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Mac Intyre, Jonatan; Raka, Doruntina; Aydin, Mehmet; Viitanen, Leevi; Koivisto, Juha Measuring biofoam anisotropy using optical coherence tomography

Published in: Journal of Materials Science

DOI: 10.1007/s10853-022-07297-3

Published: 27/05/2022

Document Version Publisher's PDF, also known as Version of record

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Please cite the original version:

Mac Intyre, J., Raka, D., Aydin, M., Viitanen, L., & Koivisto, J. (2022). Measuring biofoam anisotropy using optical coherence tomography. *Journal of Materials Science*, *57*(25), 11663-11672. https://doi.org/10.1007/s10853-022-07297-3

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Composites & nanocomposites



Measuring biofoam anisotropy using optical coherence tomography

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Received: 21 April 2022 Accepted: 30 April 2022

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ABSTRACT

Optical coherence tomography enables quick scans of translucent objects in a simple environment. Here, we apply this technique to wood-based biofoam. We measure the geometrical properties of the foam, such as bubble eccentricity and density fluctuations, in addition to characterising the possible orientation of fibres. We find that the wood-based foams are extremely suitable for optical coherence tomography due to their translucent nature and large changes of optical density between air-filled bubbles and solid films. Measurement of bubble eccentricity revealed a reasonably high aspect ratio of 1:2, enabling the orientation of long cellulose fibres if added to the mixture. The results demonstrate an effective method to characterise foamlike metamaterials. Furthermore, focusing on eccentricity enables the adjustment of the foam's manufacturing method and, in turn, helps to produce anisotropic structures.

Handling Editor: Maude Jimenez.

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GRAPHICAL ABSTRACT



Introduction

Liquid foams consist of gas bubbles that are separated from each other by thin liquid films. The unique properties of foams find a wide range of purposes in nature and industry [1, 2]. Foam forming technology enables manufacturing of new products to replace plastics with biodegradable materials [3–6], the delivery of drugs [7] and thermal insulators [8] among others.

The ultimate question for the designer is how to select the best bubble distribution for a specific structure [9]. Therefore, in order to understand the internal foam structure, different techniques have been used to characterise them such as scanning electron microscopy (SEM), light microscopy (LM) and three-dimensional reconstruction approach using X-ray microcomputed tomography [10, 11]. In this article, we use optical coherence tomography (OCT), a non-invasive optical imaging technique that can provide an internal description of the material.

Optical coherence tomography (OCT) is a method based on low-coherence interferometry for depth-resolved imaging within turbid media. It has rapidly evolved in recent years, making it applicable to a large number of fields [12]. OCT is well known for its high depth and transversal resolution (decoupled from each other) and high probing depth in scattering media, along with its contact-free and noninvasive operation. It uses scattered light, from which it derives high resolution, cross-sectional imaging for non-invasive investigation using the Fourier diffraction projection theorem [12].

From the material science perspective, optimising and verifying the unit cell properties on a microscale create the desired properties on a macroscale [13]. Creating orientated rod-shaped structures, for example by elongating bubbles, increases the compression strength in one direction while reducing it in the cross-direction. This can be achieved in the laboratory scale by freeze casting increasing compression strength in the direction of the bubbles' major axes by nearly fourfold compared to the bubbles' minor axes [14, 15]. Following this idea, we have made a bio-based porous materials (Fig. 1) by introducing anisotropy by weighting its internal structure in a particular direction [3].

The internal structure of similar porous materials has been explored using different techniques [11, 16]. In general, the 2D methods such as SEM, light microscopy or direct CCD imaging yield similar results as X-ray microtomography, but the former is limited compared to 3D methods [17]. We will show that OCT is a suitable technique for our material due to its translucent properties.

Novel metamaterials are obtained by controlling the local bending and compression via local anisotropy. The simplest example of this is auxetic foams [18] with a negative poisson ratio. A more



Figure 1 a Top view of a single rod. The major axis is set on the y-axis. b Photomicrograph of the rod. Bubbles appear to be elongated along the major axis of the rod.

complicated example of how local anisotropy can be used is compliant mechanisms. These foamlike structures can have functionalities such as bistable switches [19] and, for example, hollow tubular structures, such as foams with elongated bubbles, which can bear enormous stresses [20].

Foam-based materials are the future solution for energy efficient materials due to their lightweight and insulation properties, similar to aerogels [15]. In addition, the reduction of materials in flow state [21] and solid state [22, 23] reduces waste, addressing the demand for eco-friendly materials.

