

1 **Microstructure evolution and infrared thermography applied to tensile failure** 2 **of a dilatant polymeric foam**

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8 **Abstract**

9 In this work, the authors study the thermo-mechanical tensile response of a dilatant polymeric
10 foam, focusing on developing characterization and testing methodologies needed to better
11 understand the links between temperature, microstructure and mechanical response in these
12 materials. The authors study these links for a commercially-available shear-thickening foam,
13 named D3O LITE D. Samples were tested in tension under quasi-static conditions for a strain rate
14 of 0.1s^{-1} using an MTS 810 testing apparatus. Micro X-ray computed tomography (XCT) was used
15 to study the evolution of microstructure (pore size and wall thickness) as a function of strain and
16 this was achieved by developing MATLAB-based programs to probe pore sizes and wall
17 thicknesses. The foam specimens were loaded until failure and this allowed to examine a variety
18 of stress regimes, including elastic regime, inelastic regime, and failure. These mechanisms are
19 observed in-situ using an infrared thermal camera which record temperature profiles and these are
20 linked back to stress-strain, and temperature-strain responses of the material to understand them
21 in better detail. For this material, the yield stress and elastic modulus were found to be
22 $0.57\pm 0.1\text{MPa}$ and $5.47\pm 0.1\text{MPa}$ respectively, at a yield strain of $10\pm 4\%$. At the time of failure, the

23 average temperature of the specimen was found to increase by $\sim 3^{\circ}$ C and a local temperature
24 increase of $\sim 8^{\circ}$ C was observed in the failure region. These results represent some of the first
25 tensile studies on shear-thickening foams in the literature, and the testing methodologies developed
26 in this study will serve as the foundation for additional experimental and computational studies
27 across a broader range of foam materials.

28 **Keywords** - Thermo-mechanical behavior, Shear thickening foam, Dilatant foam, Computed
29 tomography, Infrared imaging, Microstructure

30 **1. Introduction**

31 Polymeric foams are widely used in many automotive [1, 2], aerospace [1–3], military [3, 4], and
32 packaging applications [4]. Their energy absorbing capabilities and low density make them an
33 ideal candidate for protection materials, such as personal protection equipment (PPE) like knee
34 pads [5], and specific to this study, helmet liner materials [5, 6]. One specific type of foams are
35 dilatant or shear-thickening polymeric foams which react like a rigid materials when subjected to
36 high stress or deformation rates [7–9], making them considerable for protection materials. In the
37 past few years, Traumatic Brain Injury (TBI) has received increased attention in all fields,
38 especially the military. The main function of the inner liner of the helmet, generally made of closed
39 cell polymeric foams [6], is to absorb part of the impact/ballistic energy and to reduce the amount
40 of load transmitted to the head, which directly contributes to TBI. To make efforts toward
41 improving typical foams used in helmet liners, there exist a number of studies on compressive [5,
42 10, 11], tensile [11, 12], shear [12], and fracture [13] behavior of these polymeric foams. In this
43 present paper, we seek to understand the deformation mechanisms in tension for a recent shear
44 thickening foam.

45 To fully exploit the energy absorbing capabilities of foam materials, it is necessary to understand,
46 analyze, model, and validate the constitutive response of foams under multiple stress states. In the
47 literature, limited attention has been given to tension and other stress states [11 – 13] and more
48 attention to compressive states [1–3, 5, 10–12, 14 – 18]. Ayyagari et al.[15] and others[11, 13, 19,
49 20] note the necessity of studying and understanding the mechanical behavior in polymeric foams
50 under complex stress states and recognize the importance of studying the behavior in various stress
51 states which necessitates modeling of yield behavior in foams. These models are then used to
52 determine parameters such as elastic moduli [11, 13], elastic collapse stresses [11], and failure
53 strengths and strains [13] based on information from experiments, and microstructure, which
54 enables industry to design better foam materials. Generally, polymeric foams have a definite
55 isotropic compressive yield stress whereas dilatant foams tend to have a range of compressive
56 yield stress at elastic-collapse [7, 11, 18, 21, 22]. In one study, Ayyagari et al. [15] define the entire
57 yield surface of a material which need only two yield strengths parameters from uniaxial
58 compression and uniaxial tension. In a separate study, Walter et al. [11] show in their model that
59 the elastic moduli and failure strengths of polymeric foams increase linearly with foam density.
60 Their phenomenological model predicts the entire constitutive response in both tension and
61 compression. Ultimately, the benefit of creating models is gaining insights into how to control
62 microstructure or composition (manufacturing methods) to improve the foam performance for
63 specific applications. These models require mechanical parameters that are obtained from
64 experimental data, and we seek to contribute data for future modeling purposes.

65 In many studies, authors point to the importance of microstructure (usually in terms of density [5,
66 10, 23] and cell sizes [5, 10, 24]) and failure (usually through a post-test microscopic assessment
67 of the sample [10]) on the material response of polymeric foams. It is well recognized that

68 mechanical properties of polymeric foam materials depend on the properties of the base polymer
69 material [4, 14], relative density [12, 20] (ratio of the foam density to the density of base material)
70 and microstructural geometry (e.g., pore size and wall thickness) [5, 10, 24]. For example, Chen
71 et al.[24] show the effect of variation of cell size and wall thickness on elastic and shear moduli of
72 foams. In their study, material properties are estimated through experiments, cell size and thickness
73 distributions of the foam are measured from Scanning electron Microscopy (SEM) images, and
74 existing models are applied to predict stiffness of the foam. Chen et al. [24] also note that
75 variability in the cell wall thickness and cell size is important in overall tensile and shear response;
76 this is important to note for our study. In another study, Bouix et al. [5] show the differences in
77 elastic collapse stress levels and plateau stress moduli that manifest due to microstructure
78 differences in polypropylene foams. Also, the structural response of foams is strongly influenced
79 by the cell geometry (foam apparent density, cell topology and anisotropy ratio) and by properties
80 of the base material [23]. Thus, understanding the links between microstructure and mechanical
81 properties is of great importance for designing next-generation foams.

82 Micro X-ray computed tomography (XCT) is a widely recognized characterization technique used
83 to investigate the microstructure of a range of materials [3, 25–27]. With the development of XCT
84 techniques, finite element models based on the reconstruction of real foams using CT techniques
85 have also been reported by many studies [21, 28–30]. For example, Fahlbusch et al. [30] describe
86 a Representative Volume Element (RVE) - based model that predicts failure behavior of closed-
87 cell polymeric foams whose microstructure information is extracted through XCT. This model
88 shows the numerical and experimental failure points in foams in 11 different stress states for which
89 experimental data exist. In another study, Youssef et al. [21] study the local deformation
90 mechanisms of a polyurethane foam in compression by means of XCT. In their study, the

91 tomographic data is used to reconstruct the microstructure of the foam, and by using the
92 constitutive equations of the bulk material and finite element modeling (FEM) methods, the
93 macroscopic response of the material is captured. There are other techniques to investigate
94 microstructure such as Scanning electron microscopy (SEM) [6, 13, 31], Fluorescence microscopy
95 [32], etc. but these are inherently two-dimensional and do not provide sufficient 3D information
96 in a timely manor. In contrast, XCT offers an ability to determine 3D volumetric data in relatively
97 short time (5-10 minutes), thus allowing researchers a tool to assess features at the microstructural
98 level in 3D. Altogether, the literature demonstrates the importance of microstructure on the
99 mechanical response of foams, and we seek to contribute to this understanding in this paper.

