

Influence of Cell Size on Re-Entrant Transformation of Negative Poisson's Ratio Reticulated Polyurethane Foams

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Abstract

Several foams of different cell-size, including Scott Industrial polyurethane foam with large cells (20 pores per inch, ppi, or 1.2 mm per pore, black), medium cells (65 ppi, or 0.4 mm per pore, green), and near-microcellular (100 ppi, 0.25 mm per pore, white), were processed over various time and temperature regimes to ascertain the role of cell size in transformation to negative Poisson's ratio materials. These foams were transformed successfully, and exhibited negative Poisson's ratio behavior. Poisson's ratio was measured using a new laser based setup. For all as-received (unprocessed) foams with different cell sizes, Poisson's ratio decreased with compressive axial strain and increased with tensile strain up to a maximum. The maximum Poisson's ratio in tension decreased as cell size increases. The strain at which maximum Poisson's ratio occurs, increased with cell size. In negative Poisson's ratio foams, minimum Poisson's ratios of -0.8, -0.5, and -0.4 for 20 ppi, 65 ppi, and 100 ppi foams, respectively were observed. Furthermore, the cell size effects on transformation parameters were also found.

1. Introduction

Negative Poisson's ratio, the transverse expansion of a material under tension, is counterintuitive but nevertheless possible. Theoretically, the allowable range of Poisson's ratio for isotropic materials is between -1 and +0.5 based on energy considerations of materials' intrinsic structural stability [1]. Historically, negative Poisson's ratio materials were reported by A. E. H. Love [2] in certain anisotropic single crystal iron pyrites. The first man-made isotropic materials with negative Poisson's ratios were presented by Lakes [3]. Many follow-on researches have been made by the inventor and other researchers [4,5,6,7] to explore the intrinsic properties of the

negative Poisson's ratio materials. The properties of polymeric and metallic foams with negative Poisson's ratios were presented by Choi and Lakes [5, 6]. Their results show the Scott industrial foam, mostly 20 pore per inch (ppi) polymeric polyurethane open cell foams, can be transformed to have a minimum Poisson's ratio of -0.8, and copper foams can be transformed to achieve a minimum Poisson's ratio of -0.4 to -0.8. In addition, by using a reverse method to make a pre-tension strain field instead of a pre-compression strain field in the transformation process, anisotropic foam materials with Poisson's ratio greater than 1 can be created [8]. The most important physical parameters to dominate the negative Poisson's ratio transformation are processing temperature, time, and volumetric compression ratios. The experimental relation between the processing temperature and the foam's softening point has been reported [7]. However, the exact relation between these three parameters to achieve successful transformation is still not known for a specific foam.

Deformation mechanisms of cellular solids, presented by Gibson and Ashby [9] suitably account for the mechanical properties of two and three dimensional cellular solids without introducing cell-size parameters. The structural deformation mechanism of negative Poisson's ratio materials was analyzed by introducing the concept of re-entrant cell structure and non-affine deformation [6]. There is no characteristic length scale in the theory of elasticity, therefore the positive or negative Poisson's ratio cannot depend on the presence of a coarse microstructure such as that present in the foams studied previously. As with foams of conventional structure, there is no predicted effect of cell size. Even so, processing variables may be important in foams with respect to different cell sizes. In particular, a small cell size is associated with a correspondingly large surface area per volume in the cellular solid. Surface tension effects can be important in such cases. The processing characteristics may therefore depend upon cell size. The motivation of this research is that seat cushion foams often have a smaller cell size than the Scott industrial foams studied previously. Therefore, a further understanding of cell size influence is crucial to apply current knowledge of negative Poisson's ratio foams toward applications. In addition, the improved laser Poisson's ratio measurement setup was developed and adopted to obtain accurate and reliable experimental data.

In this article we present the detailed experimental procedures of thermal transformation processes, descriptions of the laser measurement system, the corresponding measurement procedure, with the aim of determining optimal transformation parameters. Experimental Poisson's ratio vs. strain results for the conventional and the negative Poisson's ratio open-cell foams with different cell sizes, including small pores, are also presented. Furthermore, the implication and role of cell size are discussed.

2. Experimental

2.1 Sample Preparation

The foam materials (from Foamade Industries, Auburn Hills, MI, USA) were stored in room environment (21~25°C, 40~50 % relative humidity). For the as-received (unprocessed) foam samples, the density of 20 ppi ones was 0.03 g/cm³, that of 65 ppi ones was 0.03 g/cm³, and that of 100 ppi ones was 0.033 g/cm³. In order to obtain suitable sample size from the original block of foam materials, two cutting methods were used. One was a hot wire saw: a 28-gauge steel wire, heated with an electrical power supply. In this approach the heat of the wire due to electric current melted a path through the foam [4]. The other method was to use a 9" band saw, manufactured by Pro-Tech. The latter method was used extensively in the present research.

