Characterization and model-based design validation of 3D printed cookies

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Abstract

Additive manufacturing is revolutionizing processing in many applications including 3D food printing. A Fused Deposition Modelling printing method was developed to produce cookies. In order to design food of particular texture, a fine element model was established to predict the mechanical properties of structured products. Cookie structures were engineered to achieve desirable texture properties in silico and dedicated print files were created for 3D printing. In order to validate the model, the properties of the printed cookies were measured and analysed. Compression tests were performed to determine Young’s modulus. X-ray micro-CT imaging was applied to characterize the 3D microstructure of the printed cookies samples. Micro-CT imaging provided a better understanding about the effects of the 3D printing process on cookie structure. Finally, a better fit of the prediction model was obtained by adjusting the model geometry to the scanned printed structure, which indicates the importance of structure integrity for mechanical properties of printed cookies.

Keywords: Structure; 3D food printing; Cookies; Texture; Micro-CT

1 Introduction

Almost 30 years since its introduction, additive manufacturing (AM), commonly referred to as “3D printing”, is revolutionizing processing in many industrial applications [1]. Recently, interest and development of AM for food manufacturing has increased likewise, offering certainly the potential advantages of AM (more flexible, less waste) [2]. A potential application is the 3D printing of cookies with dedicated texture perception by modifying the printing design file. A cookie can be described as a solid material matrix surrounding gas spaces having different sizes and shapes. Manipulation of the shape, size and distribution of the gas spaces will influence the cookie’s texture perception. The development of models enables the in silico design of such food products based on knowledge from existing food products. This can be combined with 3D printing which allows to build dedicated structures based on structure design file resulting of the model-based analysis. The final step in this 3D printing application would be the model validation, by comparing the actual properties of the printed cookies with their predicted properties obtained from the model.

X-ray micro-computed tomography (micro-CT) is one of the most promising technologies that allows the inspection of food microstructure and macrostructure in 3D, with minimal sample preparation, and in many cases in a non-destructive way [3-6]. It constitutes an innovative sensor offering a vast potential for measurement of food structure, and thus the associated quality attributes with feasible spatial resolution.

In this contribution, micro-CT is applied to visualise and analyse the microstructure of 3D printed cookies of different designed structures, baked by traditional means, in relation to their mechanical properties. These actual properties are compared with the predicted properties from the model.

2 Materials and methods

2.1 Structure design and 3D printing

Based on a previous study focused on the relation between the microstructure and texture of cookies, we developed finite element models of mechanical deformation of honeycomb and Kelvin structures to investigate how structural parameters such as cell size, wall thickness and porosity affect the elastic properties of cookies. From this model, an inverse linear relationship was found between the porosity and the effective Young’s modulus of the structures, regardless of wall thickness and cell size [7].

Print files were generated corresponding to four honeycomb and two Kelvin structures with varying cell size and wall thickness as listed in Table 1. Those structures were successfully 3D printed based on the Fused Deposition Modelling (FDM) of cookie dough followed by a traditional baking process. Table 1 provides the cookies description and their characterization specification including mechanical characterization by compression, structural characterisation using micro-CT and moisture content measurement.
## 2.2 Structural characterisation

Micro-CT scans were conducted on all samples (in 1 or 5 repetitions for each type) in a Skyscan 1172 (Bruker MicroCT, Kontich, Belgium), with settings outlined in Table 2.

