Studies of ReCrete: A polyurethane foam

Robert B Mohan
University of Nevada, Las Vegas

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STUDIES OF RECRETE: A POLYURETHANE FOAM

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Entitled

Studies of ReCrete: A Polyurethane Foam

is approved in partial fulfillment of the requirements for the degree of

Ph. D., Mechanical Engineering

By

Examination Committee Chair

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ABSTRACT

Study of ReCrete: A Polyurethane Foam

by

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Polyurethane foam is often molded directly in place as a thermal or vibration insulator, energy absorbing material, or core material for a sandwich structure. A smooth thin skin forms between the mold and the interior cellular structure of the foam. A non-uniform microstructure is often visible when foam cross-sections are examined, resulting in density variations throughout the foam and the resulting mechanical property variations. This study investigates the effect of mold temperature and mold size on the average density and some of the resulting mechanical and thermal properties for a polyurethane foam system molded in aluminum cylinders and boxes. Resulting properties are also compared to more uniform foam samples cored out from larger molds. It is shown that the molding process, temperature, and size have a significant effect on the foam density. The density, in turn, affects the quasi-static and dynamic mechanical properties, as well as thermal and chemical properties.

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INTRODUCTION

Rigid polyurethane foams can be used for preserving sensitive electronic components from shock and other dynamic forces. Toluene Di-Isocyanate (TDI) foam has been used for many years in Department of Energy (DOE) applications. [1] They are hazardous to handle during the foaming process, so new foams are being investigated to reduce the health risk for manufacturing workers. Several foams that are safer to produce are being studied as potential replacements for TDI-based foams. [2,3] They must have equivalent mechanical properties such as strength, stiffness, and energy absorption, and they must also demonstrate similar or better aging characteristics with respect to these properties. ReCrete polyurethane foam is one candidate replacement material for BKC44402, a common TDI foam. [4]

1.1 Purpose of the Study

It is important to be able to predict the mechanical behavior of molded polyurethane foam under anticipated static and dynamic loads because there is a significant variance between each individual foam component and because the measurement of mechanical properties is a destructive process.

These predictions must be made from information that is available at the time the foam component is molded and from non-destructive measurement processes in
combination with predictive models based on empirical data gathered from previous mechanical testing.

**MOTIVATION**

- It is important to be able to predict the mechanical behavior of molded polyurethane foam under static and dynamic loads
- The prediction must be made from information available when the foam is molded
  - Chemistry
  - Morphology
  - Density and Density Distribution

Figure 1-1 Motivation

Therefore, the purpose of this study is to develop a predictive relational model that uses measurable physical properties such as chemistry, morphology, and density to predict mechanical responses of foam components to static compression and flexural bending loads as well as dynamic compression and flexural bending loads. This model will support prediction for foam components of varying sizes and for foam fabrication under varying processing temperatures.

**MISSION STATEMENT**

Develop a predictive relational model that uses measurable physical properties, such as chemistry, morphology, and density, to predict mechanical responses of the foam to static and dynamic loads.

Figure 1-2 Mission Statement
1.2 Significance of the Study

The Department of Energy (DOE) has an ongoing project to prepare and to test replacement foams for the national nuclear stockpile based on polyurethane chemistries, but containing environmentally friendly materials. DOE has provided samples of several thermally aged and normal foams that the University of Nevada, Las Vegas (UNLV) personnel used for initial experiments.

DOE provided UNLV with samples of 2 types of polyurethane foams, ReCrete and BKC44402, which is based on toluene diisocyanate (TDI) and is found in many DOE-related applications. Polymeric foam materials are used for structural supports, thermal insulation, vibration damping, impact mitigation and shock wave mitigation. These materials are designed to have high service temperatures (approaching 200°C) and long service lifetimes. BKC44402 has been used routinely for over 25 years. These foams are used for a variety of reasons; they may be used simply as volume fillers or to afford electrical isolation. In other applications, however, they may be required to mitigate vibration or impact. As such, changes in the mechanical properties of these foams as they age in the stockpile may affect their performance. Notwithstanding this, there is little documented information describing size and temperature induced changes in the mechanical and physical properties of these foams.

The foam is inherently viscoelastic because it is comprised of a polymer material. As such, its mechanical properties will depend on both the imposed strain rate, as well as test temperature. Some accelerated aging studies have shown that these polyurethane foams can suffer changes in their viscoelastic characteristics as a result of laboratory aging at elevated temperatures. However, it is unclear if there exists any relationship between
these dynamic property measurements and the more conventional kinds of physical and mechanical properties (Young's modulus, collapse strength, toughness, etc.) that a designer might use in specifying a particular foam or set of performance requirements.

Principals in the UNLV foam project received samples from collaborators at DOE for assessing the degradation mechanism of BKC44402 and its proposed replacement, ReCrete. The foam project team at UNLV has also established a ReCrete production capability, including the equipment and expertise to formulate any amount of ReCrete needed. Using a number of mechanical and spectroscopic tests, UNLV scientists and engineers showed distinct and dramatic differences in the ways these materials cure under temperature, including different changes in the chemistry in different regions (walls and struts) of individual foams.

1.3 Research Questions and Hypotheses

A major objective of the UNLV foam program is to understand the limitations of polymeric foams under the adverse conditions documented by DOE by studying effects of processing temperature and component sizes of foams on their resulting "service" properties, including mechanical and thermal properties. Therefore, the following research questions are posed as potential avenues of investigation for this study:

(1) What is the relationship between dynamic property measurements (impact energy absorption, impact crush strength) and the static physical and mechanical properties (Young's modulus, collapse strength, fracture strength, etc.) that a designer might use in specifying a particular foam or set of performance requirements for foam?
(2) It is not yet clear if there exists any relationship between dynamic property measurements and the more conventional kinds of physical and mechanical properties (Young's modulus, collapse strength, fracture strength, etc.) that would be useful to engineers during the design of a foam-based product. Can the experimental property data be used in a constituent material model to predict a structural response to some types of dynamic loads more representative of real-world engineering requirements?

