Non-destructive imaging analysis of unstable foam structures using 3D X-ray computed tomography

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ABSTRACT

For the engineering of foamed products, knowledge about the foam structure as well as about its dynamics and stability are of critical importance. Using X-ray computed tomography (CT) accurate 3D information about the foams structure is obtained, but the structural characterization of liquid foams, which feature structural changes, is still challanging.

In this contribution a fast laboratory CT setup is presented allowing the acquisition of 3D data sets almost instantaneously.

Reached limits for the CT system:

- 15 s measurement time for a complete 3D recording
- 3.5 µm resolution for small objects (I.D. 10 mm)
- time-resolved structure characterization
- constant climate conditions (temperature, humidity)
- non-destructiv

The suitability and possibilities of such a system for non-solidified foams and other porous materials are shown.

Keywords: foam, computed tomography, fast CT, X-ray imaging,

1 INTRODUCTION

Foamed products play an essential role in modern food industry. The contained air changes the physical properties and influences product attributes. Their fluffy structure fulfils the consumer's desire for light fare and has a special impact on mouth feel and flavour perception. However, the characterization of sensitive porous structures is difficult, because of the foams sensitivity against mechanical influences. Still, appropriate quantitative methods are not readily available today [1].

X-ray micro computed tomography (µCT) offers a non-destructive 3D imaging technique with a resolution of few micrometres [2]. From the 3D data the microstructure can be measured directly to analyse the air fraction of the product regarding air volume and bubble size distribution, accurately and with an extensive set of statistics [3]. µCT is suited well for foam investigation, because it is based on the different absorption of X-rays through various materials. Foams have a great amount of air, which don't absorb X-rays, and the continuous phase, which absorb X-rays depending on the used material. This resulted in a high image contrast between air and foaming material in the received images.

A challenge for CT is non-solidified foam, because of the time dependency. The recording time for a normal µCT scan is between 10 and 100 min, during this time the object should not change, but non-solidified foams change their structure within few seconds [3]. Therefore we optimized a laboratory CT system concerning recording speed and image quality to realize CT scans within a few seconds, allowing for dynamic, time-resolved measurements. Structure changes and foam development can be studied in place without interference.

2 MATERIAL AND METHODS

2.1 Foam

For the laboratory X-ray µCT scans a β-lactoglobulin (BLG) protein-foam was chosen for its good foaming properties. It is a model system for the investigation of unstable foams. The foam is produced on site seconds before the experiment by a foaming apparatus.

BLG powder was dissolved in deionized water to yield a protein concentration of 0.25 % (w/w). For the present study the pH of the solution was set to 5.2 by adding 1 M hydrochloric acid (HCl). The temperature of the solutions was kept constant around 20 °C.

For the foaming process, a DFA100 Dynamic Foam Analyzer unit from Krüss GmbH Germany was used. The system is based on the aeration method introduced by Waniska and Kinsella [4]. Afterwards the pillar with the foam is taken out of the apparatus and directly placed in the CT system.

2.2 Computed tomography

For this foaming experiment we employed a µ-CT setup at the Fraunhofer Development Center X-ray Technology (EZRT) which has already been described by Zabler et al. [5]. The µCT setup was used with a Varian PaxScan 2520D/CL detector, which is a Si-TFT panel and operates at a maximum frame rate of 10 images s^{-1} in 14-bits full frame mode (1920 × 1536 pixels) and 30 images s^{-1} in 2 × 2 binning mode. The tube was operated in 'high-power' mode at 60 kV acceleration voltage and 240 µA tube current (approx. 14.4 W power) allowing for exposure times as short as 34 ms.

The foam was generated in a 20 mm wide plexiglass column and images were recorded within 30 s at three time points: 3 min, 5 min and 7 min after the initial foaming process. The column was scanned with continuous sample rotation (fly-by) over 200°. The foam structure was thereby recorded with an effective voxel sampling of 21 µm and a spatial resolution of approx. 43 µm. Depending on the rotation speed, CT volumes can be recorded in scan times down to 15 s for a volume of 512^3 voxels (400 projection images).