Based on previous research by Choi and Lakes [5], for obtaining successful transformation, the volumetric compression ratio (VCR, the ratio of initial volume to final volume) of the foam samples should be in the range of 2 to 5. Therefore, the initial sizes of the samples for experiments were dominantly determined by those of the molds. A typical mold is shown as

Figure 1. To obtain transformed foams with isotropic properties, the compression ratio in each dimension of the sample should be close to the linear compression ratio (LCR), which is equal to the cubic root of VCR.

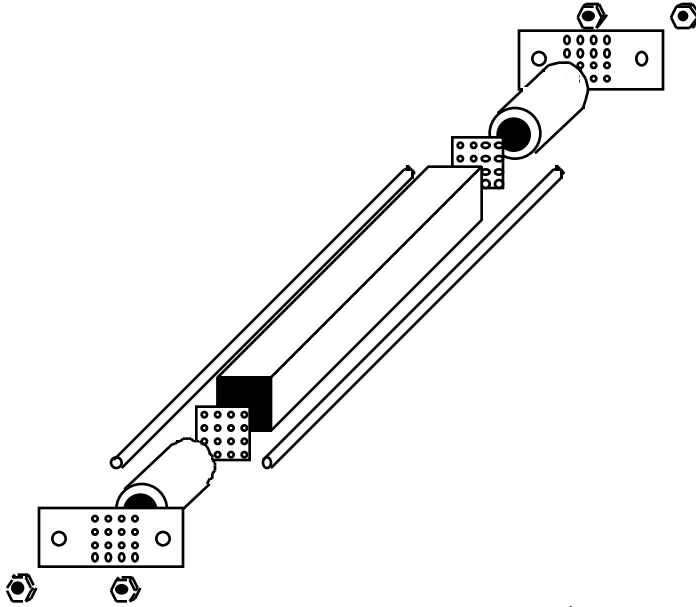


Fig. 1: Mold configuration (adapted from Brandel [4])

Use of tongue depressors to condition the foam in a mold reduces the problem of non-homogeneous compression and wrinkles on the surface. In addition, the spacers (two short metal tubes) were used to achieve a tri-axial compression strain in the sample. A short settling time (usually 10 minutes) was used to allow the strain to be re-distributed uniformly before starting the thermal transformation process.

2.2 Thermal Transformation Process

The negative Poisson's ratio behavior is a consequence of the cells forming re-entrant cell structure [6]. The principle behind creating the re-entrant cell shape foams is to transform the convex cell shape of conventional foams to a concave or re-entrant shape through triaxial compression and heating. The detailed procedure of transforming 100 ppi polyurethane foams in this study was as follows. The transformation for 20 ppi and 65 ppi foams were done by the same procedure with different processing parameters.

A predetermined temperature and time were chosen before placing the sample into the furnace. This temperature has to be equal or slightly greater than the material's softening point [8] for successful transformation. Based on experience with Scott industrial foam, the trial temperature in the present work started from 120°C. Many tests were conducted at different values of time and temperature to obtain the optimal parameters.

After the transformation process, the sample was removed from the furnace, immediately removed from the mold, then cooled at room temperature in air on a wooden desk. Then, the samples were collected at different predetermined temperature and time to find the optimal parameters by screening laser measurement tests. The screening test was, following the measurement process (describe below), to measure the Poisson's ratio at -0.15 engineering axial strain.

2.3 Measurement Process

The laser method for measuring Poisson's ratio is adapted and modified from that of Kugler, Stacer, and Steimle who studied the Poisson's ratio in elastomers [10]. They discussed the importance of accurate measurement for finite element numerical computation since Poisson's ratio values for elastomers in the difference of 0.49 and 0.4999 can lead to 40% difference in stress predictions. A simplified laser measurement system was set up [4] in our lab. Our laser set-up has been proven to provide sufficiently high accuracy in Poisson's ratio measurement [4,10]. The laser apparatus for measuring Poisson's ratio is shown in Figure 2. The spirit of the method is to measure the laser power by the light detector, and convert the change of the laser power to the Poisson's ratio at each axial strain step. In Figure 2, the position of the calibration baffles was aligned up to the front of specimen to simplify data reduction. The measurement system is calibrated with a micrometer controlling the upper baffle, and thus the relationship between transverse displacement and laser power is determined. Figure 3 shows typical calibration curves.

In the laser set-up, the spacing between each component may be freely chosen. The spacing choice is governed by the size of the specimen and the need to reduce ambient and diffusely reflected light, considered as noise. Therefore the distance between focus lens and sample was kept short. In addition, the upper half power reading (center between the baffles to the bottom end of the upper baffle) was adjusted to be equal to the lower half power reading such that the derivatives of calibration curves have the same meaning with respect to the transverse deformation. The distance between the iris and light detector was adjusted to be as small as it could be in order to reduce the background light noise. Moreover, room lights were dimmed during the tests. Typical background noise detected by the light detector was 2% of the signal at each incremental axial strain step for small axial strain range. Away from zero axial strain, the noise percentage dropped to 0.2%, since more laser light was transmitted.

