanded grain scale. Its location was defined in the lower zone of the sample to follow after each impact the evolution of the foam structure deformation embedded in the cube.

The first 3D modelling (fig. 7a) shows clearly the foam structure –before impact- in different vertical plans. Darker lines correspond to the grain walls; one finds the same thickness of these walls measured by other optical techniques (SEM, optical microscopy). Inside the grains, it is harder to distinguish the material cellular structure; some macro defaults of significant sizes can be correctly observed but foam cells (which sizes are closed to 60 mm) cannot be visualized with accuracy.
Figure 7: Evolution of the structure deformation. (a) Before impact. (b) After dynamic compression of 1 mm. (c) After dynamic compression of 3 mm. (d) After dynamic compression of 5 mm.
Figures 7b, c and d present the 3D reconstruction results carried out after the first three impacts. The grain wall buckling is not apparent on figure 7b where the structure displacement can only be observed (in comparison with the figure 7a). In figure 7c, the buckling of grain walls appears clearly in some plans in the loading direction (vertical axis). This deformation mode is not obviously observed on the walls along another direction. The last reconstruction (fig. 7d), obtained after the third impact, confirms these observations; strong localizations of buckling are highlighted on the grain walls. The inhomogeneous deformation evolution of the cellular mesostructure is shown. Some grains seem to be completely densified (in the lower part of the cube).

4 Conclusion

Compression dynamic tests were carried out on polypropylene foam. The global behaviour of the cellular material has been identified. Micro tomography measurements (done after each impact) allowed the visualization of the grain walls buckling phenomena. 3D reconstruction of a Representative Elementary Volume (REV) of cellular material structure showed the buckling of grain walls and the non-homogeneity of the deformation. Moreover, the macro default deformations can be observed at a finer scale.

This 3D reconstruction work must be continued in order to extract from these data the grain shapes before and after impacts, and more precisely, the wall geometries and their thickness. These final results could be exploited to better understand the correlation between the structure deformation and the material response.

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References