100 In addition to efforts made to investigate failure [13, 33], develop models [11, 15], and characterize
101 materials [17, 34], the sensitivity of mechanical response to temperature in foams is also well
102 documented [23, 35–37]. For two different foam materials, Williams et al. [36] show that shear
103 strength and stiffness properties of the foams exhibit an inverse relation with temperature, whereas
104 the shear strain at failure exhibits a linear relationship. Infrared (IR) thermography is one of the
105 established methods to study the effects of temperature on failure and material behavior [38, 39].
106 Chrysochoos [38] demonstrate the effective use IR techniques to study material behavior such as
107 thermo-mechanical characteristic analysis of fatigue mechanisms in polymers and steels. In a
108 different study, Ohbuchi et al. [39] use thermal image analysis to visualize plastic
109 deformation/crack propagation in stainless steel in quasistatic tension. This was developed by
110 combining thermal image processing with Elasto-plastic finite element analysis coupled with
111 transient heat conduction calculations. These studies demonstrate the importance of thermal
112 studies on mechanical behavior of materials. In the present study, tensile tests are performed on a

113 dilatant polymeric foam and the distribution of the specimen surface temperature is measured by
114 using infrared thermography techniques.

115 This current paper builds on past works investigating the effects of stress states, temperature,
116 microstructure and failure on mechanical response of polymeric foams. The present study is an
117 extension of the work performed in a study by Bhagavathula et al.[7], where only the rate-
118 dependent compressive response was examined. In this current paper, we focus on understanding
119 the evolution of pore size, spatial distribution and wall thickness on the behavior of this shear-
120 thickening foam in quasistatic tension. As limited work is available in published literature on shear
121 thickening foam materials, this study intends to establish an understanding of microstructural
122 length scales in quasistatic behavior of our foams, accomplished through experimentation and
123 characterization. This paper is comprised of the following sections: first, thermography techniques
124 and sample preparation methods are established and described. Second, experimental methods are
125 presented that include microstructure characterization techniques and mechanical testing methods,
126 followed by the presentation of the experimental results. These results are supported by stress-
127 strain curves, cumulative distribution functions of microstructural parameters, X-Ray tomograms,
128 and infrared video images obtained from high-speed infrared camera. Finally, implications and
129 contributions of this work are highlighted, and future directions are suggested.

130 **2. Experimental Methods**

131 In this study, a shear thickening foam obtained from D3O is studied. D3O foam is used in padding
132 on sports equipment and currently investigated for use in ballistic protective equipment [40, 41].
133 The variant under investigation is D3O LITE D, which is a partially open and closed-cell polymer-
134 based material. The foam density is reported as 0.22g/cc and the chemical composition is held
135 proprietary by the manufacturer. Some of the physical and mechanical properties provided by the

136 manufacturer are listed on the manufacturer website [42]. The motivation in characterizing this
137 material stems from the lack of testing of this highly marketed non-Newtonian shear-thickening
138 material, and we want to determine the behavior of this material over various stress states and
139 strain rates [7].

140 **2.1. Sample Preparation for Mechanical Testing**

141 Tensile dog bone specimens were used in this study and these were manufactured using water jet
142 cutting, which is an accepted sample preparation technique in experimental studies [43]. The
143 dimensions of the gage section were 20 mm in length and 10 mm in width with a thickness of 4
144 mm, which is governed by the thickness of the as-received foam sheet. The tensile dog-bone
145 samples were designed with ASTM D3575 Standard [44] as a starting point (geometric ratios of
146 the samples follow the standard), but were scaled down to allow for sufficient longitudinal
147 displacement in the testing apparatus to load until failure. To aid in performing the infrared
148 measurements, one of all the specimens' faces were completely painted black using an air brush.
149 This is consistent with other practices in the literature [37, 39]. Care was taken to ensure that the
150 samples end surfaces were parallel, and that minimum damage is induced to the edges during
151 sample preparation. The sensitivity of material strengths to geometry and specimen-size effects
152 are widely discussed in literature [13, 16], and we expect them to show similar differences in our
153 strengths and those provided by the manufacturer as different testing conditions may have been
154 followed. We note that potential differences in composition, pore size and wall thicknesses may
155 also occur as a result of different sizes of received sheets.

156 **2.2. Quasistatic tension experiments**

157 The specimens were tested in tension at a quasistatic strain rate of $0.1s^{-1}$ using a Material Test
158 System (MTS) - 810 machine. This assembly included infrared visualization capabilities with an

159 infrared high-speed camera - FLIR X6901sc, which enabled us to observe temperature changes
160 observed during testing. Details of the camera are discussed in the next sub-section. Both camera
161 and MTS were triggered manually at the same time, and synchronization was verified through
162 comparison between when the piston displacement was first observed in the thermal images with
163 the displacement data recorded by the MTS machine (no adjustments were necessary). To perform
164 the tension experiments, the specimens were mounted on screw-type tension grips with adequate
165 surface area provided for thermal conduction within the sample. A 10 kN load cell with a
166 background noise corresponding to approximately $\pm 1\text{N}$ recorded the time histories of the forces,
167 and the displacement of the piston was measured to an accuracy of 0.001mm using linear variable
168 differential transformer (LVDT) displacement sensor. The tension grips were connected to the load
169 cell and the actuator displacement rate was set to be 2mm/s corresponding to a strain rate of 0.1s^{-1}
170 ¹ in the sample. Specimens were driven to failure in all experiments. The engineering stresses are
171 calculated by dividing the applied load by the original specimen gage surface area. The engineering
172 strains are computed by dividing the gage displacement in the camera images by the original gage
173 length.

174 **2.3. Infrared Thermography methods**

175 An infrared high-speed camera - FLIR X6901sc system was used in this study for temperature
176 measurements. The infrared thermal images and temperature data were recorded at a rate of 100
177 frames per second. At this frame rate, the FLIR X6901sc system provided 640×512 pixel
178 resolution was sufficient to capture the whole event until failure. The integration time was 1.0432
179 ms. For the camera temperature calibration, the FLIR X6901sc camera is placed within a
180 temperature controlled chamber and set to visualize a blackbody outside the chamber through a
181 hole in the chamber wall [45]. The chamber temperature is then cycled from ambient to highest

182 specified operating temperature, then down to lowest, then back to ambient. A correction curve is
183 generated from the data recorded and saved in the camera as part of the factory calibrations. These
184 measurements are compared to a National Institute of Standards and Technology(NIST)-calibrated
185 thermocouple. The calibration is performed for a range of temperatures. The infrared camera
186 directly records all data to an onboard Solid state disk (SSD). To obtain temperature measurements
187 during experiments, the detector output of the thermal camera is estimated from emissions of both
188 target body and that of background. These emissions depend on ambient temperature, T_a ($^{\circ}$ C),
189 target temperature, T ($^{\circ}$ C),and target emissivity ϵ . The measurement principle applied in this
190 thermography method is given by:

$$191 \quad V(T, T_a) = \epsilon W(T) + (1 - \epsilon)W(T_a) \quad (1)$$

192 where $V(T, T_a)$ is the detector output, $W(T)$ is the Black body emission energy, $\epsilon W(T)$ is the
193 emission of the target, and $(1-\epsilon)W(T_a)$ is the background emission. The emissivity of the target
194 body depends on its surface condition. The accuracy of temperature measurement increases with
195 increase in the emissivity of the surface [37, 39]. For example, for highly reflective (polished)
196 surfaces, the emissivity and corresponding temperature readings will be lower. This has been a
197 challenge for ductile materials [37], and has resulted in many authors painting their surfaces black
198 [37, 39]. In this study, following convention, the target specimen was sprayed with black paint to
199 obtain a higher emissivity of 0.92. The accurate measurement techniques of emissivity have been
200 proposed [46], but there are many difficulties because of various kinds of errors, especially the
201 atmospheric transmission coefficient. The coefficient of the band transmittance of the atmosphere
202 should be estimated accurately because it is an important factor for measuring temperature with
203 such a method when the distance between the measured surface and the camera is very large [46].
204 In our experiments, the distance between the camera and the object was only 0.25 m, so it can be

205 assumed to be unity in this experiment. In the experiment, the ambient temperature and atmosphere
206 temperature was recorded to be 21 °C. Before the experiment, the specimens were kept in the test
207 room for a long time to equilibrate to the room temperature.

