Structural analysis of advanced polymeric foams by HR X-ray CT

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• Advanced polymeric foams
• Structural investigation of foams
  • CT set-up for acquisition and reconstruction
  • Segmentation. Image processing and analysis
  • Quantitative description of the structure
  • Anisotropy and orientation
  • Interconnected network and strut analysis
• Future objectives
• Conclusions
Advanced polymeric foams

Advantageous properties

• Low mass density (about 60 kg/m^3)
• Superior thermal and acoustic insulation
• Excellent strength/weight ratio

Wide range of applications

• Refrigerated boxes in transportation industry
• Lightweight packaging solutions
• Core in sandwich panels for aircraft structures
• Furniture and building construction industries
The characteristics of a foam are defined by both the polymer used and its cell structures.
Quantitative description of the structure of foam

- Equivalent cell diameter distribution, volume distribution and average cells per mm$^3$;
- Sphericity (roundness) distribution of the cells;
- Anysotropy and orientation of the cells;
- Average thickness of the cell walls;
- Mean strut’s thickness and length;
- Node’s connectivity;
- …
**Structural investigation of foams**

**Optical or Electron Microscopy**
- External surface or one cross section (thin slice);
- Conductive coating/variable pressure in the case of electron imaging;
- Thin walls of the cells resolved in closed-cell foams

**X-Ray CT**
- Non destructive 3D analysis/virtual cross sections through all the sample;
- No special sample preparation required;
- Thin walls of the cells detected but not resolved
Structural investigation of foams

Image of polymeric closed-cell foam via confocal microscopy

Virtual section (slice) of reconstructed polymeric foam of the same type via X-ray CT
Case study 1

Rigid high performance polyurethane foam used for cryogenic thermal insulation systems in the Aerospace sector.
CT setup for acquisition and reconstruction

X-ray parameters (GE Phoenix Nanotom s)

- Target suitable for weak absorbing specimens (Mo)
- Tube voltage and current so as to increase the image contrast (50 kV and 180 μA)
- Detector timing and number of averaged images for each saved image so as to improve the signal to noise ratio (0.750 sec and 5 images)

Number of projections

- Greater than X-ray image width measured in pixel so as to improve sharpness
CT setup for acquisition and reconstruction

Geometric parameters

• The geometric magnification is determined by the focus-detector distance (FDD) and the focus-object distance (FOD): \( M = \frac{\text{FDD}}{\text{FOD}} \).

• The minimum magnification required to resolve a feature depends on the object contrast and on the ability of the detection system to transfer signals from object to recorded image, i.e. its modulation transfer function (MTF).
CT setup for acquisition and reconstruction

Use of the MTF to design the analysis

Size of feature: 39 μm (estimated)
sub-structure $s = 13$ μm

$M = \frac{\lambda}{s} = 11$

$\lambda = 146 \mu m$
Segmentation. Image processing and analysis

1. Denoising of grey-scale images
2. Segmentation of grey-scale images
3. Objects separation (first phase: distance transformation)
Segmentation. Image processing and analysis

4. Objects separation (second phase: watershed transformation)
5. Labeling
Segmentation. Image processing and analysis

3D reconstruction of the foam’s structure

3D reconstruction of the cells

3D cross section
Quantitative description of the structure

<table>
<thead>
<tr>
<th>Measure</th>
<th>Pore Volume (mm³)</th>
<th>Equivalent Diameter (mm)</th>
<th>Pore Sphericity</th>
<th>Anisotropy</th>
</tr>
</thead>
<tbody>
<tr>
<td>min</td>
<td>0.0003</td>
<td>0.088</td>
<td>0.652</td>
<td>0.20</td>
</tr>
<tr>
<td>max</td>
<td>0.147</td>
<td>0.656</td>
<td>0.953</td>
<td>0.92</td>
</tr>
<tr>
<td>mean</td>
<td>0.007</td>
<td>0.222</td>
<td>0.874</td>
<td>0.57</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>0.007</td>
<td>0.058</td>
<td>0.034</td>
<td>0.10</td>
</tr>
</tbody>
</table>
Quantitative description of the structure

• Equivalent diameter
For a given particle, the equivalent diameter measure computes the diameter of the spherical particle of the same volume

\[ EqD = \left( \frac{6V}{\pi} \right)^{1/3} \]

• Sphericity
The sphericity is a measure of how spherical an object is

\[ S = \frac{4\pi(EqD / 2)^2}{A} = \frac{\pi^{1/3}(6V)^{2/3}}{A} \]

Ex. octohedron \( S = 0.846 \), dodecahedron \( S = 0.910 \), icosahedron \( S = 0.939 \)
Analytical aliquot is a representative sample?