<table>
<thead>
<tr>
<th>Source Voltage (kV)</th>
<th>60 or 100</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source Current (µA)</td>
<td>167 or 100</td>
</tr>
<tr>
<td>Image Pixel Size (µm)</td>
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</tr>
<tr>
<td>Exposure (ms)</td>
<td>590</td>
</tr>
<tr>
<td>Rotation Step (degree)</td>
<td>0.4°</td>
</tr>
<tr>
<td>Frame Averaging</td>
<td>3 frames</td>
</tr>
<tr>
<td>Scan duration</td>
<td>00:24:31</td>
</tr>
</tbody>
</table>

Table 2: Settings of the micro-CT scans of the 3D printed cookies

Reconstruction of the images was conducted in NRecon 1.6.8.0 (Bruker MicroCT, Kontich, Belgium). 3D images of cookies were processed by means of CT Analyser version 1.14.4.1 (Bruker MicroCT, Kontich, Belgium) as reported in Table 3. To this end images were binarized by automatic thresholding using Otsu method to separate the pores from the material matrix. Then speckles were removed using the despeckle and morphological treatments: sweep, closing and opening of radius 3. Those treatments cleaned the binary data with respect to the dough porosity as presented in Figure 1. A first 3D analysis was performed on the resulting binary data to characterize the two phases in terms of parameters including porosity and the structure thickness.

Then, the data were cleaned again in order to remove the dough porosity and only keep the structure induced by the 3D printing process. A closing operation of radius 12 was applied to the binary data and the second 3D analysis was performed. The image processing was performed in a rectangular region of interest fitting the cookie base.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtering</td>
<td>Median: Radius 2</td>
<td>Smoothing, noise reduction and unsharpening of image</td>
</tr>
<tr>
<td>Thresholding</td>
<td>Otsu: Automatic</td>
<td>Segment the foreground from background to binary images</td>
</tr>
<tr>
<td>Sweep</td>
<td></td>
<td>Remove speckles from image</td>
</tr>
<tr>
<td>Closing</td>
<td>Radius 3</td>
<td>Connect objects that are in close proximity but initially distinct</td>
</tr>
<tr>
<td>Opening</td>
<td>Radius 3</td>
<td>Separate objects that are connected at a narrow junction</td>
</tr>
<tr>
<td>3D analysis</td>
<td></td>
<td>Calculate 3D parameters of binary image</td>
</tr>
<tr>
<td>Closing</td>
<td>Radius 12</td>
<td>Connect objects that are in close proximity but initially distinct</td>
</tr>
<tr>
<td>3D analysis</td>
<td></td>
<td>Calculate 3D parameters of binary image</td>
</tr>
</tbody>
</table>

Table 3: Image processing workflow used for the 3D analysis.
2.3 Mechanical characterisation
The stress-strain curves were obtained from force-deformation measurements using a TA.XTPlus texture analyzer device with a cylindrical metal compression plate of 75 mm diameter (Stable Microsystems, Godalming, UK). The compression test was performed with a load cell of 30 kg and maximal strain $\varepsilon$ of 5 % at a speed of 0.1 mm/s. The engineering Young’s modulus $E$ was determined by the slope of the linear part of the stress-strain curves of the compression according to Error! Reference source not found. 

$$E = \frac{\sigma}{\varepsilon} = \frac{\Delta F}{(A \cdot \Delta \varepsilon)}$$

Equation 1

where $\sigma$ is the stress (Pa), $\Delta F$ is the difference in compression force between a strain of 0.1 and 0.2, $A$ is the initial surface area of the sample and $\Delta \varepsilon$ is the difference in strain $\varepsilon$.

3 Characterization of printed cookies
3.1 2-D Honeycomb structure
From the micro-CT cross-sections presented in Figure 2, we can observe some internal cracks occurred during the baking process. The importance of the nozzle size can also be illustrated with the comparison of the cookie structure with cell size 7.5 mm and wall thickness 2.6 mm and the structure with cell size 5 mm and wall thickness 2.6 mm. For the first structure, the nozzle diameter of 1 mm was used resulting in the detachment of the wall during the baking treatment. In comparison, the structure with cell size 5 mm and wall thickness 2.6 mm that was printed with a nozzle of 2 mm diameter, did not have this detachment problem. The nozzle must be adapted in function of the wall thickness.