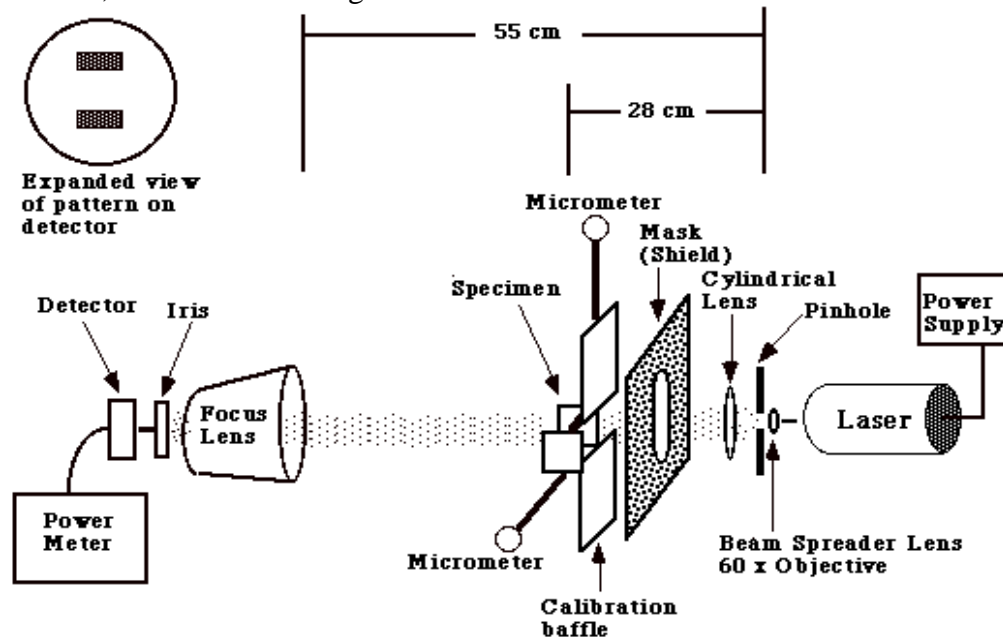


Fig 2: Poisson's ratio laser measurement set-up.

Calibration Curves: Laser Method Upper and Lower

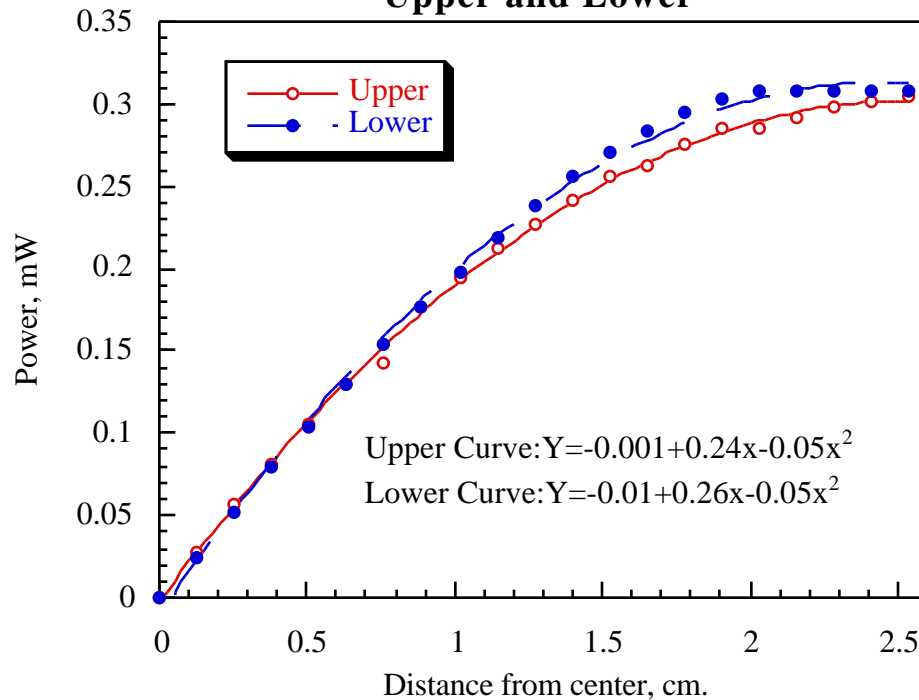


Fig 3: Characteristic calibration curves of the laser measurement system. Upper refers to upper baffle.

As for tension tests in the laser set-up, aluminum plates with screws were used as holders to attach specimen ends. There were two small pieces of acrylic plates mounted between sample and aluminum plates for keeping the integrity of the sample after detachment. The interfaces between any two materials were glued with cyanoacrylate. The aluminum plates, then, were tightened with screws on a loading device controlled by micrometers. In addition, the aluminum plates were sprayed black to reduce the optical noise influence.