1.4 Terms of Reference

The following terms of reference establish a common foundation of understanding for the foam project team as well as the readers of this and other foam project study reports.

Closed-Cell, Rigid Foam – Material made up of a network of solid struts and plates which form the edges and faces of cells. Solid faces isolate the cells of a closed-cell foam. Cells are formed by a blowing agent (carbon dioxide). Strut bending, face stretching, and compression of the blowing agent are mechanisms of foam strength. Plastic buckling and brittle fracture are mechanisms of compressive strength for rigid foams. Rigid foams have high toughness but low resilience. [5,6,7] Rigid foams can exhibit either elastic-brittle behavior as depicted in Fig. 1-3 and Fig. 1-6, or elastic-plastic behavior as depicted in Fig. 1-4 and Fig. 1-5.

Relative Density (p*/ps) – Density of the cellular material divided by the density of the solid from which the cells are made (polyurethane). Also referred to as normalized density. [8,9] Normalizing is necessary in order to compare foam samples of different densities.
Structural Anisotropy (R) – Dependency of physical and mechanical properties on orientation of the material, as a result of elongation of cells in the rise direction [10].

Cell Geometry – Cells are composed of vertices, edges, and faces. Common geometric models include cubes, rhombic dodecahedrons, pentagonal dodecahedrons, and icosahedrons. [11] The ReCrete mean cell diameter is 390 um [12].

Cream Time – Time from the start of mixing to the start of foam rising (5% of final height) [13].

Rise Time – Time from the start of mixing to the end of foam rising (95% of final height). Also referred to as blowing reaction time. [14]

Gel Time – The curing reaction time of the polymer [15].

Rise Time = Gel Time – A polymer foam formula is optimized when the rise time equals the gel time. The formula is manipulated by changes in the proportional amount of the blowing agent and the catalyst. [16]

Young’s Modulus (E) – The initial slope of the stress-strain curve in the elastic region. The modulus of rigid foam is dominated by elastic bending of cell struts. [17,18,19]. See Fig. 1-3 through Fig. 1-6.

Fracture Strength – For rigid foams, the stress at which the cells rapidly fail beyond the linear-elastic regime. The point at which the cell struts transition from bending (stretching) to brittle fracture. The tensile failure mechanism is the propagation of a single crack [20]. Also referred to as ultimate tensile strength [21]. See Fig. 1-3 and Fig. 1-4.
**Toughness** – Energy absorption per unit volume, calculated by measuring the area under the compressive stress-strain curve. Toughness is a product of strength and ductility [22]. See Fig. 1-5 and Fig. 1-6.

**Plastic Yield Stress** – For rigid foams in compression, the stress at which the cells begin to collapse plastically beyond the linear-elastic regime. The point at which cell struts transition in compression from bending to buckling [23]. See Fig. 1-5 and Fig. 1-6.

**Collapse Strength** – Compressive stress at the plateau region of a compression stress-strain curve, associated with the brittle crushing of cell struts in a rigid foam.
Also referred to as the Euler buckling stress. [24,25] See Fig. 1-5 and Fig. 1-6.

**Densification** — Region of rapidly increasing stress, to a slope of $E_s$, as the cell material itself begins to compress. When the relative density is above 0.3, there is a transition from cellular material to porous material. When the relative density is above 0.5, the material begins to behave as a solid. [26]

---

**Figure 1-5** Compressive Stress-Strain Curve for Elastic-Plastic Foam

**Figure 1-6** Compressive Stress-Strain Curve for Elastic-Brittle Foam

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Uni-axial Compression – A load that causes a body to shorten in the direction of the applied load [27].

Uni-axial Tension – A load that produces elongation in the direction of the applied load [28].

Flexure – The ability of a beam to bend without fracture. Flexural strength is the stress at the outer edge of the beam at the moment of a crack or break [29].

Quasi-Static – Strain rates at or below 1/second. The initial strain rate for ReCrete testing was 1.7x10^{-4}/second. The range of strain rates tested was 1.7x10^{-5}/second to 1.7x10^{-2}/second. For ReCrete, quasi-static strain is limited to 0.20 (20% strain of the test sample). [30,31]

Dynamic – Strain rates above 40/second. The initial strain rate for ReCrete testing was 70/second. Dynamic strain rates are achievable with a drop tower with 1 microsecond resolution. Dynamic strain is limited to 0.60 (60% strain of the test sample). [32,33]

Impact Failure – The point on the stress-strain diagram where the impact load drops to 60% of the peak load [34].

Absorption Energy – Energy that is retained without reflection or transmission on passing through a medium. The transmitted energy is absorbed by conversion into energy of another form, usually thermal. Closed-cell, rigid foam absorbs energy through brittle crushing and collapse of cells. Absorption energy is characterized as the area under the stress-strain curve for foam in tension, as shown in Fig. 1-7, or compression, as shown in Fig. 1-8.
Figure 1-7  Absorption Energy in Tension

Figure 1-8  Absorption Energy in Compression
Endnotes – Chapter 1


[16] Extended Abstract of Whinnery, L.L. et al., p. 3.

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CHAPTER 2

LITERATURE REVIEW

The focus of the literature review was on discovery of previous research that would support two avenues of study. The first was research that related to polyurethane foams with similar characteristics, such as low density, integral skin, rigid structure, and closed cells; and similar applications, such as vibration damping, shock absorption, thermal isolation, and electrical insulation. The second was research that related to similar experimentation, such as static compression, flexural testing, and dynamic impact testing; and modeling processes, such as morphology studies. The following sections summarize the research that the study team found being done by other research organizations, as well as a summary of the research efforts within the study team, leading up to this effort.