The resolution of 1 μ m and imaging volume of 1 mm³ hits the sweet spot of current mass production of modern insulation and packaging materials. OCT is suitable for investigating the characteristic length, bubble size, grain size and fibre size. In this article, we use OCT to extract the 3D location and shape of the bubbles of our in-house made bio-based solid foam material. Using this technique, we characterise the anisotropic structures found in foam created by a simple industrially scalable process, using a proprietary cellulose-based FoamWood solution.

Materials and methods

A foam forming method was utilised to create the rods [3]. The material used is an aqueous solution of a proprietary FoamWood mixture containing fibres, cellulose and its derivatives, with a solids content of 15%.

Figure 2 shows the set-up used to create the foam rods. The foam is deposited onto a solid surface from a nozzle. The final product was pushed from the foaming device, which consists of a sealed cup and a spinning blade, onto the solid plate using a pressure controller (pressure $\Delta P = 0.1$ Pa). As the foam is deposited, the solid plate moves linearly, creating a rod of l = 150 mm in length and w = 6 mm in width. The hot plate moves at a speed of v = 1 mm s⁻¹. The process is repeated until 5 rods are deposited onto the plate in succession. The rods are dried using a radiant heater.

The rods were then scanned using an optical coherence tomography system, GAN620 from Thorlabs[®], which uses a laser with a wavelength of 900 nm. Figure 3a shows the device together with a sample of foam. The OCT system consists of one fixed and one moving mirror. By adjusting the position of the latter, an interference pattern is observed when the difference in optical path length between the two mirrors equals the difference in path length from the



Figure 2 a The schematic view of the set-up shows how the white foam is driven onto the hot plate from the foamer device via controlled air pressure. The linear stage moves as the foam is extruded, creating a solid, rod-shaped object that is dried by the radiant heater. **b** A picture of the measurement device showing the rod creation process.





Figure 3 a In OCT systems, light is split into a reference arm that consists of mirrors and a sample arm. The light reflected from the sample and the mirror system recombine into the camera. The time dependent interference signal between the two beams determines

the optical density of the sample in 3D. **b** Side view of a single rod. The scanned volume is marked with a green box. **c** Rendering figure of a 3D block of foam. The walls of the bubbles are shown as dark blue.

individual parts of the sample. The OCT used here is capable of measuring a depth of 1.4 mm in water, with a horizontal (*x*, *y*) resolution of $\Delta x = 2.2 \,\mu$ m and $\Delta z = 1.64 \,\mu$ m in depth direction. Consequently, this allows us to scan the internal structure of the samples with high resolution.

Multiple rods were scanned along a square cuboid of maximum size $x \times y \times z = 10 \times 16 \times 2.01 \text{ mm}^3$. Figure 3b shows a single rod, where the green rectangle represents the area that was scanned. The reconstructed signal of that area is shown in Fig. 3c, where the major axis of the rod is along the *y*-axis and the *z*-axis is parallel to the direction of the laser beam. Once the sample is scanned, we proceed to detect and characterise the bubbles contained within the rod, which are seen as holes in Fig. 3c.

Results and discussion

Figure 4a shows an xz cross section of a rod, where the solid material appears in the white level, and the air around and inside the foam appears in the black level. The OCT is capable of scanning the entire thickness of the rod. Each surface point of the rod (x, y) provides a reflective signal from the material. As an example, Fig. 4b shows the signal captured along the blue line in Fig. 4a. Sharp refractive index variations between layers in the sample correspond to the intensity peaks in the interference pattern. The dashed black line indicates the mean air intensity, whose value is used as a threshold T. Above this value, the signal depicts the interaction with the films of the foam. Figure 4c shows the main steps for the image processing.

The raw data were analysed using MATLAB and Python softwares. For each surface point (*x*, *y*), a Gaussian filter of standard deviation γ , with a value range of 0.4 < γ < 0.75, is applied.

Once the noise is attenuated, the signal is re-scaled using the threshold value T given by the intensity of the surrounding air (dashed black line in Fig. 4b) to separate the solid phase from the porous phase. Every intensity value below the threshold I < T is converted to zero, while I > T to ones. We then extract the 26-connected voxels (3D pixel) from these binarized data, and each collection of neighbouring voxels is labelled as one bubble. The 26-connected voxels are neighbours to every voxel that touches one of their faces, edges or corners. The entire image processing is depicted in (Fig. 4c). Figure 5 shows a rendering of the rod with films in blue and the biggest bubbles detected in orange, confirming that the detection algorithm does detect bubbles. Here, to properly visualise the bubbles, we only show the bottom part of the rod.