The experimental procedure was conducted in the room environment (21~25°C, 40 ~ 50 % RH). Two marks were made on the upper surface of the sample to define the axial gauge length, typically 2 cm. The transverse width, defined as TW, under stress-free status was measured. The sample was mounted into the center of the holders with a minimum pre-stress, and adjusted so that the upper and lower power readings were equal with zero or minimal stress in the sample. The distance between the top of the sample and the free end of the upper baffle, defined as UD was measured. The distance between the bottom of the sample and the lower baffle, as LD, was computed. LD was equal to 5.08 cm (2 in, the distance between the ends of the baffles) minus TW and UD. Since the calibration curves are not linear, the variables UD and LD are crucial to determine the corresponding slopes on calibration curves to convert laser power difference to the transverse displacement. The axial gauge length (L_0), was measured manually by a caliper. Axial engineering strains of ± 0.025 , ± 0.05 , ± 0.075 , ± 0.1 , ± 0.125 , ± 0.15 , ± 0.175 , ± 0.2 , ± 0.225 , ± 0.25 , ± 0.275 , ± 0.3 , ± 0.325 , ± 0.35 , ± 0.375 , ± 0.4 , ± 0.425 , ± 0.45 , ± 0.475 , ± 0.5 were typically applied. Positive signs denote tension. The maximum and minimum axial strains were limited by the apparatus, specifically the range of the micrometer screw, and by the buckling of samples in compression tests. The sample was strained to each strain level. The laser power readings were taken 5 seconds after the strain was completed, and the sample was strained to the next desired status 1 minute after the previous strain was started. The axial strain

rate at each strain level was approximately 0.025 in 10 seconds. The compression and tension tests were done separately. The waiting time between any two tests on the same sample was set at least 30 minutes to allow for viscoelastic recovery.

The mathematical relationship between Poisson's ratio and the upper, lower, and total power readings is shown below. The power (mW) of the upper light beam per transverse displacement (cm) is denoted as S_U , which is the slope of the upper calibration curve corresponding to the point UD away from the origin on the horizontal axis, and for the lower beam, denoted as S_L , obtained through the variable LD using the same procedure as the upper beam. The S_U and S_L are computed from the derivatives of the calibration curves, and the S_T is the average of S_U and S_L . P_U , P_L and P_T are defined as upper, lower, and total power reading. Theoretically, P_T is equal to the sum of P_U and P_L . Then, the total transverse displacement was calculated as follows.

$$\text{transverse} = \frac{P_L}{S_L} + \frac{P_U}{S_U}, \text{ or} \quad (1)$$

$$\text{transverse} = \frac{P_T}{S_T} \quad (2)$$

The differences of transverse displacement from the above two equations are small. This difference is an indication of the error in the laser system from the laser itself. The possible error sources come from the fluctuation of the readings and the misalignment between upper and lower light beams before straining the sample. Finally, the Poisson's ratio, ν , can be computed as following.

$$\nu = -\frac{\text{transverse}}{\text{axial}} = -\frac{\text{transverse}}{L_0 \times TW} \quad (3)$$

3. Results and discussion

To explore the properties of small cell negative Poisson's ratio foams with respect to different processing time and temperature, the Scott reticulated 100 ppi white foam was used in the screening tests to find the optimal processing parameters. Similar Scott polyurethane foams are available in pore sizes from 10 ppi to 100 ppi. Screening tests were performed controlling the volume compression ratio to be about 2.5, and the Poisson's ratio was recorded at an engineering axial strain, -0.15. The rationale for choosing -0.15 axial strain is that experimentally significant negative Poisson's effects can be detected with minimal error due to noise. In the screening tests, the processing temperature was from 120°C to 280°C, and the processing time was from 5 to 21 minutes. The results are shown as Figure 4. As seen in the diagram, the optimal time was 8 to 12 minutes, and the optimal temperature was 210 to 230°C. Figure 5, which is the contour map of the data shown in Figure 4, further elucidates the optimal transformation range. Comparing to the optimal transformation parameters for 20 ppi (170°C, 17 minutes)[3] and 65 ppi green (190°C, 13 minutes) foams, it shows smaller cell size foams prefer higher temperature and shorter processing time. This could be due to the role of surface tension in foams of smaller cells. It is also possible that there is some difference in the chemistry.