Two levels of structures can be distinguished in Figure 2:
- The dough microstructure corresponding to the internal cookie structure
- The macrostructure corresponding to the pores induced by the 3D printing design

Figure 1: 3D structure analysis of the 3D printed cookies: (a) Cross-section image after filtering. (b) Otsu thresholding. (c) Sweep, closing of radius 3 and opening of radius 3. (d) Closing of radius 12.

The volumes of individual pores and the 3D volume rendering were obtained using Avizo software (version 9.0.1, VSG, France). The workflow of the image processing was similar to this presented on Figure 1. In order to obtain the volume rendering of pores, a bitwise operation “NOT” was applied followed by a border kill treatment.
Figure 2: Micro-CT cross sections of the 3D printed cookies having Honeycomb structure (scale bar = 10 mm), volume rendering and volume pore spaces.

Table 4 presents an overview of the measured properties of the cookies having Honeycomb structures. We can observe that the cookies having similar Young’s modulus value have also similar porosity. From previous analysis [7], we concluded that the Young’s modulus increases with the decrease of the porosity. However, this fact was not always observed, especially for the cookies having a Honeycomb structure of 5 mm cell size and 2.6 mm of wall thickness. Moisture content is expected to be the predominant factor responsible for this deviation, as it is known that the $E$ modulus for cookies declines rapidly above a moisture content of 6% [8]. Repeated printing and baking of the Honeycomb cookie with 10 mm cells and 1.3 mm had a substantially lower moisture content of 4.11%, confirming that the porosity of the structure had a large effect on moisture content, implicating the mechanical properties of the different cookie structures.

<table>
<thead>
<tr>
<th>Cell size 5 mm Wall thickness 2.6 mm</th>
<th>E (MPa)</th>
<th>$\varepsilon$ (cubic volume) %</th>
<th>$\varepsilon$ (printed pores) %</th>
<th>Moisture content %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.74 ± 0.34</td>
<td>28.10 ± 2.80</td>
<td>23.97 ± 3.43</td>
<td>9.03 ± 1.19</td>
<td></td>
</tr>
<tr>
<td>Cell size 3.5 mm Wall thickness 1.3 mm</td>
<td>6.98 ± 0.6</td>
<td>30.69</td>
<td>26.87</td>
<td>NA</td>
</tr>
<tr>
<td>Cell size 7.5 mm Wall thickness 2.6 mm</td>
<td>6.02 ± 0.15</td>
<td>31.00</td>
<td>27.74</td>
<td>NA</td>
</tr>
<tr>
<td>Cell size 10 mm Wall thickness 1.3 mm</td>
<td>4.27 ± 0.52</td>
<td>62.31</td>
<td>60.66</td>
<td>NA</td>
</tr>
</tbody>
</table>

Table 4: Measured properties of the 3D printed cookies having Honeycomb structure

3.2 Kelvin structure

From the micro-CT cross-sections presented in Figure 3, we can also observe some internal cracks induced by the baking process. Two kinds of pore structures are shown in Figure 3:

- The dough microstructure corresponding to the internal cookie structure
- The designed macrostructure corresponding to the pores induced by the 3D printing
Figure 3: Micro-CT cross sections of the 3D printed cookies having Kelvin structure (scale bar = 10 mm), volume rendering and volume pore spaces.

Table 5 reports an overview of the measured properties of the cookies having Kelvin structures. We can observe that the cookies having similar Young’s modulus value have also similar porosity. Similar conclusions can be deduced from those results.