208 The temperature data measurement was tried using the FLIR ResearchIR software's rectangular
209 ROI function and conforming to camera positioning, selecting a fixed ROI on the sample surface
210 for the entire course of the experiment was not possible, necessitating an external program for
211 temperature data analysis. This software allowed for export of csv files containing individual pixel
212 temperatures of every infrared image. These csv files were imported to MATLAB and a program
213 was designed track displacements and to calculate average temperature on the sample. The infrared
214 images are also exported as monochromatic tiffs and the edge of the tension grip is tracked using
215 image processing techniques. The infrared image index is converted to strain by synchronizing
216 with the MTS data, and monochromatic TIFFs and csv files are synchronized through strain. The
217 temperature data corresponding to those pixels containing the grips are deleted. The background
218 pixels are also deleted using a threshold background temperature.

219 **2.4. X-Ray Computed Microtomography**

220 Synchrotron radiation based X-ray microtomography was used at the Biomedical Imaging and
221 Therapy (BMIT) facility - Canadian Light Source (CLS)[47] 05ID-2 - SOE-1 hutch, Saskatoon, to
222 obtain volumetric information of the microstructure. The facility's capabilities are mentioned in
223 table 1 [48]. Samples of 5 mm × 5 mm × 4 mm were manually cut from the center of the sample
224 gage section for imaging. This was done so as to accommodate the sample on the mounting pad.
225 The samples were mounted with their loading axis parallel to the scan direction. The beam energy
226 was set to 30 keV. The imaging setup used a Hamamatsu ORCA FLASH 4.0 detector. This
227 detector provided an effective voxel resolution of 3 μm where the distance between the specimen

228 and detector was 20 ± 1 mm. This resolution provides sufficient scan volume in order to sufficiently
 229 resolve the cell wall thickness (3 to 30 μm), and pore size (30 to 120 μm) analysis. The sample
 230 loading stage operated in intermittent motion and each sample scan comprised of 900 tomograms
 231 being acquired within 10 minutes over 180° of rotation. Reference images without the sample, and
 232 dark images without X-rays were also obtained before and after every scan to increase the quality
 233 of images during background filtering reconstruction.

234 Table 1: Beamline parameters of 05ID-2 – SOE-1

Source	Superconducting (SC) Wiggler
Energy Range	25 – 150 keV
Wavelength	0.5 – 0.08 Angstroms
Resolution $\Delta E/E@E$	M_1 CT: 10^{-3} and M_4 KES: 10^{-3}
Beamline Length	55 m
Maximum Beam Size (Horizontal x Vertical)	220 mm \times 11 mm @ 55 m
Maximum sample-detector-distance	6 m

235

236 Three-dimensional reconstruction of the tomograms was achieved using a MATLAB- based
 237 reconstruction program. This program performs 2D characterization on slices obtained from the
 238 3D scans of the sample volume. To demonstrate these capabilities, we show a flow chart of the
 239 image processing methodology adopted in this study in Figure 1. Figure 2 shows a sample
 240 tomogram and sub-sequential image processing applied to it and is used to explain the flow chart

241 in Figure 1. First, a threshold ranging from 0 to 1 is defined for converting greyscale images to
242 binary images. The images are converted to data type uint16 for better handling and processing,
243 and to minimize loss of quality. The tomograms have varying contrast and brightness at different
244 slices of the foam. The brightness is adjusted and the contrast is sharpened to obtain images with
245 consistent lighting exposure to improve segmentation of distinct pores and walls. For example, the
246 intensity of each pixel in greyscale images varies from 0 to 65535 corresponding to black and
247 white pixel, respectively, for data type 'uint16'. Depending on the amount of contrast in the
248 original image, the threshold is varied manually to obtain the best result. For the tomograms
249 obtained from CLS, it was kept at a constant value of 0.34. Using the threshold, the adjusted and
250 converted image is converted to binary values. Binarizing often results in several noises and aliases
251 being created. Most of these unwanted features stand out because they are often small and large in
252 terms of pixel area. They are filtered out by defining a range of pixel sizes to delete. In the binarized
253 images, walls have a value of 0, and the pores have a value of 1, and this is shown in Figure 2(b).
254 Each slice is read and stored in a large 3D array for processing. Following this, a series of image
255 processing operations are performed to segment the pores.

256 After image processing, the binarized image is stored in a 3D array. The segmented image is then
257 visualized by plotting it and it is determined whether or not the image segmentation is acceptable.
258 Parameters in the image processing step are varied until a desirable segmentation is obtained.
259 Using the binarized 3D image, a disk structuring element is used to grow the pore size on the image
260 in order to thicken the walls using a function called 'erosion'. The disk size was appropriately
261 selected depending on the sizes of the pores for image erosion. Erosion is then implemented to
262 close walls that may have been opened and filtered out during the image processing step. This
263 process further minimizes the noise for improved data analysis. Similar to the output binarized

264 image, the eroded image is visualized to determine if the erosion is appropriate as shown in Figure
265 2(c). After eroding the walls, the pores that are along the border of the scan are closed and filtered
266 out. These regions are not of interest for data analysis since they are incomplete. The image without
267 the border pores are stored in another variable. The eroded image without border pores are used to
268 determine the pore sizes and wall thicknesses.

269 Using the output binarized images, the connectivity of pores in 3D is used to find the connected
270 regions in the images, and the coordinates of the 3D pore centroids are calculated. The coordinates
271 of the pore centroids and resolution of the scan are input into a custom function that calculates the
272 pore diameters and wall thicknesses. In this custom function, the pore diameters and wall
273 thicknesses are calculated by counting the number of white and black pixels, respectively, in 6
274 orthogonal directions. The coordinates of the centroids are used as the starting point for all the
275 pores. Corresponding to each centroid, 3 diameters (1 diameter in each x,y and z directions) and 6
276 wall thicknesses are calculated. These values are stored in a 3-D array for further data processing
277 and plotting. For porosity measurements, the pore volume was calculated using the reconstructed
278 XCT scans and was subtracted from the total sample volume.

279 **3. Experimental Results**

280 Three trials for tension were performed at the same nominal strain rate to verify repeatability of
281 the material behavior. We show the stress-temperature-strain curves in Figure 3. The x, y₁ and y₂
282 axis represent strain (%), stress (MPa) and temperature change (°C), respectively, in a linear scale.
283 The solid black curve (trial 1) is highlighted for clarity as we later use the figure to discuss
284 temperature curves and microstructure changes, presented in a subsequent section. The dotted
285 curves for trials 2 and 3 are shown for repeatability. In the quasistatic tension experiments, the
286 yield stress was measured to be 0.57 ± 0.2 MPa corresponding to a yield strain of $10\pm 4\%$ across all

287 samples. The elastic modulus in the corresponding strain range was measured to be $5.47 \pm 0.1 \text{ MPa}$.
288 In the inelastic deformation regime, the stress was found to linearly increase with strain at rate of
289 $0.44 \pm 0.05 \text{ MPa } \epsilon^{-1}$. The inelastic deformation regime extends until a strain of $298 \pm 7\%$ is achieved,
290 and no other notable features are observed on the stress-strain curve until failure. The failure
291 strength and strain was measured to be $1.99 \pm 0.05 \text{ MPa}$ and $305 \pm 5\%$ across all samples,
292 respectively. To observe the effects of black paint on the tensile response, one experiment was
293 performed under similar experimental conditions with an unpainted specimen. This trial is
294 represented by trial 3 (T3) in Figure 3 with a solid blue curve. As observed from the figure, there
295 is no effect of presence of black paint on the mechanical response.