2.1 Research - General

The UNLV study team is made up of researchers from the Mechanical Engineering and Chemistry Departments. For purposes of organization, the research efforts of the respective departments are summarized separately.

2.2 Research - Mechanical Engineering

Rigid polyurethane foams are used as a thermal and vibration insulator and energy absorption material. They are often molded directly in place, where a smooth, thin skin
forms between the mold and the cellular structure of the foam. Density gradients and the skin are shown to have an effect on the mechanical properties of the foam.

The study team investigated the effects of processing temperature and mold size on the average density, density gradient, compressive modulus, and compressive strength, for ReCrete; a molded, free-rise, water-blown, polyurethane foam system. Four processing temperatures were used during foam fabrication: 25°C, 40°C, 65°C, and 85°C. Three aluminum cylinder mold sizes were used with diameters of 29mm, 41mm, and 51mm. The properties were also compared to reference samples with a uniform density of 0.101 g/cc. Results showed that processing temperature and mold size have a significant effect on density, density gradients and some mechanical properties. [35]

2.2.1 Foam Density - Introduction

In order to examine the effects of the density and density gradients on material properties, the effect of the manufacturing process on the density gradients also had to be examined. Two aspects of manufacturing were considered: mold geometry and mold processing temperature. Once consistent gradients were formulated, the gradient effects on the material properties could be examined.

2.2.2. Foam Density - Statistical Analysis

The study team characterized the experimental density data for all of the foam samples through statistical analysis. The results are shown in Fig. 2-1. A number of probability distributions were fitted to the data. The best fit was a Weibull distribution, with the minimum, alpha, and beta parameters listed in Fig. 2-1. The x-axis of the histograms is in units of density (g/cc).
To serve as a baseline, density data from the reference foam samples was broken out, plotted separately, and probability distributions were fitted. In this case, the best fit was a Weibull distribution with the minimum, alpha, and beta parameters listed in Fig. 2-2.
The difference in fitted distribution parameters was strong evidence that the reference sample was from a different population than the overall sample of all foam specimens.

Figure 2-3 is a histogram of density data from the foam samples made with a rise temperature of 65°C. This histogram was selected to show two features. The first feature was the clear difference in the distribution of the data from the reference samples. The position of the histogram along the x-axis, and the fitted Weibull distribution parameters both indicated that the two data sets were from different populations, giving strong statistical evidence that density is dependent on rise temperature. The second feature was the bi-modal shape of the histogram. This suggested that there was another independent variable influencing the density. The hypothesis of the study team was that mold size was also affecting density. The data shown in Fig. 2-3 includes density measurements from foam samples from different size molds.

![Histogram of Density for 65°C Foam Samples](image-url)

Figure 2-3  Histogram of Density for 65°C Foam Samples
Figure 2-4 is a histogram of density data from the foam samples made in the large cylinder (1935-mm$^2$) molds. The distribution of this data was substantially different, resulting in an entirely different probability distribution being the best fit. In this case, a Beta distribution was the best fit. This also is evidence that the mold size was an independent factor that affected density.

![Density Distribution Model](image)

**Figure 2-4  Histogram of Density for Large Cylinder Foam Samples**

Only a representative sampling of the statistical analysis results are presented in this chapter for discussion purposes. The complete results of the statistical analysis of density data for different mold sizes and different rise temperatures are contained in a previous report authored by a member of the study team. [36]

### 2.2.3 Foam Density - Density vs. Position

Density was measured as a function of vertical and radial position for each processing method.
2.2.4 Foam Density - Density vs. Vertical Position

For all mold types, density increased from the top of the mold to the bottom. The small aluminum mold showed a modest increase in density of 2.3%. The 1-liter and 4-liter molds showed an increase in density from top to bottom of 5.7% and 9.6%, respectively. [37] Figure 2-5 shows density as a function of vertical position for three mold types. The top sample taken from a mold specimen is identified as level 1. The bottom sample is identified as level 4.

![Density vs Vertical Position](image)

Figure 2-5 Density as a Function of Vertical Position

Figure 2-6 presents the same data as shown in Fig. 2-5 in the form of a bar chart. In this format, Fig. 2-6 shows that density was also a function of mold volume. As the volume increased, the mean density decreased. [38] Figure 2-6 identifies the foam samples from levels 1 to 4 by alternate names: top, middle top, middle bottom, and bottom; respectively.
This trend is also depicted in Fig. 2-7. The density increase as the mold volume decreased (~50%) is more significant than the density increase from the top to the bottom of a given mold volume (less than 10%). [39]
2.2.5 Foam Density - Density vs. Radial Position

Figure 2-8 shows the densities of core samples taken. This figure shows the mean density of the core samples taken from each radial position. The center of the mold specimen was position 1. The core samples from the outer edge of the specimen were identified as position 6 for the 1-liter mold specimens, and position 15 for the 4-liter mold specimens. Figure 2-8 shows the densities from the radial positions of the 4-liter mold, and from 2 levels, top and bottom, of the 1-liter mold. As discussed in the previous section, Fig. 2-8 shows that the bottom level of a mold specimen had a higher density than the top. [40]

![Average Radial Densities for 4-L and 1-L Molds](image)

**Figure 2-8** Density as a Function of Radial Position

Figure 2-8 shows there was a significant increase in density from the center to the outside of each sample. Density gradients for the cylindrical molds showed a trend from
less dense in the center of the samples to more dense on the outside. [41] The 1-liter and 4-liter molds were compared on an absolute scale because samples were taken from the same diameter increments as shown in Fig. 2-8. Due to size differences in the molds, the 1-liter molds had only 6 diameter increments compared to the 4-liter mold’s 15.