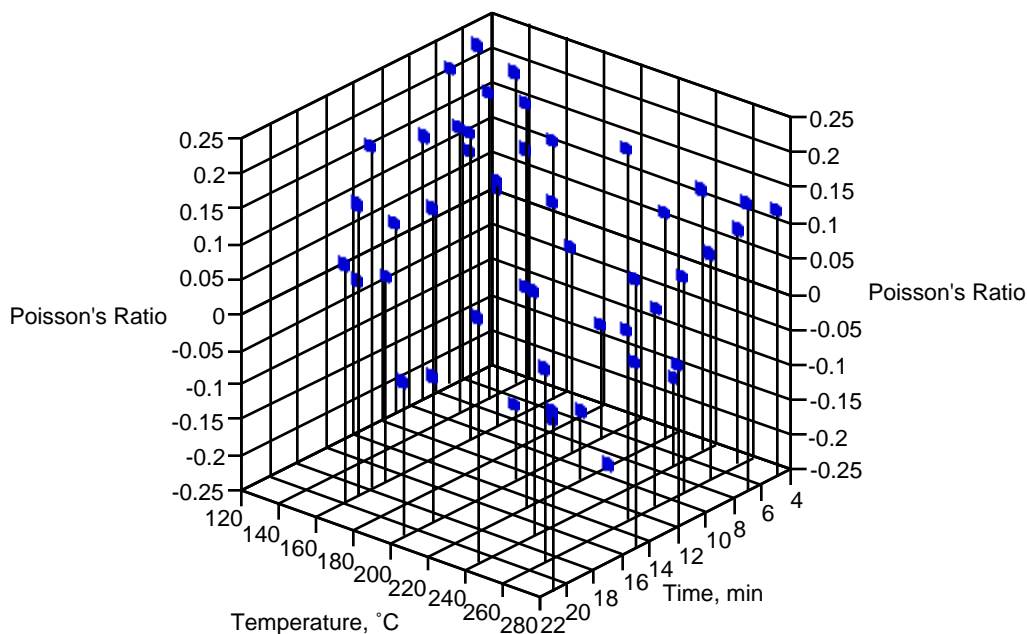


Figure 4: Screening test results of 100 ppi polyurethane foams. Strain = -0.15, Volumetric compression ratio = 2.5, Total 48 samples.

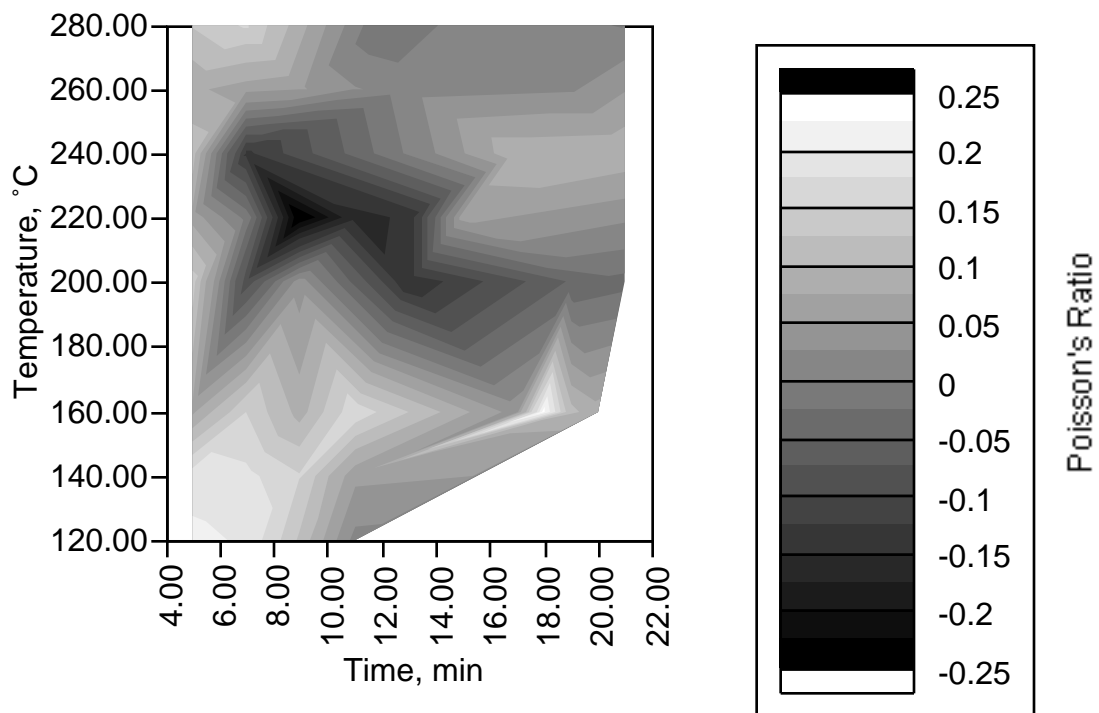


Figure 5: Contour map for screening tests of 100 ppi polyurethane foams. Strain = -0.15. The darkest area represents optimal region for creating negative Poisson's ratio foams.

Plots of the Poisson's ratio for both the unprocessed and transformed foam processed with optimal parameters with different cell sizes are shown in Figures 6 and 7. Figure 6 shows the repeatability test results of experimental data with error bars for the same 100 ppi foam. Maximum compressive strain for unprocessed or processed foam was limited by buckling. The error bars for small axial strain become larger since transverse displacement becomes comparable to the

resolution in displacement. Figure 7 shows the comparison of data for different cell size foams. Previous study on polyurethane Scott industrial foam [3,5] disclosed a minimum Poisson's ratio for 20 ppi processed foams of -0.6 to -0.8. However, the minimum Poisson's ratio of -0.3 to -0.4 was found for 100 ppi foam, and -0.4 to -0.5 for 65 ppi foams in this study. This suggests the negative Poisson's ratio effects decrease as the cell size decreases. However the foams may differ in ways other than cell size. The reason is that cell size is controlled by modifications in constituent concentration during the manufacturing process. Such modifications are certainly capable of altering the transformation characteristics. It is also observed that for unprocessed foams the peak Poisson's ratio shifts left (toward smaller engineering axial strain) when cell size decreases as shown in Figure 7. Peak Poisson's ratio can exceed 0.5 since the large deformation elongates the cells and consequently induces anisotropy.