296 **3.1. Infrared Thermography results**

297 Shown in Figure 3 are the stress-temperature-strain curves of tensile response of D3O LITE D. As
298 noted earlier, the y_2 axis represents temperature changes observed on the specimen surface. To
299 account for effects of atmospheric conditions, absolute temperature differences (ΔT) observed on
300 the specimen surface are reported here (note the y-axis on the right side of the graph in Figure 3).
301 The black dotted curve represents the temperature changes observed using a local ROI at the failure
302 region. As observed from the curve, the temperature initially decreases by $\sim 0.05^\circ \text{ C}$ from 0%
303 strain until a strain of $\sim 25\%$ is reached in the sample. Following this, the temperature continues
304 to increase steadily at a rate of $1.02 \pm 0.05^\circ \text{ C } \epsilon^{-1}$ in the whole sample until failure. This drop and
305 rise in temperature corresponds to the presence of an inflection on the curve at $\sim 30\%$ in the stress-
306 strain curve corresponding to the offset of the elastic regime. At a strain of $\sim 300\%$ at the region
307 of failure, the temperature starts to increase rapidly at a rate of $90 \pm 5^\circ \text{ C } \epsilon^{-1}$ as the specimen starts
308 to fail. A peak temperature rise of approximately 8° C is observed at the region of failure. The
309 dashed black curve represents the average specimen temperature changes observed using a large

310 ROI over the specimen cross section. A similar inflection is observed on the average specimen
311 temperature curves as well, suggesting that the initial thermal response is globally similar
312 throughout the sample. This can be corroborated from the figure where dashed and dotted black
313 curves overlap each other until a strain of 30% is reached in the specimen. Hereafter, the average
314 temperature of the specimen continues to rise steadily at a rate of $1.02 \pm 0.05 \text{ } ^\circ \text{C} \epsilon^{-1}$ until failure. No
315 notable features are observed on the temperature-strain curve until failure.

316 Next, we consider global measurements on the surface of a specimen by examining individual
317 images from the infrared camera. Figure 4 shows the infrared video frames from one of the
318 quasistatic tension experiment obtained using the FLIR X6901sc infrared camera at a framerate of
319 100Hz. Conforming to positioning of the camera, and using a fixed ROI for entire course of the
320 event, a small ROI was selected as visualized in Figure 4. The dashed black curve in the
321 temperature-strain axes in Figure 3 represents the average specimen temperature changes observed
322 using the selected ROI over the specimen gage section. The temperature then continues to rise at
323 a rate of $1.02 \pm 0.05 \text{ } ^\circ \text{C} \epsilon^{-1}$ until a strain of 300% is reached in the specimen. We then observe some
324 large temperature fluctuations on the curve at higher strains but no further rise in overall
325 temperature. This is caused due to the positioning of the ROI. At strains $< 150\%$, an appreciable
326 amount of surface area of the entire gage section is under observation but as strain increases, a
327 large considerable area remains outside the fixed ROI. At strains higher than $< 150\%$, the specimen
328 elongates in such a way that the gripped area of the dog bone specimen starts to appear inside the
329 selected ROI. This presence reduces the measured rate of temperature increase as the gripped area
330 does not contribute to the thermal measurements. This effect increases with increasing strain, and
331 at strains higher than 225%, the measured temperature plateaus until failure. It is to be noted that
332 small changes in emissivity and surface condition occur during tensile testing and the errors caused

333 by these effects are infinitesimally minute when compared to the overall temperature rise. The
334 uncertainties associated with the ROI selection and corresponding temperature measurements were
335 found to be less than 1%. This was found from temperature measurements using different size and
336 shape ROIs on multiple locations on the sample surface. Overall, the infrared camera provided
337 consistent results and this paper demonstrates that heat release associated with tensile testing can
338 be successfully monitored using high-speed infrared imaging.

339 **3.2. Microstructure results**

340 Shown in Figure 5 is a composite figure of XCT images showing evolution of microstructure in
341 quasistatic tension. Note the loading direction is in the y direction. These images are obtained
342 through XCT slices of the samples' center starting from 10% strain, at increments of 20% strain
343 until 100% strain. Image (1) shows the scan orientation of the specimens, where it can be observed
344 from image (1) that there are irregularly shaped pores with a circularity of $\sim 80\%$ [7]. Some form
345 of additives appear to be concentrated at the pore boundaries which are visualized by small white
346 dots in the XCT images. Images (2) to (6) shows the differences in microstructure observed with
347 increase in strain. There are no signs of permanent damage or pore elongation observed in image
348 (2) as the pores remain intact. Signs of damage to the cell walls are observed in image (3) and they
349 become increasingly prominent in images (4) to (6). Pore elongation and cell wall stretching also
350 becomes more visually apparent in images (4) to (6). As observed from the images, the foam has
351 a stochastic pore structure. The quantitative measurements of pore sizes and wall thickness as a
352 function of strain are explored next.

353 **3.2.1. Pore characterization: shapes and sizes**

354 The procedure for determining the pore size and wall thickness measurements were discussed
355 earlier. Note, we recognize that foam samples recover up to $\sim 20\%$ of the applied strain following

356 tension testing, however, as will be shown, insights can be gained as to what pore size length scales
 357 are activated during testing. Pores with sizes less than 250 μm were considered for pore size
 358 measurements based on visual confirmation from XCT scans. Now that interesting characteristics
 359 of the pore size measurements have been identified, the effect of tensile loading on pore sizes is
 360 examined next. Here, we plot the cumulative distribution of pore size in both x and y directions.
 361 The cumulative distribution is defined as:

$$G(x) = \int_0^x g(\bar{x})d\bar{x} \quad (2)$$

362 where $g(\bar{x})$ is the probability distribution of pore sizes. The pore size data set is a discrete set of
 363 n pores with sizes of l_i ($i=1\dots n$). Ordering this data for increasing pore size, and assigning a
 364 probability of $1/n$ to each pore, the normalized empirical cumulative distribution function, or
 365 eCDF, can be computed as the sum of these probabilities:

$$G_e(\ell) = \frac{1}{n} \sum_{i=1}^n I(\ell_i \leq \ell) \quad (3)$$

366 where the indicator function I has a value of 1 if $\ell_i \leq \ell$ and 0 otherwise. For each tomography
 367 scan, a total of $\sim 70,000$ pores were considered for size and thickness measurements. Shown in
 368 Figure 6 are the eCDFs which are used to identify the likely range of the pore sizes and trends.
 369 This is achieved by looking at the values between the 10th and 90th percentiles. This range is
 370 projected to be between 40 μm and 120 μm in both x and y directions for a pristine sample. In
 371 Figure 6, D_x and D_y represent the pore sizes in x and y directions respectively. With increasing
 372 strain up to 30%, a peculiar behavior is observed among the curves that smaller pores tend to
 373 elongate in both x and y directions while larger pores tend to contract in both directions. For strains
 374 greater than 30%, the pore size distribution follows a constant and an expected trend [12, 13]
 375 where all pore sizes reduce in the x direction and increase in the y (loading) direction with
 376 increasing strain. From Figure 6, when the specimen is loaded in tension at a initial strain range of

379 0 – 70%, it is observed that pores sizes of $D \sim 100 \mu m$ show larger changes in the x direction when
380 compared to y direction. This is caused due to smaller pores getting stretched initially, and thus
381 increasing the number of pores that are greater than $100 \mu m$. At strains greater than 70%, the
382 differences in pore sizes with $D > 100 \mu m$ becomes more prominent and this can be visualized by
383 90% and 100% strain curves having bigger pore diameters than other strains. For the 90% and
384 100% strain curves, there are more number of bigger pores possibly due to tearing and ripping of
385 walls of small pores, resulting in coalescence into bigger pores.

386 **3.2.2. Wall thickness characterization**

387 Shown in Figure 7 are the eCDFs of the wall thicknesses for strains of 0% to 100%. Wall thickness
388 sizes less than $50 \mu m$ were considered based on visual confirmation from XCT images. In Figure
389 7, T_x and T_y represent the wall thicknesses in x and y directions respectively. The wall thickness
390 range is projected to be between $3 \mu m$ and $30 \mu m$ in both x and y directions for a pristine sample
391 and also profiles of T_x and T_y are similar. For all samples, it is consistent that $T_x < T_y$ as strain in
392 the y -direction increases. These observations are made from looking at individual plots of each
393 sample with T_x and T_y on the same plot. When compared across samples, both T_x and T_y decreases
394 as strain increases. This trend is followed consistently throughout for T_x . For T_y , it generally
395 follows a similar trend but with slight variations. T_y for a pristine sample is very different from the
396 other strains for T_y . Also, there is another effect that makes T_y smaller as strain increases. This is
397 possibly due to tearing of walls, making thicker walls thinner. Although relative differences
398 between these values are small, these variations in wall thickness evolution are to be noted.