The radial positions that are shown in Fig. 2-8 were converted into normalized positions as seen in Fig. 2-9. This was done to provide the relative position of each cylindrical sample in relation to the outside diameter of the respective sectioned mold. By normalizing the position, the density gradient was compared between molds of different sizes. Therefore, the center of each cylinder shown in Fig. 2-9 is denoted as “0” and the outside diameter of each cylinder is denoted as “1”.

2.2.6 Foam Density - Density vs. Mold Size

It is possible to calculate point densities from the aluminum and 1-liter molds up to about 80% of the total mold radius. It is also possible to calculate point densities from the 4-liter molds up to about 95% of the total mold radius. It must be pointed out that for the 1-liter and 4-liter molds only 1 sample was taken from the center. Because of this limited sample size, the measured density at the center was not conclusive. However, the aluminum molds had 4 samples from the direct centers that were averaged. Density increased from the center to the outside for all mold types. The aluminum mold samples averaged an increase in density by 15.3% while the 1-liter and 4-liter molds experienced an increase of 9.3% and 10.6%, respectively. [42]

In Fig. 2-9, the radial positions of the samples were normalized to facilitate comparison of data between the large (1-liter and 4-liter) reference molds and the small aluminum molds.
Differences between mold types was seen when the density was viewed as a function of normalized position, as shown in Fig. 2-9. The 1-liter molds showed an increase in density for every increment from the center outward. The 4-liter molds showed almost constant density for the first seven increments before increasing to the outside density. This behavior was attributed to the larger size of the 4-liter mold; the sides of the mold had less of an effect on the center increments because of the distance between them. The small aluminum molds also showed an increase in density for every increment from the center outward. The density gradient in the small aluminum mold was the largest, indicating that the effects of the sides of the mold increased as the mold volume decreased. [43]

2.2.7 Foam Density - Density vs. Processing Temperature

Figure 2-10 shows the mean density of foam samples formed under different processing temperatures. Figure 2-10 clearly shows the trend of decreasing mean foam density as the processing temperature decreased. In addition to the mean density
indicated by each bar, Fig. 2-10 also shows the variance of the density across the foam samples, indicated by the boxes and vertical lines. Figure 2-10 shows the average density for the foams made under 5 different processing conditions. The foams made in the ice bath had large density variations, as illustrated by the large deviations about the mean. The rectangular boxes and the vertical lines on each bar in the chart indicate the first and second standard deviations, respectively. The figure also shows a trend towards decreasing density with increased processing temperature. [44]

![Density vs. Processing Temperature](image)

**Figure 2-10 Mean Density as a Function of Processing Temperature**

In Fig. 2-10, values inside the box are within ± 1 standard deviation, and values covered by the span of the vertical lines are within ± 2 standard deviations. As the processing temperature increases, the variance of the samples decreases. This indicates that the foaming process is more stable and consistent at higher temperatures. [45]
When compared as a group, the foam processed in the ice bath exhibited the most deviation from the average. This deviation is attributed to the inconsistency of the foam; there were many irregularities found within the samples, such as large air pockets and visually denser areas dispersed randomly throughout each sample. [46] Examples of the voids are shown in Fig. 2-11. These samples had a 2-step foaming process, described in Chapter 3, which could have contributed to this heterogeneity. Figure 2-11 illustrates the microstructure of an ice bath foam sample compared to a sample prepared at room temperature.

![Large Voids](image)

**Figure 2-11 Microstructures For The Ice Bath (Left) and Room Temperature (Right) Processed 28.7-mm Samples (Small Cylinder Mold)**

These observations can be explained by discussing both the effects of temperature on the rate of reaction and the viscosity of the reaction mixture. Differences in density between samples and variations in density within the same sample were highly dependent on processing temperature, where mean densities both decreased and were more consistent as processing temperature increased. Gupta and Khakhar reported a decrease in density as the mold temperature increased while forming integral skin polyurethane foams. [47]
Density trends from temperature effects showed a decrease in density with an increase in temperature. Also, density deviations decreased with increasing temperature. These trends can be explained by analyzing the viscosity of the foaming chemicals. [48] High deviations in the ice batch occurred because the foaming chemicals were more viscous. With colder temperatures, the foaming chemicals were unable to completely fill the cylinders because of the increase in viscosity. Voids were created where the foam could not fill in. These voids create inconsistencies throughout the foam causing deviations in density measurements. Colder temperatures resulted in more dense foam because of the temperature effects on the foaming agent: water. At colder temperatures the water does not introduce air bubbles as easily, preventing the foam from expanding as much as foam under warmer conditions. With the same mass but less expansion, foam created in ice temperatures are denser than foams made in warmer conditions. [49]

Similarly, foam made with a 90°C mold temperature during the first 30 minutes was less dense than foam made in colder temperatures. Decrease in viscosity allowed the foam to expand fully, causing the same mass to expand more than colder batches. The greater expansion caused the foam to be less dense. Density variance within the 90°C batch was small because the decrease in viscosity allowed the foam to fill in all voids. [50] Completely filling in the cylindrical molds made the foam more consistent. For comparison of temperature effects on density and material properties, the room temperature molds, both water and air exposure, were grouped together. Grouping the two batches improved sample size, which in turn better validated the results. The batches showed small enough differences that combining the two was appropriate. [51]
Figure 2-12 shows the radial density gradients of foam samples formed at various processing temperatures. The density gradient is indicated by the change in density from the center of the mold (on the left of the graph) to the outside edge of the mold (on the right of the graph). All sample data shown came from small aluminum molds (80-mL and 93-mL volumes). As indicated by the slopes of the curves shown in Fig. 2-12, the density gradients decreased slightly as the processing temperature increased. Figure 2-12 also indicates how the average density decreased as the processing temperature increased. [52]

![Density Variations for 80 and 93 mL Molds at Different Processing Temperatures](image)

**Figure 2-12 Density Gradients as a Function of Processing Temperature**

As shown in Fig. 2-13, the same density gradient existed regardless of which processing temperature or mold was used. It was also evident that the overall density of foam decreased with increasing processing temperature and increasing mold size. [53]
The dashed line shown in Fig. 2-13 indicates the reference density taken from a reference specimen formed in a 4-liter mold and processed at room temperature. Figure 2-13 shows the mean density and variance for foam samples Small (S), Medium (M), and Large (L) aluminum cylinder molds at 25°C (25C), 40°C (40C), 65°C (65C), and 85°C (85C) processing temperatures. As indicated by the very small boxes and vertical lines at the tops of the bars, the standard deviations of the mean densities were very low.