2.3 Data processing and analysis

Prior to tomographic reconstruction the radiographic images were filtered with a Median kernel. The radiographs were then processed by a filtered back-projection (FBP). Figure 2a shows a typical slice from the CT-volume data.
The structural analysis of the foam is detailed in Figure 2. First the volume images are binarized by applying an Otsu threshold (see Figure 2b) [6]. This method avoids the erroneous selection of non-foam particles during the first step of the binarization. For retrieving the complete foam borders, the resulting binary 3D images are segmented using a Euclidean distance transformation (see Figure 2c) and an adaptive water-shedding algorithm (see Figure 2d) from the software ToolIP (Tool for Image Processing, Fraunhofer ITWM, Kaiserslautern, Germany). Single bubbles down to 54 µm diameter are thus segmented, labelled and analyzed in terms of position, size and volume. Based on this data histometric bubble size distributions can be calculated.

For the CT series of further foam studies published by Eggert et al. [3] individual growth and shrinkage of a selected bubble cluster was analyzed, which coalesced at the latest time point into one single bubble. Starting from the latter a 3D mask was created based on this the coalesced bubbles were identified in the previous time step, and so on until the initial bubble ensemble was determined at the first time step. Prior to this 3D masking-and-finding procedure it was necessary to register all datasets in polar coordinates so that central coordinates of corresponding bubbles would match. This registration is performed manually using the software ImageJ.

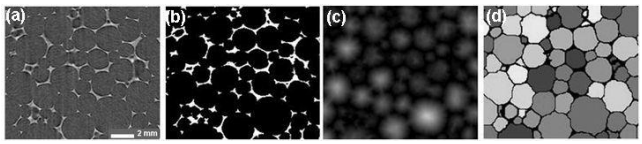

Figure 2: Pivotal steps in the image processing chain shown for the central slice: (a) binarization of the original image, (b) the binary image serves as input for the distance transformation and (c) the watershed image masked with (a) to obtain the resulting label image (d). [3]

2.4 Climatic chamber

The climatic chamber, schematically shown in Figure 3, guarantees temperature and humidity of microstructures during radiographical investigation. The specimen is placed into an EPS foam cup (max. volume: 250 ml) to allow transmission of the X-rays. Cup base is cut off and watertight fastened to a copperplate. An experimentally determined amount of water is pipetted into the cup in order to ensure certain humidity. Control of temperature is possible through a regulated TEC1-12706 Peltier element situated between copperplate and cooling element. The 127 couples 40 x 40 mm size module is supplied by a DC voltage using the NTS 150 W multi USB EuP 8.5A power supply (Goobay, Germany) and linked to a QC-PC-CO-CH1 controller (Quick-Ohm Küpper & Co. GmbH, Germany). The temperature can be varied within the range of -40 to 100°C. A NTC 10 Ω (β=3977K) temperature sensor, that is linked to the controller, is situated at the height of the specimen to provide the feedback signal for the temperature control.

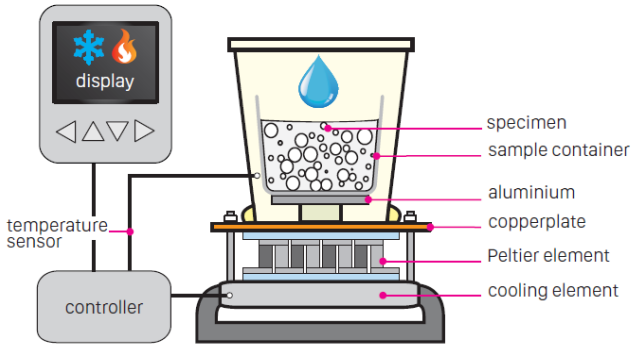

Figure 3: Schematic setup of the cooling/heating chamber with certain humidity.

3 RESULTS AND DISCUSSION

The aim of the work presented here was to generate a comprehensive knowledge of foam forming and stabilizing using non-destructive, fast and high-resolution techniques.

3.1 Foaming

As a first step, a method for the gentle production of foams had to be found, which allowed an in-situ characterization of the aerated structures.