Small cube S
Statistical sample of 2106 cells
(excluding border cells)

Large cube L
8 times greater than the small one
(different voxel size)

The difference between L and S is equal to that one among any of the eight small cubes
Quantitative description of the structure

Filter by measuring

Image of the first 50 pores having highest equivalent diameter, taken from the Z axis
Quantitative description of the structure

Filter by measuring

Image of the first 50 pores having highest equivalent diameter taken from the X axis
Quantitative description of the structure

Filter by measuring

Image of the first 50 pores having highest equivalent diameter taken from the Y axis
Anisotropy and orientation

Anisotropy of a cell

The anisotropy is investigated by replacing the cells by equivalent ellipsoids. 3D inertia matrix of the equivalent ellipsoid is determined for each cell. The eigenvalues $\lambda$ of this matrix are computed for each cell. Then, the anisotropy is defined as

$$An = 1 - \left( \frac{\lambda_{\text{min}}}{\lambda_{\text{max}}} \right)$$
Anisotropy and orientation

The arrows represent the eigenvectors associated to the maximum eigenvalue for each cell.

This graph shows the orientation of the cells with anisotropy greater than 0.7, superimposed on the volume rendering of the cells using blending technique.

It was found that cells are more elongated in one direction.
Anisotropy and orientation

The eigenvector coordinates in the three orthogonal planes of the laboratory frame confirm that the cells have a preferential direction.
Anisotropy and orientation
Anisotropy and orientation
Interconnected network

Case study 2

Flexible polyurethane foam used in Mattress and living sector.
Interconnected network

3D reconstruction of the foam’s structure

3D reconstruction of the cells

3D cross section
Interconnected network

Skeletonization

Thinning methods

Skeleton: Locus of points equidistant from the boundary
Interconnected network

Binary image → Distance map → Skeleton

Average strut thickness = 250 \( \mu \text{m} \)
Classification of skeleton voxels

- **End-point voxels**
  If they have less than 2 neighbours.

- **Junction voxels**
  If they have more than 2 neighbours.

- **Ligament voxels**
  If they have exactly 2 neighbours.

Voxel skeleton

↓

Spatial graph

↓

Further strut analysis
Interconnected network

<table>
<thead>
<tr>
<th>Measure</th>
<th>Struts Length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>min</td>
<td>0.0219</td>
</tr>
<tr>
<td>max</td>
<td>1.076</td>
</tr>
<tr>
<td>mean</td>
<td>0.261</td>
</tr>
<tr>
<td>Std. Deviation</td>
<td>0.338</td>
</tr>
</tbody>
</table>
Future objectives

- Perform quantitative description of the structure in conjunction with in-situ mechanical testing to evaluate how the properties of foams change under different load conditions.
- Give to the industrial laboratory a sufficient feedback to manufacture foams having controlled physical properties along a given direction.
Conclusions

• Micro-CT is very useful to get high resolution 3D information about the various foams.
• 3D image processing methods applied to CT data allow the complete characterization of foam structure.
• Structural analysis of foams allows a better understanding of the correlation between the structure and physical properties of foams.
• Structural analysis of foams enables polymer processing companies to optimise their products and production processes.
• Quantitative description of the structure in conjunction with in-situ compression stages could provide an immediate interpretation of how the properties of foams change under different loading.
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Anisotropy and orientation

Open-cell polyurethane foam showing an almost isotropic behaviour, with low values of anisotropy and no preferential direction.
Anisotropy and orientation

The eigenvector coordinates in the three orthogonal planes of the laboratory frame confirm that the cells have an almost isotropic behaviour.