![Average Density vs Processing Temperature and Mold Size](image)

**Figure 2-13** Mean Density as a Function of Processing Temperature and Mold Size

The study team plotted the density gradients of samples from three different mold sizes (S, M, L), formed at four different processing temperatures (25°C, 40°C, 65°C, 85°C), and compared the density gradients to a reference foam sample. The small physical size of the foam samples prevented the study team from measuring the density in
the outer 10% of the sample. Thus, the graph did not display density data beyond 0.90 of the normalized positions. See Appendix I for details.

The study team found no optimal density for ReCrete. The desired values depended on the application of the material and how the density related to the mechanical properties of the foam. Determining how the processing temperature and size of mold affected the average density of the material was important to its application.

Both increasing the processing temperature and increasing the mold size brought molded ReCrete average density closer to the reference density. The highest average density of 0.15 g/cc was in the 25°C processing temperature, small mold size batch. The lowest average density of 0.106 g/cc was in the 65°C processing temperature, large mold size batch.

Overall, increasing the mold size decreased the average density. The average density of the foam sample took into account the foam and a thin, dense skin layer on the outer edge of the foam. Increasing the processing temperature decreased the average density. Higher processing temperatures showed less variation in the average density between different mold sizes. [54]

2.2.8 Foam Morphology – Foam Qualities

The study team visually inspected all of the foam specimens during manufacturing, immediately after, and then some time later, to observe the physical morphology of the foam (visible to the naked eye) and how the morphology changes over time. The following sections describe general visual observations from batches of foam divided by processing temperature.
(1) 0°C Ice Bath

The ice bath produced foam with numerous irregularities. Large voids, sometimes the full diameter minus the skin, could be found. The bottoms of the molded samples generally exhibited the most/largest voids. Sometimes a denser ring (similar to skin appearance) could be found about 1/8" in from the outside. The foaming process appeared to have two stages in many samples. The appearance was almost like the chemicals “foamed” to a certain point, and then finished in the oven where the temperature was higher. The skin looked more opaque than other higher temperature batches, making the cells harder to see. Many areas in the foam showed significant yellow discoloration. The discoloration was usually inside the ring when the ring was evident.

(2) 25°C Air

The foam skin was clear (cells were easily distinguishable). Foam was mostly consistent in appearance with occasional small voids (1-2mm in diameter). Some yellow discoloration was noted on samples that were 10-14 months old. The discoloration was not consistent within batches, so it was not purely a function of age. The skin was consistent in thickness and opacity. The skin bonded well to the foam core, even after compressive failure of the core.

(3) 25°C Water Bath

The 25°C water bath batches and 25°C air batches were mostly similar. A small difference was in skin appearance. The water bath foam skin was slightly more opaque than the air batches. Older samples showed the same yellow discoloration, but the discoloration was evident around the outer 1/8" of the sample, between the
skin and the ‘ring.’ This was notably different than the foam samples made in the 0°C ice bath, where the discoloration was typically found inside the ‘ring.’

(4) 40°C Water Bath

Foam voids were more frequent and larger (1-7mm in diameter) than the 25°C batches. The skin had an appearance closest to 25°C water bath. On year-old samples, there was some yellow discoloration noted on the skin, but not in the foam core. This was different than both the 0°C and 25°C foam samples that showed discoloration in the foam. The skin was otherwise consistent in thickness and failure mode. In compression-failed samples, the skin showed horizontal collapse lines, with local separation from the foam core.

(5) 90°C Water Bath

Voids in this sample were the most frequent of all the batches. The skin was extremely clear, giving the effect of almost being separate from the core. The average cell size also appeared larger. The skin more easily separated from the foam core because of the large voids at the interface. The foam core, close to the center of the mold, was more normal in appearance, with the largest voids near or at the skin. In some samples, the skin separated from the foam core during removal from the mold, suggesting very poor bonding, something not seen in any samples made at lower temperatures.

2.2.9 Foam Morphology – Foam Cell Microscopy

The study team had six different microscopy capabilities available to study foam morphology. The first was confocal microscopy, which was used to view complex 3-D objects, and provided detailed sectional views within a 3-D object. The second was dark
field microscopy, used to view living cells, where cells appeared as illuminated objects against a black background. Third, was fluorescent microscopy. For this method, the foam samples were dyed. The fluorescent dyes were detected by the microscope and glowed against a dark background. Fourth, was optical microscopy, which used objectives and projected light onto a sample to make fine details visible. Fifth, was phase contrast microscopy, which was also used to view living cells. Finally, there was scanning electron microscopy (SEM), which was used to view 3-D surfaces. With this method, electrons were scattered or emitted from the specimen’s surface to stimulate the scanning electron sensor and create a visual image.

From the six methods available, the test team selected two methods to study foam morphology. The team chose optical microscopy because it used unaltered foam samples, and it was very efficient. Optical images could be taken in seconds. The quality of the images was satisfactory. The team also chose SEM because it provided a clear image of the surface without the interference of multiple layers. Images could be taken in seconds, once the samples were prepared. The test team elected not to use confocal, dark field, or phase contrast microscopy because the quality of the images were not satisfactory. [55] A confocal microscope image is shown in Fig. 2-14.