Each bubble is labelled and its three dimensions measured. In order to determine the dimensions, we calculate the first and second central moments: the first is the centroid of the bubble \vec{r} , and the second is



Figure 4 a 2D scan of the foam: solid material appears in the white level and the air in the black level. b Intensity of the backscattered light versus depth, an example of a signal captured



Figure 5 Reconstruction of the OCT signal: the bottom part of the rod is in blue and the biggest bubbles detected in orange.

its variance σ_i^2 for i = (x, y, z). We assign each length of the bubble as σ_i , which represents the expanse of the voxel group. The bubbles' orientations are determined by the angles α_i for i = (x, y, z). To compute them, the major eigenvector of every bubble is projected to each ordered pair.

Table 1 shows all the scanned volumes and the number of detected bubbles. There is no relation between the number of detected bubbles and the size of the sample. This is mainly related to the threshold T, which certainly depends on the structure of each sample.

by the OCT along the blue line in Fig. 4a. The mean value of the air is marked with a dashed black line, which corresponds to the threshold T. **c** A flow chart for image processing.

Bubble size is restricted to a range $V_{\min} < V < V_{\max}$: while the lower limit neglects clusters of only a few points, the upper limit removes the surrounding air. Using these limits, the number of bubbles for each sample is listed in Table 1. The average bubble size confirms that the bubbles are indeed elongated in a particular dimension.

To evaluate the degree of elongation, we calculate the length of the bubbles along the *x*, *y* and *z* directions. Figure 6a shows the standard deviation of *y*coordinates, σ_y , of the bubbles as a function of the standard deviation of *x*-coordinates, σ_x . As we can see, the bubbles are elongated along the main axis of the rod (the *y*-axis) given that most of the points are above the black line and σ_y deviates from the relation $\sigma_x = \sigma_y$, which represents bubbles of circular cross section. Moreover, Fig. 6b reveals that the bubbles are mainly flat.

The fibre networks and bubble size distributions in mixing are characterised by a log-normal distribution [24]. To improve their description, each distribution of bubble dimensions was fitted by a log-normal distribution with coefficient *A*:

$$f(\sigma_i) = A \operatorname{lognorm}(\sigma_i; \tilde{\mu}, \tilde{\rho}) , \qquad (1)$$



Table 1 Rod size, number and average bubble size. The size is $x \times y \times z$ and the main axis of the rod is on *y*-axis

Sample label	Sample size [mm ³]	Number of bubbles	Average bubble size [mm ³]
S1	$10.0 \times 10.0 \times 1.68$	396	$0.039 \times 0.034 \times 0.0036$
S2	6.0 imes 14.0 imes 1.68	1198	$0.032\times0.039\times0.0033$
S3	6.0 imes 14.0 imes 1.68	419	$0.029\times 0.034\times 0.0026$
S4	6.0 imes 14.0 imes 1.68	1301	$0.032\times0.034\times0.0025$
S5	8.0 imes 5.0 imes 2.01	1649	0.021 imes 0.061 imes 0.0057
S 6	3.0 imes 4.44 imes 1.0	145	$0.021\times 0.032\times 0.0032$
S7	3.0 imes 4.0 imes 1.0	102	$0.020\times0.028\times0.0027$
S8	$3.0\times 4.0\times 1.0$	105	$0.021\times 0.031\times 0.0034$



Figure 6 a Bubble dimensions σ_x versus σ_y for all the samples in Table 1. Black dashed line represents $\sigma_x = \sigma_y$. The relation $\sigma_y > \sigma_x$ implies that the bubbles are elongated along the *y*-axis. **b** Bubble dimensions σ_z vs. σ_y for all the samples in Table 1. Dashed black line represents $\sigma_z = \sigma_y$ and solid black line is $\sigma_y = 8.25\sigma_z$. The relation $\sigma_y > \sigma_z$ implies that the bubbles are flat.

 $\tilde{\mu}$ and $\tilde{\rho}$ being fitting parameters that describe the mean and standard deviation, respectively. Figure 7 shows the distribution regarding each axis. The excellent fit supports the fact that the deposition of the material on the plate follows a random process. We note that the intensity of the peaks is always higher and closer to zero for σ_x , which means it is more likely to obtain narrower bubbles.