Therefor the DFA100 Dynamic Foam Analyzer unit from Krüss GmbH Germany was used. The system is based on the aeration method introduced by Waniska and Kinsella [4]. The foam is produced on site seconds before the experiment. Afterwards the pillar with the foam is taken out of the apparatus and directly placed in the CT system, see Figure 1.

3.2 CT-system "Fly-by"

To describe foam decay bubble size distributions and further bubble properties were examined using 3D-µCT. Therefore we improved a CT-system concerning recording speed and image acquisition suited for unstable foams.

The measurement time for standard CT recordings is about 10 min at best. This is far too long for unstable foams, which constantly change their structure. The movement of the foam causes image artefacts, that's why the recording time has to be reduced. A standard CT-system works after stop-and-go principle, meaning that a projection is recorded and saved for each angle step. In fact that's quit time consuming.

Using the fly-by method the CT-recording is restricted to the essential steps only. The image acquisition occurs "on the fly" and the image seqeuence is processed afterwards for 3D reconstruction. With this method the recording time is only linked to the rotation speed of the specimen during the CT-scan. An important condition for fly-by is a fast detector with a frame rate above 15 images s^{-1}. For a further speed up the CT-scan can be cut down from a whole 360° rotation to a limited angel of about 180°. The recording time can thus be reduced by factor two down to 15 s at all. This allows for new and detailed insights into the dynamics of instable foams, i.e. to assess foam properties during foam decay.

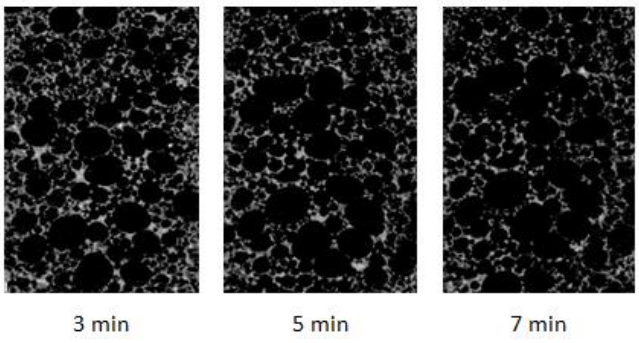

Figure 4: Sequence of binary axial CT slices of BLG foam at pH 5.2 after 3, 5 and 7 min.

3.3 Foam analysis

For foam analysis during foam decay the example of BLGs foaming behavior is used here. Figure 4 shows an image sequence of BLG foam recorded with fly-by CT in 15 s.

The time development of the BLG foam is regarded at the isoelectric point (pH 5.2). The image sequence in Figure 4 shows a fast structure change at the beginning of the recording. After 5 min the shapes in figure 4 seem to have stabilized. In the next minutes only slight changes in the microstructure can be observed. The foam moves up in the pillar because of the drainage collects below and pushes the foam up. The liquid loss because of drainage can also be investigated by the decreasing thickness of the lamellas between the foam cells. The borders between the foam cells become less visible, because the thinner lamellas absorb less X-rays.

In Figure 5 the bubble size distribution is shown to visualize the foams change over time. The bubble size distribution shifts in the area of bigger bubble diameters with the time. The number of bubbles decreases, especially the number of small bubbles reduces strongly. The mean diameter rises from 469 to 549 µm accordingly.

3.4 Foam development

Furthermore, an image processing chain was created and subsequently automated using the software ToolIP (Tool for Image Processing) from the Fraunhofer Institute ITWM. Thus, a quantitative analysis of the microstructure even of instable foams was possible, providing time-resolved information about bubble size, diameter, shape and position. The obtained results were supplemented by an additional series of measurements at the European Synchrotron Radiation Facility (ESRF) in Grenoble, which