![Confocal Microscope Image](image.png)
The test team elected not to use fluorescent microscopy because the images were less than satisfactory, and the dying and imaging processes were time-intensive. A fluorescent microscope image is shown in Fig. 2-15. [56]

![Fluorescent Microscope Image](image)

**Figure 2-15** Fluorescent Microscope Image

Optical microscope images range in magnification from 5X to 10X. Foam samples are placed directly on the microscope stage, and are undamaged during the process. Thus, samples can be used for other tests and measurements after imaging, as well as imaged multiple times, as is the case during aging studies. Foam samples must be cut into thin sections, less than 1-mm thick, and have fairly uniform surfaces to provide high quality images. Examples of optical microscope images are shown in Fig. 2-16 and Fig. 2-17. Both images are 5X magnification, but the image in Fig. 2-16 is from a thin foam section, while the image in Fig. 2-17 is from a thicker foam section. The quality of the image in Fig. 2-17 is degraded by the thickness of the section. [57]
SEM images are on a scale ranging from microns (1500X) to millimeters (10X). Foam samples are sputter-coated with 24 karat gold plate before being placed on the manual stage. The stage is capable of holding a maximum sample size of 32-mm. Since the samples are coated with gold, they cannot be used for other tests or measurements after the microscopy and, since the gold coating forms a boundary layer, coated specimens are no longer suitable for further aging. Foam samples can be up to 10-mm thick, and do not have to be completely uniform, in order to provide a high quality image. This makes specimen preparation easier and allows greater latitude in specimen selection. For the best image resolution, SEM spot size is specified at 20. [58] Scanning electron
microscope images are shown in Fig. 2-18, Fig. 2-19, and Fig. 2-20. The image in Fig. 2-18 is 10X magnification.

![Figure 2-18 10X SEM Image](image)

The image in Fig. 2-19 is 100X magnification.

![Figure 2-19 100X SEM Image](image)

The image in Fig. 2-20 is 1500X magnification. The image shows a portion of a strut between cells. Even though ReCrete is considered a closed-cell foam, the image clearly shows that even the cell struts are cellular in nature on a microscopic scale.
SEM and optical microscopes are capable of producing images of sections as large as 7.00-mm x 5.25-mm and 2.2-mm x 1.75-mm, respectively. To completely image a 28.7-mm diameter section of foam, such as the surface of a sample from a small aluminum mold, the team collected 31 images using the SEM microscope, and at least 191 images using the optical microscope. [59]

2.3 Research - Chemistry

As shown previously, processing ReCrete foam at elevated temperature resulted in a decrease of the modulus (stiffness), which the foam chemistry study team believed was related to chemical changes during processing. The chemical composition, density, and modulus of Polyurethane (PU) foam was examined as a function of processing temperature (25°C, 45°C, and 85°C). Degradation of ureteneimine cross-links and the emergence of carbodiimide functional groups were monitored and the remaining composition in the PU foam was determined using Fourier Transform Infrared (FTIR) spectroscopy. The density and modulus of the PU foams after thermal processing were
measured and discussed with respect to the change in chemical composition of the materials. The data indicated that the composition of chemical cross-links, the density, and the modulus all decreased as a function of increasing processing temperature. The modulus of the PU foam showed a linear dependence of the processing temperature decreasing by 20% at the highest temperature. The results suggest that processing temperature may be used to target defined physical and mechanical properties of PU foams without changing the composition of reactants used. [60] The change in modulus was related to thermal decomposition of uretoneimine linkages in the diisocyanate starting material by monitoring the change in intensities of diisocyanate, carbodiimide, and uretoneimine bands in the Infrared (IR) spectra. Differential Scanning Calorimetry (DSC) and Thermal Mechanical Analysis (TMA) were consistent with IR findings, where endothermic and exothermic events could be associated with chemical changes in the foam. [61]

The stability of uretoneimine linkages and unreacted diisocyanate was shown to be affected by the processing temperature and aging of foam made from Rubinate. [62] An approximate 20% decrease in energy absorption and impact strength was observed for aged ReCrete. The aging characteristics may have been a direct result of the decomposition of the uretoneimine linkage prevalent as a major functionality in the diisocyanate starting material in the ReCrete foam (absent in TDI-based foam). Although density gradients were induced in PU foam samples processed at different temperatures, the change in modulus with processing temperature was more likely a result of the breaking of the uretoneimine ring during high temperature (85°C-90°C) processing. The result was foam with a lower degree of cross-linking and with lower rigidity. [63]
Infrared analysis confirmed that foam processed at high temperature exhibited a higher intensity and consequently larger amount of carbodiimide and urethane functionalities relative to unreacted isocyanate groups. In comparison, the foam processed at low temperature showed a larger contribution of uretoneimine C=N vibrations. Infrared analysis of the diisocyanate functional groups in urea was used to examine and identify changes in uretoneimine functionality of Rubinate as the materials were heated. The same phenomena occurred during high temperature processing of ReCrete foam. The same trend was examined for PU foams. The data indicates that processing temperature directly influenced the concentration of the rigid uretoneimine structures in the foam. Thermal degradation of the units in PU foams resulted in the generation of equal molar quantities isocyanate and carbodiimide in the foam. The continued reactivity of isocyanate was observed over a period of 3 months indicating the chemistry of the PU was changing over time. [64]

Thermal analysis of foams processed at room temperature showed a predominant endothermic transition at ~100°C consistent with the temperature range reported for the exchange reaction of the uretoneimine functionality to form isocyanate and carbodiimide. [65]

As time progressed, the 25°C samples exhibited more variation in DSC, TMA, and IR than the 85°C samples because of the initial lower curing temperature. The study team recommended closer inspection of these species because they play a significant role in the stability of foam. [66]

The processing temperature affected the mechanical and chemical properties of ReCrete. It may also affect the shock properties due to the loss of chemical structures
within the polymer matrix. The study suggested that future investigation explore intermediate processing temperatures between 25°C and 85°C, which may identify foams with better mechanical and chemical properties. [67]

2.3.1 Foam Chemistry – Thermal Analysis

TMA testing was used to determine chemical behaviors of the material and mechanical behaviors. Sample sizes for TMA testing were approximately 6.9-mm across and 2.8-mm in height. TMA samples are shown in Fig. 2-21.