Lastly, to confirm that the major axes of the bubbles are in the main direction of the rod, we calculate the eigenvectors and -values of each bubble. Using the major eigenvectors \vec{v} , we are able to determine the principal axes of the bubbles. The angle α , the major eigenvector formed with each ordered pair, represents the orientation of the bubble. It is defined as

$$\alpha_i = 90 - \operatorname{acos}(\vec{v} \cdot \hat{n}_i) , \qquad (2)$$

where \hat{n}_i is the normal vector of each ordered plane *i*. Figure 8 shows the distribution of the angles for each scanned sample. The angles, α_y , between the vectors and the *yz*-plane centre around 0°. Therefore, the major axes of the bubbles run mostly parallel to the *y*-axis, which is also the major axis of the rod, as previously mentioned.

OCT proofs to be an suitable technique for explore the internal structure for bio-based solid foams. In comparison with X-ray tomography, OCT is able to detect bubble (pore) size for similar materials [25]. The bubble orientation caused by foamer device can be also detected under the OCT [26]. Consequently, the internal structure studied here lead to significantly excellent macroscale properties of the biobased foam [3].



Figure 7 Bubble dimension distribution (probability density functions, PDFs) for three different scans: a σ_x and b σ_y .



Figure 8 Distribution of the bubble orientation for each sample in Table 1. The bubbles run along the major axis of the rod which is represented by the peak around 0° .

Conclusions

The foam forming method described was used to create long rods made of fibres, cellulose and its derivatives. The resulting material contains a high number of bubbles, whose shape and orientation was studied using optical coherence tomography (OCT).

The optical properties of our bio-based foam allows us to explore its internal structure. By filtering the OCT signal, we were able to detect multiple spots (air pockets) inside the sample. We have shown that the bubbles are mainly longer in the main axis of the rod, which is attributed to the shear introduced during the manufacturing process as well as the foam forming and drying method. In addition, the short height of the bubbles allows them to be represented as flat ellipsoids, as shown in Fig. 9a. Finally, we have measured the orientation of each bubble, showing that the bubbles are in fact orientated along the *y*- axis. To visualise the bubble distribution inside the sample, Fig. 9b shows a top view of the rod, where each bubble is represented by an ellipse projected on the *xy*-plane.

As a consequence of the elongations of the bubbles, the surrounding mixture is also along the major axis of the rod. Therefore, the material properties described here prompts us to hypothesise that the fibres are distributed along the *y*-axis (see Appendix). The method used to make the rods creates an anisotropic



Figure 9 a Average size, in millimetres, of the resulting bubble using the foam forming process. b Top view of the rod, where each bubble is represented by an ellipse.





Figure 10 Foam rod under Scanning Electron Microscopy (same bar scale). a Clusters of fibres going in the same direction.b Multiple fibres coming out of the foam (white part). Figure is reproduced from [3].

material that enhances its mechanical properties [27]. Future work will focus on the mechanical properties of our bio-based rods [3].

From these results, we conclude that optical coherence tomography is a perfect technique to explore FoamWood solutions without any modification of the material.

Acknowledgements

The authors acknowledge support from the Academy of Finland Center of Excellence programme, 278367. JK acknowledge the funding from Academy of Finland (308235) and FinnCERES flagship programme (151830423), Business Finland (211715) and Aalto University (974109903) as well as Aalto Science IT project for computational resources. LV acknowledges the funding from the Vilho, Yrjö and Kalle Väisälä Foundation via personal grant.

Funding

Open Access funding provided by Aalto University.

Declarations

Conflict of interest The exact composition of bubble matrix is a proprietary biodgradeable foam manufactured in Aalto University in the project FoamWood.

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Appendix A Scanning Electron Microscopy

A Zeiss EVO HD 15 scanning electron microscopy (SEM) was used to image the foam rods. Samples were coated with 4 nm gold–palladium using a Leica EM ACE 600 vacuum sputter coater to improve the conductivity of the samples and thus the quality of the SEM images. Figure 10 shows our best results. Both figures shows the fibres align in a particular direction, while Fig. 10a shows clusters of fibres within the foam, Fig. 10b shown few fibres coming out of the foam. The ridges continue as "whiskers" further confirming that we can create oriented structures not only in bubble, but also in the (nano)fibre level.

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