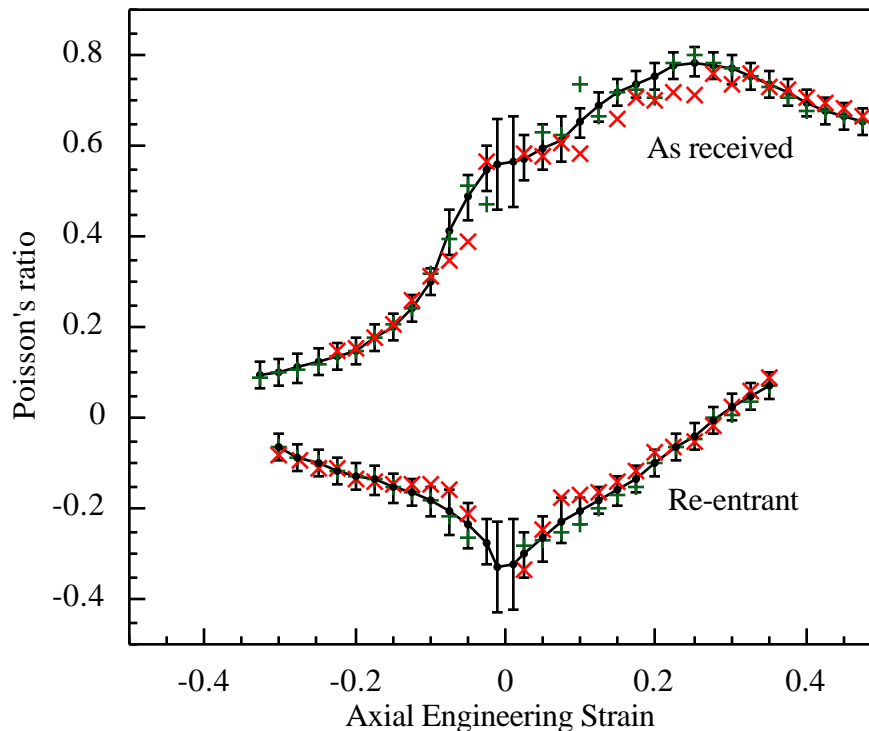


Figure 6: Repeatability test for the 100 ppi foam. Solid line is provided as a guide to the eye. Cross and plus symbols denote two different tests on the same sample and direction. The upper curve represents the unprocessed (as received) foam; lower for the transformed (re-entrant) foam.

In prior research on the negative Poisson's ratio foams, Choi and Lakes [5] determined the relationship between Poisson's ratio and longitudinal engineering strain for conventional and transformed Scott foam as replotted in Figure 8. Comparing Figure 7 and 8, the difference on the tail behavior of transformed 20 ppi foams may be due to the different volumetric compression ratios and their chemistry.

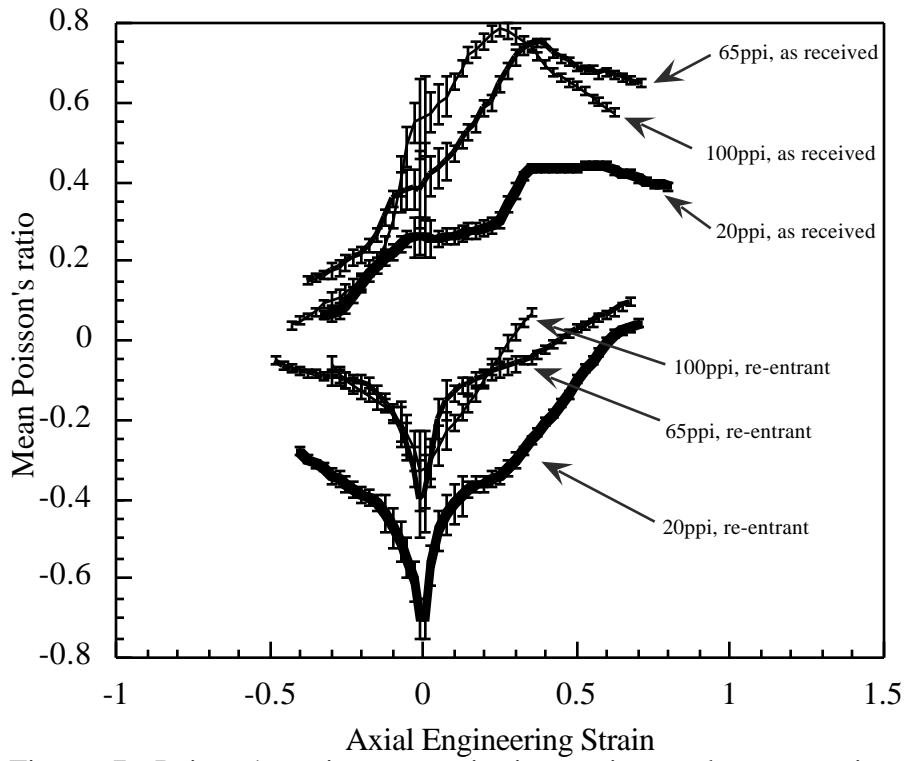


Figure 7: Poisson's ratio vs. strain in tension and compression for processed and unprocessed foam of different cell sizes: 20 pores per inch, ppi, or 1.2 mm per pore; 65 ppi, or 0.4 mm per pore; 100 ppi, 0.25 mm per pore.