![Thermal Mechanical Analysis Samples](image)

2.3.2 Foam Chemistry – Thermal Analysis Characterization

Thermal analysis was determined by DSC and thermal gravimetric analysis (TGA) performed simultaneously using a Netzsch Simultaneous Thermal Analyzer (STA) system. TMA was conducted using a Netzsch TMA 202 instrument. Temperature calibration was performed using an indium standard and height calibration. For the TMA experiments, calibration was performed using silica gel plates.

Four foam cylinders from each batch of foam were used for thermal analysis. From each foam cylinder, 6.90-mm diameter sections were cored from the center and the side closest to the skin edge. All sections were cored in the rise direction with a coring tool.
Two center samples and two side samples were sliced from each of the 4 cylinders, yielding sixteen samples for TMA experiments. All samples had an average height of 2.80-mm. TMA experiments are destructive, so a limited number of tests could be done for each foam batch.

TMA experiments were run from 25°C to 250°C under N₂ gas (50ml/min) with a constant force of 5cN applied to the sample. STA samples of ~5 mg weight were tested in alumina crucibles without lids from 25°C to 540°C at a rate of 10°C/min under N₂ gas (50 ml/min). The average temperature at which the foam collapsed was obtained from the physical alpha curve and the average decomposition temperatures from STA analysis.

2.3.3 Foam Chemistry – Thermal Analysis Results

There were no significant differences in the TGA traces with sampling position and processing temperature and all foam samples exhibited a one-step decomposition at ~300°C, as shown in Fig. 2-22. However, larger variations were observed in the TMA traces.

![Figure 2-22 TGA Curve for Foam Samples Processed at Different Temperatures](image)
On average, all samples showed two expansion/collapse steps and the magnitude of these steps, and the temperatures at which they occurred, could be related to the processing temperature and, in some cases, the sampling position.

2.3.4 Foam Chemistry – Chemistry vs. Processing Temperature

Differences in density between samples and variations in density within the same sample were highly dependent on processing temperature. The average densities decreased and were less variable as processing temperature increased, as shown in the previous section. These density trends were explained by the effects of temperature on the rate of reaction and on the viscosity of the reaction mixture.

Water blown polyurethane foams are formed by the simultaneous reaction of the diisocyanate with the polyol (gel formation), where urethane linkages are formed, and reaction of the diisocyanate with water (gas generation). The gas generation reaction primarily determines the resulting density of the foam. Gas generation produces an unstable carbamic acid intermediate that decomposes, rapidly producing CO$_2$ gas and primary amines. The primary amines further react to form urea linkages, a major component of polyurethanes. [68]

Since all foam samples prepared here were mixed at room temperature, the rate of the gel formation step should be similar. However, the processing temperature was controlled during the foaming step, thus affecting the formation of both CO$_2$ gas and urea linkages. At low processing temperatures, the rate of gas generation decreases, but the viscosity of the solution increases. The decrease in gas generation reduces the even dispersion of gas, which is necessary for uniform foaming, and the diffusion of mixture reagents (E.g., silicone surfactant) that are added to aid in foam stabilization. Both of
these factors were responsible for the increase in foam density and non-uniformity for foam samples prepared at low temperatures. In the case of 0°C processing, the rate of the gas generation reaction was significantly reduced and the major rise of the foam occurred during the 30 minute processing stage and the 4 hour curing stage.

Figure 2-23 General Polyurethane Foam Reactions

Furthermore, it is well established that the ratio of urethane/urea components has a large impact on the resulting mechanical properties of foams. [69] It was expected that the chemical composition of the resulting polymeric foams had different ratios of urethane/urea linkages and, at higher processing temperatures; the formation of allophanate and biuret side reactions was also possible.

The formation of polyurethane foams follows the same general reaction of isocyanate with polyol as shown in Fig. 2-24. The reaction demonstrates the basic structural
backbone of the polyurethane polymer. However, structural cross-links and microstructures within this system can provide additional structure within the polymer matrix. Typical R' groups of the polyol include polyether and polyester units. Industrially important diisocyanates include 2, 4- and 2, 6-toluene diisocyanate (TDI), 4, 4'-methylene diphenyl diisocyanate (MDI), and 1, 6-hexamethylene diisocyanate (HDI). The R groups can be aromatic rings (increase rigidity) or alkyl chains (increase flexibility). More flexible R and R' groups and low functionality of the isocyanate and polyol favors the formation of more flexible foams. In contrast, rigid R and R' groups and high functionality favors more rigid foams. [70]

![Diisocyanate + Polyol](image)

Figure 2-24 General Reaction of Diisocyanate with Polyol to Form Polyurethane Linkages

A method for introducing the uretneimine structure involves the use of liquid 4,4'-methylene diphenyl diisocyanate (MDI), a commonly used isocyanate in polyurethane formation. The modified pure MDI can be reacted to form carbodiimide structures with excess isocyanate in the system yielding a cross linked uretneimine structure with three un-reacted isocyanate groups, illustrated in Fig. 2-25. [71]
This compound mixed with pure MDI results in a liquid carbodiimide modified MDI, which is stable and slightly viscous, with a melting point below 20°C. The uretoneimine thermally reversible four member rings are incorporated into the PU foam matrix as rigid structures that thermally degrade at temperatures greater than 40°C. This temperature dependence can be utilized to adjust the concentration of uretoneimine cross-link structures present in the PU foam. At higher processing temperatures, the thermally unstable uretoneimine degrades, giving equal molar quantities of isocyanate and carbodiimide. The carbodiimide formed from the uretoneimine degradation is a stable functional group that does not react appreciably at higher processing temperature. The result is a high intensity peak at higher processing temperatures that does not diminish in comparison to isocyanate, which can continue to react over time.
2.3.5 Foam Chemistry – FTIR Analysis

