Investigation of damage development of polymer foams using interrupted in-situ X-ray tomography

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Abstract
Thermoplastic foam materials were investigated by interrupted in-situ X-ray computed tomography. Scan setup and image processing procedure were optimized for optimal quantification results. High resolution laboratory CT was applied at varying compressive load. Qualitative image analysis showed the beginning of cell collapse in the core of the sheets. The reason could be found in the variation of cell size over sheet thickness. Smaller cells that form thinner struts were located predominantly in the core. The expected trend of reduced cell size and reduced sphericity at increased load was observed.

1. Introduction
Polymeric foams are used intensively for a widespread of different applications. In order to extend the range of applications further, especially for novel polymeric materials, knowledge about microstructure and change in microstructure during mechanical loading is needed.
X-ray computed tomography (CT) has already been used for the characterisation of pores in polymer foams. Synchrotron radiation tomography has been applied together with stepwise compression testing (1). The materials contained hollow spheres for which damage mechanisms were studied. Polypropylene foam was studied also with Synchrotron radiation tomography (2). On the mesoscopic scale, 3D image processing was applied to generate models of established beads. Microstructural pores were not considered in this work.

This contribution uses laboratory CT to characterise cell structures of thermoplastic foams while interrupted compression testing. The mechanisms of damage formation and evolution are studied.

2. Materials and methods
The investigated specimens were punched out of 3 mm thick injection moulded sheets of polypropylene and had cylindrical shape. The finally foamed material shows compact layers on top and bottom and a closed cell foam structure in the core of the sheets.
For mechanical loading the specimens were put on modified clamps of a compression testing machine that was placed onto the CT turntable. Clamp modification allowed for scanning a bigger part of the specimen by reducing cone beam artefacts that are present due to the metallic clamps.

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From a first compression test without stopping the experiment, the positions on the force-extension curve were defined for interrupting and scanning. Figure 1 shows the curve of the interrupted compression test. After the desired force was reached, a delay time was applied to prevent relaxation during CT scanning which might lead to motion artefacts. The resolution needed for microstructural characterisation is depending on the cell size and the cell wall thickness. For the investigated materials a voxel edge length of 2.5 µm was reasonable. This lead to a maximum possible specimen diameter of 4 mm. CT scans were performed at a laboratory CT device.

![Figure 1: Force-extension curve showing the points of interruption for CT scanning](image)

Pore segmentation was done using MAVI (Fraunhofer ITWM, Kaiserslautern) (3, 4). After pre-processing in order to reduce noise, pores are separated from material by global thresholding. Pores that are still falsely connected are separated using a watershed approach. The steps that are described in (5) were applied in a similar way to these data. Image processing of deformed cells required adapted pre-processing and parameter optimization compared to the undamaged state.

The shape factor was calculated using this formula:

$$ s = 6 \frac{\sqrt{\pi} V}{S^{3/2}} $$

### 3. Results

A first impression about damage mechanisms can be received by looking at the slice images. Figure 2 (left) shows collapsing of cells which is pronounced in the centre of the sheet and can be detected at 93 N. At 70 N and below the cells are only plastically deformed. The lateral view also shows bright triangular shadows that are generated by so called cone beam artefacts. These are introduced by the metallic clamps and were reduced by a reduction of clamp diameter.

A detailed, zoomed view shown in Figure 3 illustrates the mechanism of cell collapse which is rather the collapse of cell walls in regions where pores are located close
together. In these regions the material forms struts that are the weakest structure within the foam material and collapse first for this reason.

Figure 2: left: lateral CT slice images at increasing force. Right: Slice images with segmentation result for 0 N.

Figure 3: Zoomed CT slice images of un-deformed (left) and deformed condition at 105 N (right)

The quantification of cell size distributions in dependence of load is shown in Figure 4.
4. Conclusions

The experiments conducted were optimized in multiple ways to adjust mechanical parameters like clamp design, scan parameters like resolution and parameters needed for image processing. The final pipeline allowed for the quantitative description of foam microstructure at different compression load levels. Relatively high resolution was needed to separate the pores correctly although pore diameters were in the range of 100 µm. The smallest structures that are important for cell separation are the struts which were very small at some regions. Compressing the pores leads also to small structures as the pores are flattened and in the end completely compressed.

The investigated foam specimens showed a variation in pore size across sheet thickness as seen in Figure 2. Bigger pores are located near the surface, smaller in the core. The expected trend of reduced cell size at increased load was observed. Changes in shape starting from rather spherical cells to elongated and flattened structures were quantitatively described.

The qualitative analysis showed collapsing of cell walls especially in the centre of the sheets where the cells tend to be smaller and struts thinner.

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References