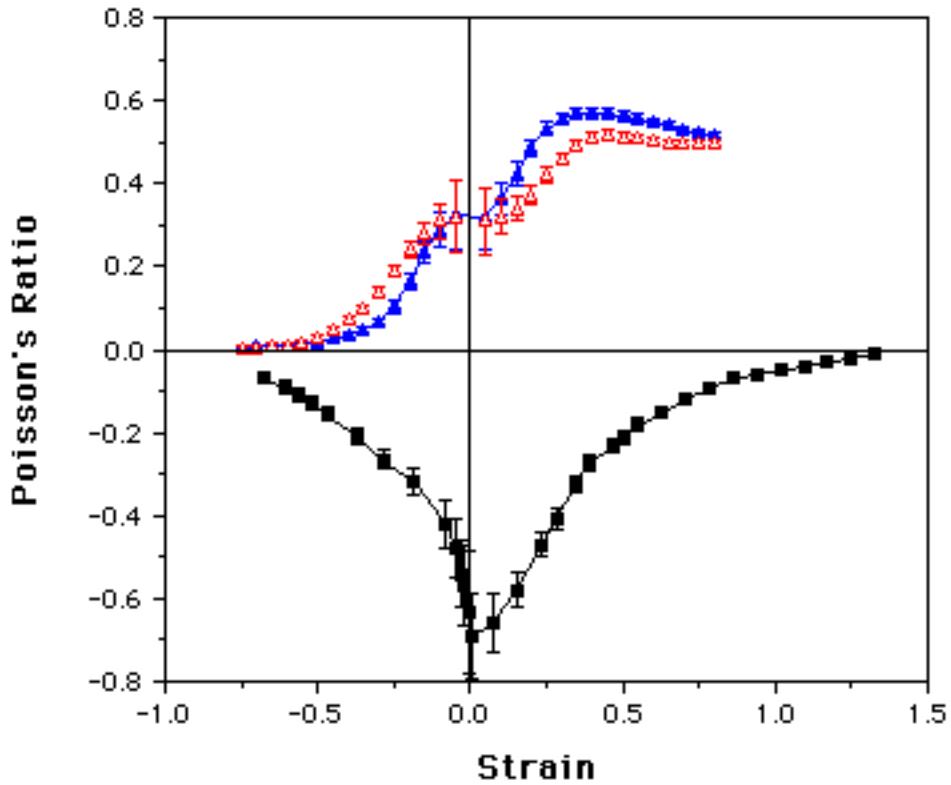


Fig. 8: Poisson's ratio vs. strain: Polyurethane Scott foam [5]. 20 pores per inch, ppi. Open and closed triangles are unprocessed (as received) foams, closed square is processed with a volumetric compression ratio of 3.2.

Figure 9 (a) to (f) show the optical microscopic pictures of the transformed foams studied in this paper, in which the volumetric compression ratios are 2.02, 2.77, and 2.47 for the 20 ppi, 65 ppi, and 100 ppi foams, respectively. The broken ribs are due to the cutting from the bandsaw. However, several broken ribs on the surfaces do not change the foams' overall bulk properties. The reentrant cell structure can be readily seen in these pictures. Transformation of even smaller cell size foams, such as microcellular foams, may be possible. Microcellular foams commonly refer to thermoplastic foams with a cell size in the range of 0.005 to 0.05 mm per pore. Surface tension effects may exacerbate adhesion of foam ribs at high volumetric compression ratios.