399 3.2.3. Percentile plots

400 To better understand the evolution of microstructure during the quasistatic tensile response
401 of this D3O LITE D foam, all quantitative microstructure data is consolidated and presented in
402 form of percentile plots for both pores sizes and wall thickness variations in Figure 8. The
403 percentile plots are obtained from the CDF function for $G(x) = 0.1, 0.25, 0.5, 0.75$ and 0.9 (e.g.
404 $G(x)=0.1$ for the tenth percentile). This is done to clarify trends observed in the cumulative
405 distribution plots. In Figure 8(a), the x and y axes represent pore size (μm) and strain (%)
406 respectively, and wall thickness (μm) and strain (%) respectively in Figure 8(b). A single color
407 scheme is used for both pore size and wall thickness percentile curves, and are denoted by $x - P_i$
408 and $y - P_i$ in the common legend. The percentile plots are denoted by P_i where i represents the
409 strain percentage. In both subplots, porosity is plotted as a function of strain which gives more
410 basis of discussion. However, it is to be noted that porosity calculations were performed on the
411 final segmented images from pore size measurements. Since different disk sizes were used to
412 compute the segmented images for different strains, there is approximately 1% error in porosity
413 calculations.

414 Shown in Figure 8(a) are the percentile plots corresponding to those in Figure 6. At strains
415 less than 20%, the trend is clearly observed that smaller pores tend to deform first and they deform
416 equally in both x and y directions. This results in a sudden decrease in porosity. As strain increases
417 greater than 20%, it is observed that pores of all sizes show expected behavior that sizes increase
418 in y direction and decrease in the x direction. This results in a near constant rate of increase in
419 porosity of the material. From Figure 8(a), it is also observed that the number of pores with size
420 $< 100\mu\text{m}$ is smaller for higher strains than the pristine sample. This is consistent with the previous
421 statement that as smaller pores gets stretched, there will be fewer of the small pores. Shown in

422 Figure 8(b) is the percentile plots for the wall thicknesses in both x and y directions. Again, we
423 discuss percentiles of $G(x) = 0.1, 0.25, 0.5, 0.75$ and 0.9 . From the figure, it is observed that there
424 is no effect of tensile loading on evolution of wall thicknesses throughout the strain range. This
425 implies that the stress response is primarily governed by geometry change of the pore structure.
426 Generally, with increase in strain, the trend of the observed microstructural length scales tend to
427 follow a very slight linearly increasing path until onset of failure occurs. And as noted before,
428 bigger pores are susceptible for larger pore size variations and this true until the onset of
429 localization is observed.

430 **4. Discussion**

431 This paper investigated the thermo-mechanical behavior and microstructure evolution
432 during quasistatic tensile failure of a shear-thickening polymeric foam. It is important to have a
433 better understanding of behavior of these materials since this class of foams are currently being
434 employed in energy absorption equipment (e.g., helmet liners for US team sports such as Football,
435 Baseball and Ice Hockey, as well as protective inserts for Motorcycle jackets) and also in some
436 military applications [40, 41]. Limited data on these foams, and shear thickening foams in general,
437 exist in the literature, and so this paper seeks to make contributions towards better understanding
438 how microstructural features and thermal response of these types of foams may be related to
439 quasistatic tensile failure. In what follows, the authors discuss the results of behavior of this foam
440 in the context of their general understanding of how polymeric foams behave in tensile loading,
441 highlighting our key results where necessary. The results obtained through infrared thermography
442 and mechanical measurements, as well as qualitative and quantitative data obtained from Micro
443 X-ray computed tomography provide the basis for this discussion. First, each regime in the

444 stress–strain curve in Figure 3 is discussed in greater detail with the help of microstructure data,
445 temperature–strain curves, and infrared video images from Figure 4.

446 **4.1. Elastic regime**

447 The first part of the stress–strain curve is the elastic regime, which represents strains less
448 than $10 \pm 4\%$ for D3O LITE D. For polymeric foams, the linear-elastic strain limit is found to be
449 different for different foam materials throughout literature [11, 13]. Limited data exists on the
450 thermal response of foams in the literature. For the D3O LITE D material studied here, we observe
451 that the average temperature of the sample initially decreases by $\sim 0.05^\circ\text{C}$ over $\sim 30\%$ strain,
452 which corresponds to the end of elastic regime of the material. This decrease is believed to be a
453 result of a small decrease in the emission surface area during tensile loading. In the infrared images,
454 no notable features are visible on the specimen and temperature is uniformly distributed across the
455 surface in the elastic regime. From the XCT data, it is visualized in images (1-2) of Figure 5 that
456 larger pores remain the same sizes and there is no visible damage to the microstructure in the
457 elastic regime ($< 10 \pm 4\%$). In this range of strain, it is observed from Figure 8(a) that all pore
458 sizes do not undergo deformation, but there is a slight increase in the porosity. This is believed to
459 be caused due to relative differences in sufficient permanent elongation/contraction being
460 experienced by the different pore sizes as seen in Figure 6 and Figure 8.

461 **4.2. Inelastic regime**

462 Over the inelastic deformation regime, the stress in the sample rises linearly with strain at
463 a rate of $0.44 \pm 0.05\text{MPa}\epsilon^{-1}$ over 30 to $298 \pm 7\%$ range of strain. It is to be noted that elastomeric
464 foams can undergo much higher strains than those experienced before the linear-elastic strain limit
465 and the deformation can still be mostly recoverable, but is non-linear [16]. After a strain of $20 \pm$
466 5% is reached in the specimen, there is an inflection in the temperature curve at roughly the point

467 in the stress-strain curve that corresponds to post elastic part. The temperature starts to increase
468 beyond a strain of $30 \pm 5\%$ resulting from thermal energy release in the process of
469 micro-cracking of the cell walls. Over this entire range of strain, average temperature rise of 2
470 degrees is observed within the specimen at the end of the inelastic regime. The temperature initially
471 decreases during the elastic part and then steadily increases. In XCT images (3-6) of Figure 5, it
472 can be observed that with increasing strain in the y direction, pore sizes show visually observable
473 differences in sizes. For strains greater than 30%, it can also be observed from the images that
474 pore boundaries and walls show signs of damage and permanent deformation. The porosity is
475 observed to linearly increase at a rate of $0.075\% \epsilon^{-1}$ until failure.

476 **4.3. Pre-failure and Failure regime**

477 The pre-failure regime starts at a strain of $298 \pm 7\%$ for D3O LITE D. The ultimate tensile
478 strength was measured to be $1.99 \pm 0.05\text{MPa}$ which the observed during failure and the failure
479 strain was measured to be $305 \pm 5\%$ across all samples. Prior to failure, minor fluctuations in
480 temperature are observed and this is likely due to the result of localization because of pore tearing.
481 At the beginning of pre-failure regime, a local heat zone is observed in image 7 of Figure 4 which
482 indicates local failure being initiated within the sample. In Figure 3, the dotted black curve shows
483 the temperature measured across the region of failure. In the local failure region, there is sharp rise
484 in temperature by $\sim 8^\circ\text{C}$ degrees observed at the point of failure at a strain of $305 \pm 5\%$. This heat
485 spot results from energy release in the course of the breaking of chemical bonds during failure. At
486 the onset of failure, the failure zone initiates at a point on the specimen perimeter and failure
487 propagates at $\sim 0.05\text{m/s}$ through the sample leading to failure. XCT images and microstructure
488 data corresponding to strains $> 100\%$ to failure are not shown or discussed as steady trends in
489 both stress-strain, and temperature-strain curves are observed throughout, and no deviations are

490 observed until failure. The contributions for XCT of foam can provide insights into the cell-by-
491 cell deformation of the foam, which is hypothesized by Di Landro et al. [6], Mills et al. [18] and
492 other microstructure based models.