<table>
<thead>
<tr>
<th>Wall thickness</th>
<th>E (MPa)</th>
<th>ε (cubic volume) %</th>
<th>ε (printed pores) %</th>
<th>Moisture content %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.3 mm</td>
<td>3.06 ± 1.05</td>
<td>44.43 ± 3.87</td>
<td>40.85 ± 4.29</td>
<td>6.87 ± 0.09</td>
</tr>
<tr>
<td>2.6 mm</td>
<td>2.62 ± 0.45</td>
<td>35.29 ± 1.12</td>
<td>31.10 ± 1.38</td>
<td>7.86 ± 0.29</td>
</tr>
</tbody>
</table>

Table 5: Measured properties of the 3D printed cookies having Kelvin structure

4 Validation of the prediction model

The mechanical measurements were compared to the simulations of the designed structures as well as of the scanned structures. Figure 4 and Figure 5 compare the predicted properties obtained by the FEM model with the actual measured properties. Systematically, the measured results deviated from the predicted results. These deviations can be due to:

- Internal cracks occurring during the baking process
- Spreading of the dough during the Fused Deposition Modelling resulting in a spread cubic shape and smaller pores.
- Different moisture content due to different baking times influencing the Young’s modulus of the cookie material.

Particularly the more extreme cases for the honeycombs both at the lower end and higher end of the porosity scale have large deviations in E-modulus. The predicted values follow the trend of decreasing E-value with porosity, and most measured structures do follow this trend. The measured value of the low porosity structure, however, does not match this relationship, having a very low E-modulus compared to the other measured structures. This deviation is expected to be caused by the high moisture content of this structure (9.03%).

For the Kelvin structures, we also find large deviations in porosity between the model designs and the actual printed structures, as a result the values of effective E-modulus also are significantly different.
Figure 4 and Figure 5 indicate large differences in porosity between the model designs and actual printed cookies making interpretation of the comparison difficult. Using the 3D micro-CT images it was checked whether the comparison improves if the actual 3D structure is used in the prediction of effective mechanical properties of the structures. Furthermore, as moisture content varied among the samples affecting the E-modulus of the cookie material, different values of material E-modulus (EM) were also explored in the computation.
For the honeycomb structure, the results of this analysis are given in Figure 6. For two of the structures the E-modulus now falls in the range of measured standard deviation. Deviations remain large for the cookies of low and high porosity. Although the effective E-modulus is affected by the E-value of the matrix, this does not allow to improve the predictions relative to the measured values. Particularly for the low porosity cookie, the deviation remains high.

For the Kelvin foams, the results of this analysis are given in Figure 7. Using the actual cookie structure considerably improves the prediction of the effective E-modulus of the two investigated structures. The actual porosity of the printed structure is significantly lower than those of the model designs. As a result, the effective E-modulus increases and comes in range much more comparable to the measured values.
5 Conclusions and perspectives

From the results obtained for 3D printed honeycomb and Kelvin foam structures, some conclusions and perspectives can be formulated:

- The model is as good as it represents the actual printed structure. We found still considerable differences between the model design structures and those actually achieved by 3D printing. It was shown that model-based design of the texture will highly depend on the level of correspondence between the printed and design structure. Using the actual printed structure improved correspondence between model and measurement. Achieving enhanced structure correspondence by 3D printing will be required for effective texture design and control.

- The mechanical properties of the matrix material after baking determines the effective texture properties of the printed cookie. We have found that for the material studied in this project, there is also a large effect of the moisture content of the material after baking. Measurement of the E-modulus of the material after baking at different moisture contents revealed that the value of E may double with a decrease of moisture content of only 2% from 8 to 6%. So accurate control of the baking process to target a uniform moisture content will be required for effective texture design.

- The microporosity variation can influence the mechanical properties of the matrix material after baking. In this project, the mechanical properties were determined of solid cube-shaped cookies that could have a different microstructure than that of the 3D printed ones. Investigating the influence of the 3D printing on the microstructure is recommended to evaluate better the mechanical properties of the matrix material after 3D printing and baking. A 3D printed cubic solid cookie already showed to have a different E-modulus than one that was manually formed.

- Some observations could not be explained with the present model, even if the structural deviations were taken into account. This indicates that a more elaborate modelling approach may be required for predicting accurately texture properties of printed structures across a wide range of structural charactistics.

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References