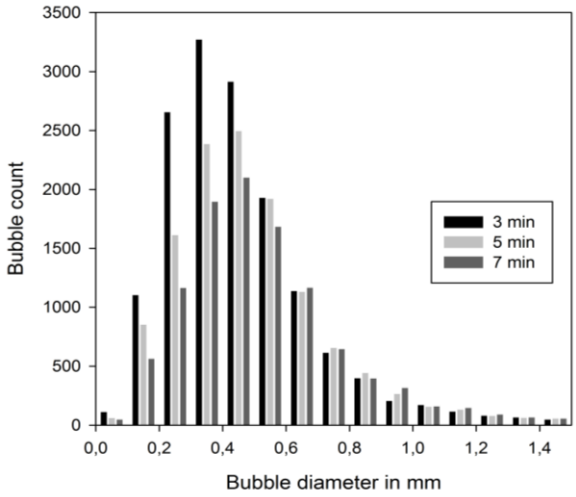

Figure 5: Bubble size distribution of BLG foam at pH 5.2 after 3, 5 and 7 min.

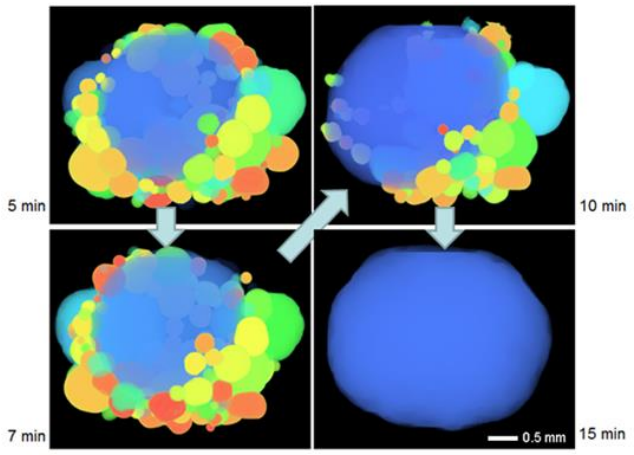

Figure 6: Development of a foam cluster over 15 min [3].

allowed the quantitative description of phenomenological characterized destabilization mechanisms like coalescence and disproportionation presented by Eggert et al. [3]. Thereby bubble growth, shrinkage and fusion can be observed or followed (see Figure 6).

The results of these investigations can be used for developing new micro structured food products and for a better understanding of the relation between the production and the structure of foamed material.

3.5 Temperature and humidity

The climatic chamber has been designed for controlling temperature and humidity of microstructures during radiographical investigation to guarantee a native investigation. The climatic chamber is placed within the X-ray beam and images of microstructures are recorded in a temperature and humidity controlled environment, thus long term X-ray scans of sensitive materials are possible (see Figure 7).

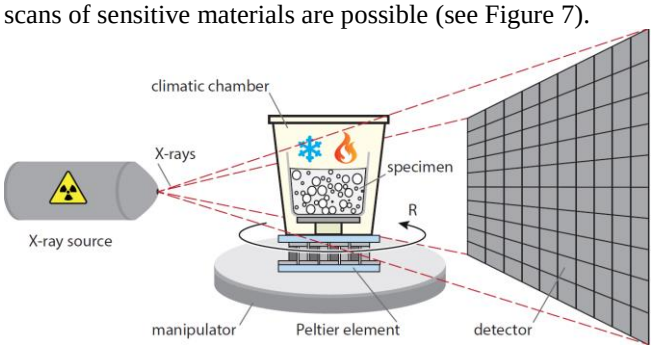

Figure 7: Schematic measurement setup with climatic chamber between X-ray source and detector.

4 CONCLUSION

In this contribution a laboratory µCT setup was presented allowing the acquisition of 3D data sets in down to 15 s. The suitability and possibilities of such a system for non-solidified foams was shown.

Reached limits for the CT system:

- 15 s measurement time for a complete 3D recording
- 3.5 µm resolution for small objects (I.D. 10 mm)
- time-resolved structure characterization
- constant climate conditions (temperature, humidity)
- non-destructive

From the 3D data the microstructure of the foam can be measured directly to analyze the air fraction of the product regarding air volume and bubble size distribution, accurately and with an extensive set of statistics. Apart from the foams structure, its dynamic changes over time can be studied in place without interference. Thereby bubble growth, shrinkage and fusion can be observed or followed.

The results of these investigations can be used for developing new micro structured food products and for a better understanding of the relation between the production and the structure of foamed material.

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