493 **5. Conclusion**

494 The thermo-mechanical quasistatic tensile response of a shear-thickening polymeric foam D3O
495 LITE D is explored at a strain rate of 0.1s^{-1} . Important microstructural parameters like pores sizes
496 ($40\mu\text{m}$ to $120\mu\text{m}$) and wall thicknesses ($3\mu\text{m}$ to $30\mu\text{m}$) have been identified using Micro XCT
497 and image processing techniques. Overall, pore sizes were found to increase linearly with
498 increasing strain, and it was observed that bigger pores ($D > 100\mu\text{m}$) are susceptible for larger
499 variations in size when compared to smaller pores. The changes in wall thicknesses were
500 comparatively negligible through the entire strain range (0 to $305 \pm 5\%$). The thermal response
501 of this foam during quasistatic tensile testing was established using Infrared Thermography and
502 the average specimen temperature was found to increase by $\sim 3^\circ\text{C}$ and concurrently a local
503 temperature increase of $\sim 8^\circ\text{C}$ was observed at the failure region over a strain of $305 \pm 5\%$. These
504 inferences are supported by stress-strain curves, XCT images of original and evolving
505 microstructure, eCDFs of pore size and wall thicknesses, and high speed infrared thermography
506 images. As this is the first time that the D3O LITE D foams have been characterized in this way,
507 the authors believe these experimental results will also serve as a good starting point for impactful
508 modeling [6, 18]. The results of the tests performed and the future tests will be put together to
509 make models to predict the effect of microstructure, temperature and stress states on the
510 mechanical response of shear thickening foams.

511

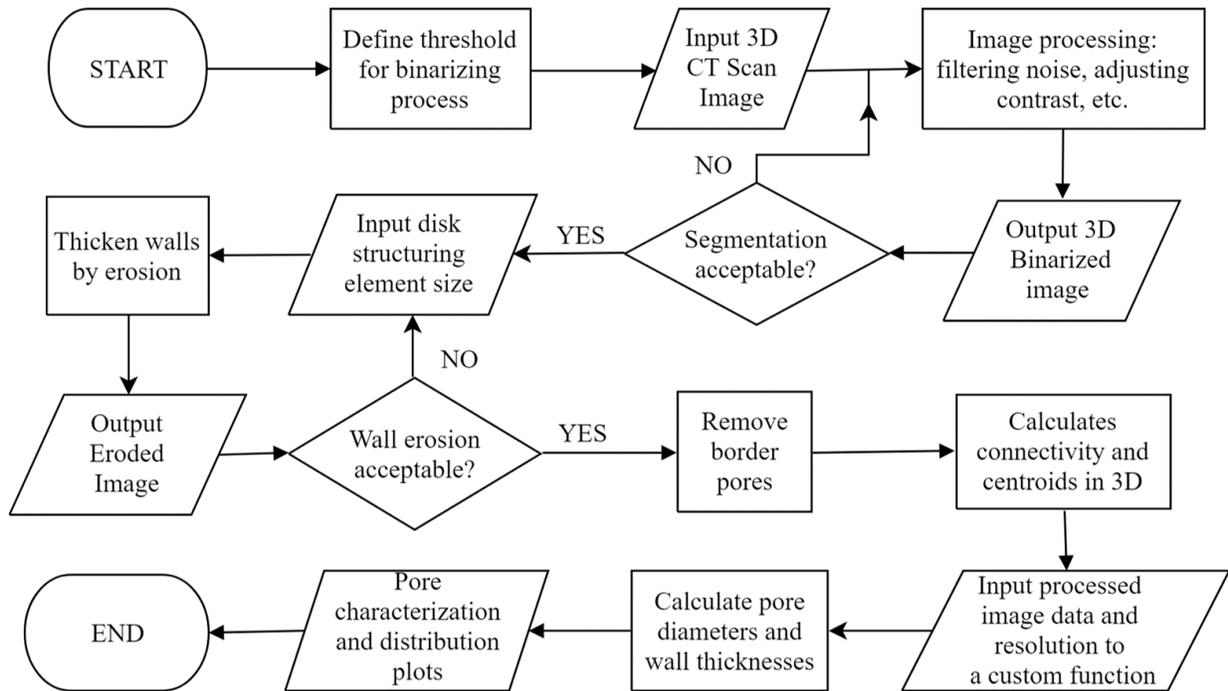
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527

528 **List of Figures**

529

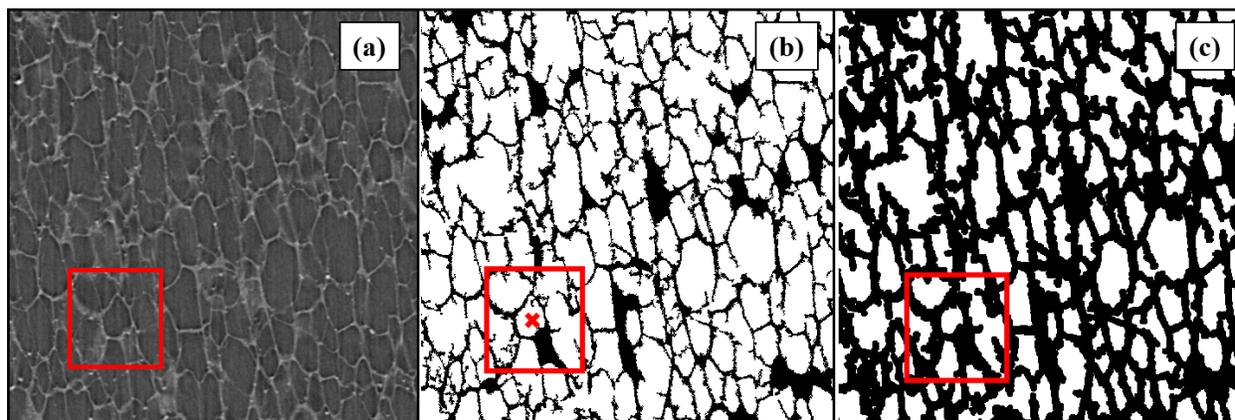


530

531 Fig. 1: Flow chart of the MATLAB program used to reconstruct and analyze the Micro X-ray
532 computed tomography scans of the polymeric foam specimen obtained from Canadian Light
533 Source.

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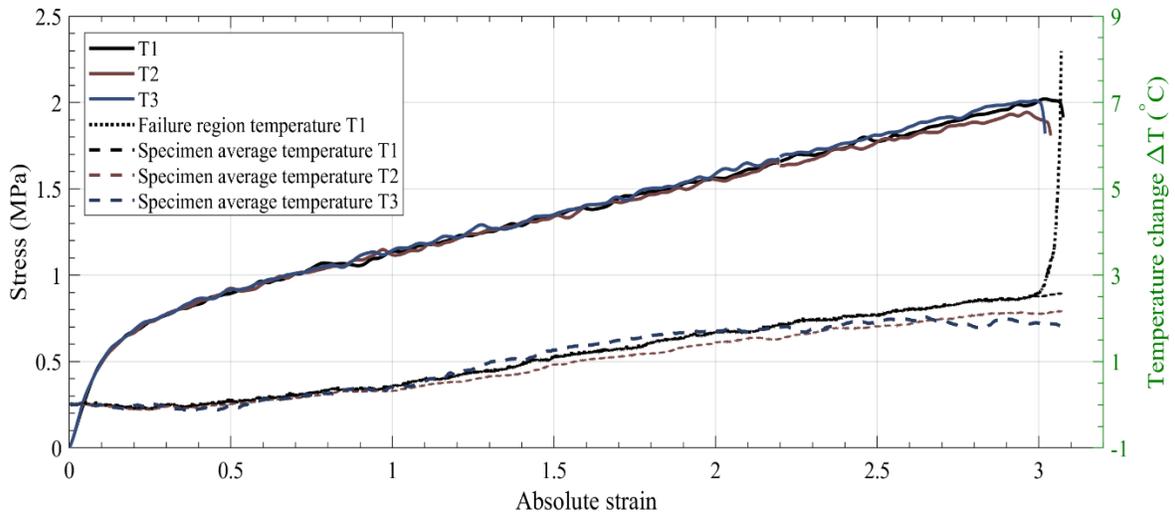
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536

537 Fig. 2: Segmented XCT image used to identify locations of additives and throats (constrictions
538 between two pores) within the sample, and for reconstruction. (a) Original XCT image with a
539 specific pore in interest shown in the red box. (b) Binarized image after image segmentation with
540 centroid. (c) Output eroded image showing thickened walls for closing off pores that opened up
541 during binarization which is used to calculate pore centroids.