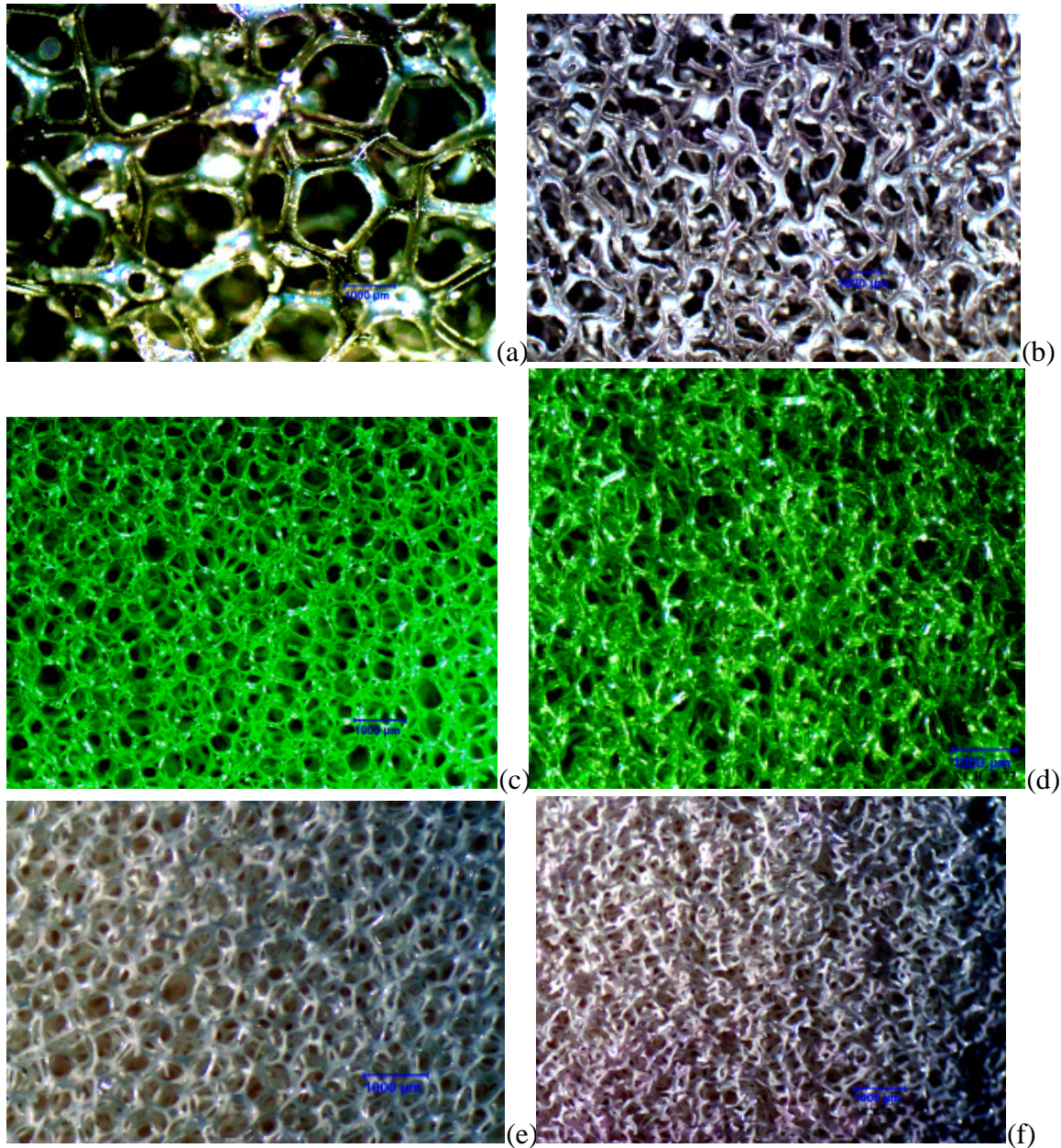


Figure 9: Microscopic pictures for the (a) unprocessed 20 ppi foam, (b) transformed 20 ppi foam, (c) unprocessed 65 ppi foam, (d) transformed 65 ppi foam, (e) unprocessed 100 ppi foam, and (f) transformed 100 ppi foam.

4. Conclusions

1. The laser measurement system provides accurate Poisson's ratio of materials at different axial strains. Since Poisson's ratio is a ratio of strains, error bars are most prominent for the smallest strains.
2. The three different cell-size, polyurethane open-cell foams were transformed successfully to negative Poisson's ratio foams via a thermal processing procedure. For the larger cell-

size 20 ppi foam, the optimal parameters were 170°C and 17 minutes. This gave a minimum Poisson's ratio of -0.8. For the medium cell-size 65 ppi foam, they were 190°C and 13 minutes with a minimum Poisson's ratio of -0.5. For the small cell-size 100 ppi foam, they were 220°C and 10 minutes with a minimum Poisson's ratio of -0.4.

Figure captions

Fig. 1: Mold configuration (adapted from Brandel [4])

Fig 2: Poisson's ratio laser measurement set-up.

Fig 3: Characteristic calibration curves of the laser measurement system

Figure 4: Screening test results of 100 ppi polyurethane foams.

Strain = -0.15, Volumetric compression ratio = 2.5, Total 48 samples.

Figure 5: Contour map for screening tests of 100 ppi polyurethane foams. Strain = -0.15. The darkest area represents optimal region for creating negative Poisson's ratio foams.

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Figure 7: Poisson's ratio vs. strain in tension and compression for processed and unprocessed foam of different cell sizes: 20 pores per inch, ppi, or 1.2 mm per pore; 65 ppi, or 0.4 mm per pore; 100 ppi, 0.25 mm per pore.

Fig. 8: Poisson's ratio vs. strain: Polyurethane Scott foam [5]. 20 pores per inch, ppi.

Open and closed triangles are unprocessed (as received) foams. Closed squares represent foams processed with a volumetric compression ratio of 3.2.

Figure 9: Microscopic pictures for the (a) unprocessed 20 ppi foam, (b) transformed 20 ppi foam, (c) unprocessed 65 ppi foam, (d) transformed 65 ppi foam, (e) unprocessed 100 ppi foam, and (f) transformed 100 ppi foam.

Acknowledgment

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