542



543

544 Fig. 3: Figure showing Stress-Temperature-Strain (y-y-x) graph where black curve represents
 545 quasistatic tensile response of D30 LITE D foam. The black dashed curve represents average
 546 temperature measured across the entire gage section tracked through time. The black dotted curve
 547 represents temperature measured across the cross-section at localized region of failure tracked
 548 through time. The temperature of the sample is observed to uniformly increase with respect to
 549 strain until failure is initiated where a localized temperature increase of $\sim 8^{\circ}\text{C}$ is observed.

550

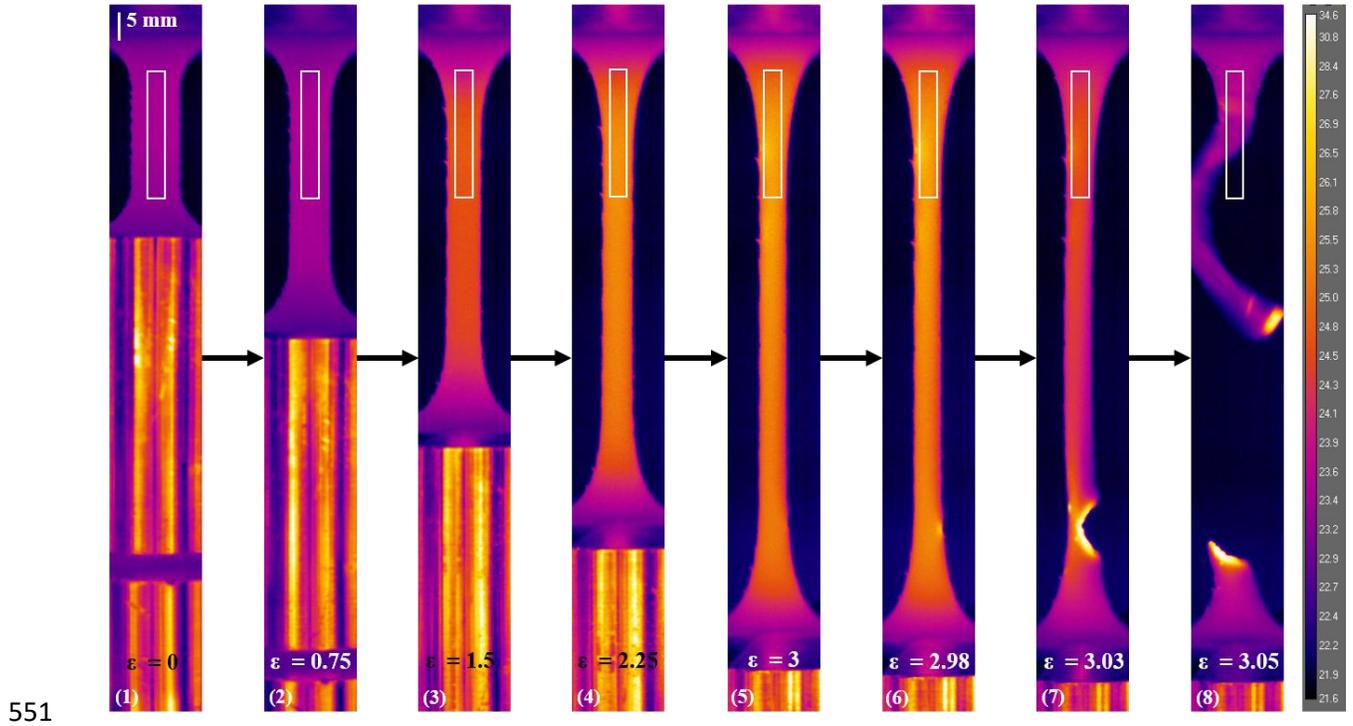
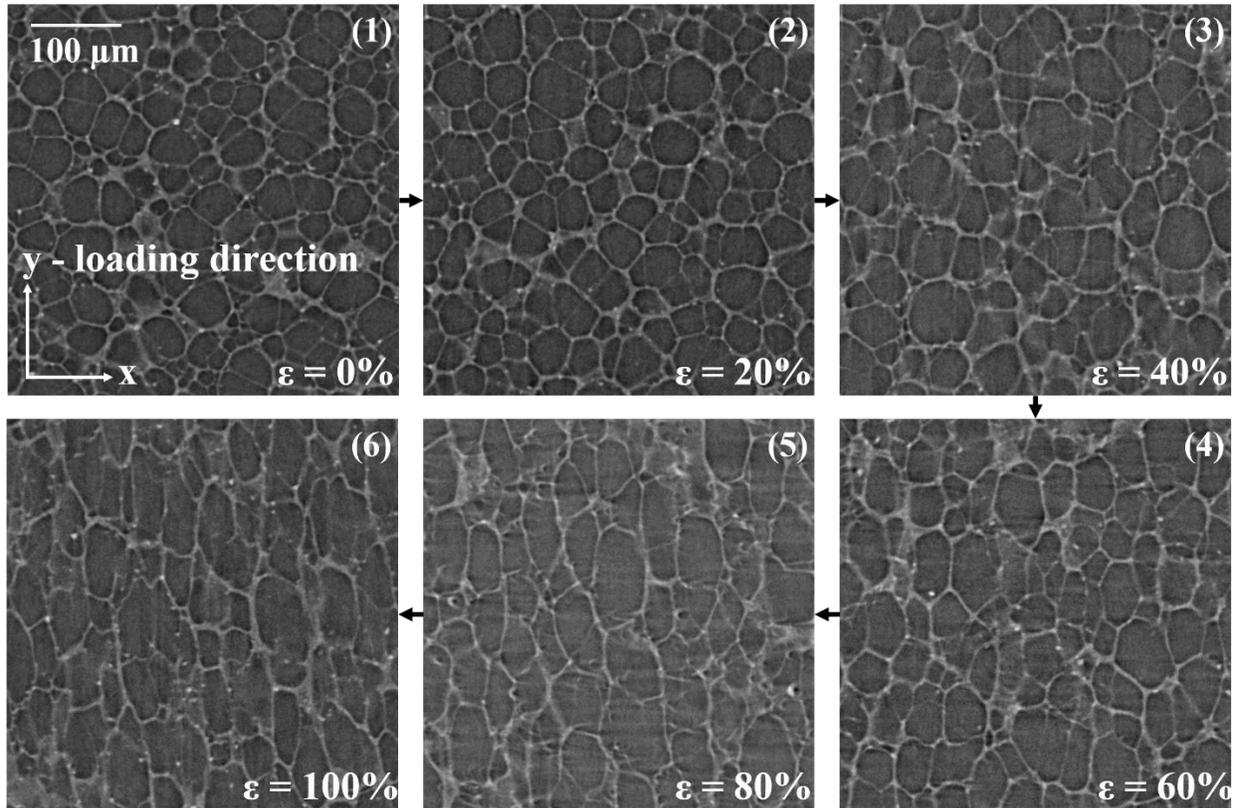


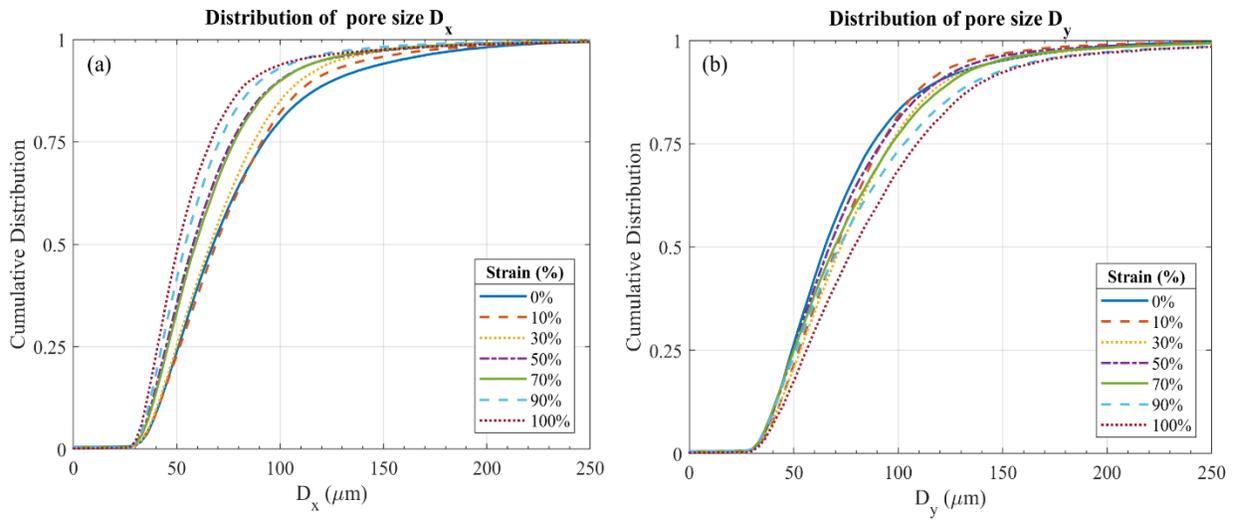
Fig. 4: Thermal camera video frames from the quasistatic tension experiment at a strain rate of 0.1s^{-1} obtained using FLIR X6901sc at 100Hz. Inter-frame strains and specimen lengths are denoted. Color bar on the right denotes temperature scale in Celsius. The last three images emphasize localized temperature increase that is observed at the failure region at a failure strain of 3.05.



558

559 Fig. 5: Composite figure showing microstructure evolution in quasistatic tension through XCT
 560 images of the sample center at increments of 20% strain until 100% strain. Visibly distinguishable
 561 differences in pore sizes are observed from strains greater for 30%. Micro-cracking and damage
 562 of the cell walls is also observed for strains greater than 30%.

563

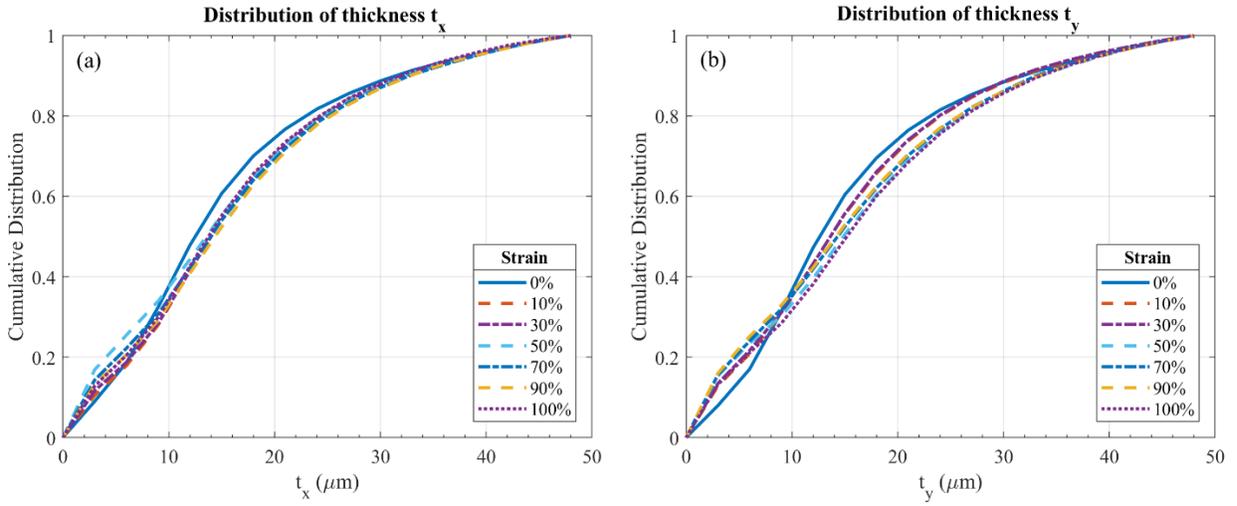


564

565 Fig. 6: Cumulative Distribution Function (CDF) plots of pore sizes shown for strain increments

566 of 10% until 100% for quasistatic tension of D3O LITE D foam for both x and y directions.

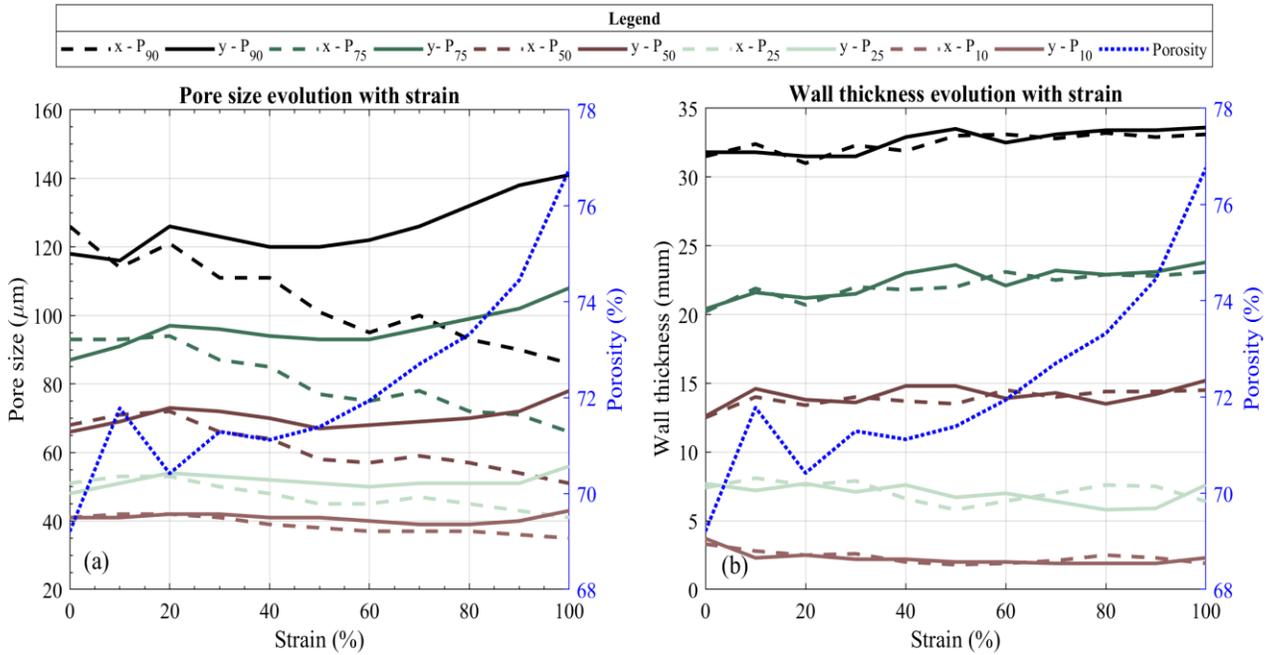
567



568

569 Fig. 7: Cumulative Distribution Function (CDF) plots of wall thickness shown for strain
 570 increments of 10% until 100% for quasistatic tension of D3O LITE D foam for both x and y
 571 directions.

572



573

574 Fig. 8: Compiled percentile plots of pore size and wall thickness shown for 10th, 25th, 50th, 75th
 575 and 90th percentile in both directions for quasistatic tension of D3O LITE D foam. Plot also shows
 576 evolution of porosity with strain.

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580

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