Through Photoacoustic Spectrum (PAS) FTIR analysis, the study team observed similar trends in Rubinate MDI and ReCrete PU foam processed at elevated temperatures. The amount of un-reacted isocyanate and the composition of uretoneimine decreased as the temperature reached 45°C. There was little difference in terms of the FTIR response of the 45°C and 85°C samples, indicating that both contained comparable amounts of the un-reacted isocyanate and uretoneimine functional groups. The decomposition of the uretoneimine was verified by the increasing carbodiimide functional groups in the foam. The amount of carbodiimide was fairly small at 25°C, but increased rapidly for the 45°C and 85°C processed PU foams. The study team found that there was a ~45% and ~42% decrease in the uretoneimine group at 45°C and 85°C, respectively. Conversely the opposite trend was observed for the carbodiimide functional group. The presence increased by 90% relative to the initial presence for the carbodiimide species for the PU foam processed at 25°C. [72]

2.3.6 Foam Chemistry – FTIR Imaging

The foam chemistry study team used FTIR imaging from their multi-point detector as a complementary technique to provide the spatial distribution of the chemical characteristic for the ReCrete samples analyzed over a two dimensional array. The spatial distribution of carbodiimide (2110 cm⁻¹) and uretoneimine (1373 cm⁻¹) are extremely important and can be easily identified in two dimensions for the foam samples processed at 25°C and 85°C temperatures. The FTIR images agreed with the data presented previously showing that the uretoneimine structure remained largely intact at the 25°C processing temperature. The images also confirmed single point measurements
that indicated the rigid uretoneimine structure decreased as a function of increasing processing temperature in the struts of the foam. Since the struts are the key to the rigidity of the foam, it is not surprising that the modulus would be affected by the changes in the chemistry of the structure. As uretoneimine structure degrades in the struts the rigidity of the structure decreases and the foam becomes more flexible. [73]


[37] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 68.

[38] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 78.


[40] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 81.

[41] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 79.


[52] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 46.

[53] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 78.

[54] Jackovich, D.J., Formulation of Integral Skin Polyurethane Foams, p. 78.
[55] Nelson, M.C., 2002, “Annual Review Morphology Presentation”, University of Nevada, Las Vegas, NV, p. 2. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.


[57] Nelson, Annual Review Morphology Presentation, p. 3.


[60] Hatchett, D.W., Kodippili, G.C., Kinyanjui, J.M., Benincasa, F., Sapochak, L., 2004, “FTIR Analysis of Thermally Processed PU Foam,” unpublished paper, University of Nevada, Las Vegas, NV, p. 2. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.


[65] Kodippili, Structural Characterization of Polyurethane Foam, p.76.

[66] Kodippili, Structural Characterization of Polyurethane Foam, p.76.

[67] Kodippili, Structural Characterization of Polyurethane Foam, p. 79.


[70] Hatchett, D.W., et al., FTIR Analysis of Thermally Processed PU Foam, p. 3.


[73] Hatchett, D.W., O'Toole, B.J., 2003, “High Pressure Science and Engineering Center Polymer Foams Project Annual Report,” University of Nevada, Las Vegas, NV, p. 12. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.
CHAPTER 3

STUDY METHODS

This study directly assessed the relationship between physical properties, chemical properties, static mechanical properties, and dynamic mechanical properties of a proposed replacement for stockpile foam, ReCrete. The study was based on information collected through three different means: research, experimentation, and modeling.

3.1 Research

The study team had already accumulated a substantial library of research on the subjects of polyurethane foam, foam applications, mechanical properties, physical properties, and dynamic impact response. This study effort began with a literature search to ensure the historical information available to the study team was suitably current and relevant. The study team reviewed all of the literature in order to collect all available data and to ensure the study complemented similar research rather than duplicating it. New literature was revisited throughout the course of the study to keep the study as current as possible. Research results are summarized in the previous chapter.

3.2 Experimentation

Four different types of testing were identified as relevant to stockpile environments: high rate compression and flexural impact tests for the determination of energy
absorption characteristics and yield or fracture strength; and quasi-static compression and flexure testing for the determination of modulus, collapse strength (compression), and fracture stress/strain (tension).

This dissertation measured and evaluated two parameters under impact loading conditions. The first, energy absorption, is calculated as the area traced out by the impact stress-strain curve and has the unit energy/volume. The second is the collapse, yield, or fracture strength and is equivalent to the yield point stress for conventional compression or tension testing. This study also assessed modulus, collapse strength, and fracture behavior through a series of quasi-static compression and flexure tests performed on a mechanical test frame.

Specimens were prepared from molded cylinders and blocks of foam and tested to establish an empirical model of ReCrete’s mechanical behavior and to search for relationships to effects measurable by quasi-static flexure and compression testing. The study included dynamic mechanical analysis tests to directly correlate the results with conventional quasi-static mechanical test results.

3.3 Modeling

The information gathered from research and experimentation allowed the study team to develop a relational model of the foam. The relational model built upon other, simpler models. Experimental data from impact, compression, and flexural testing on ReCrete foam were used to develop statistical models of foam mechanical properties. See Fig. 3-1 for an example.
Constitutive models were found in the literature searches that described foam behavior in terms of classic mechanical functions. See Fig. 3-2 for examples.

Figure 3-2  Constitutive Models: Basic (left) and Complex (right)
Microscopic images of foam cells were developed into a morphological model of a typical cell. See Fig. 3-3 for an example. Together, these models were used to develop a set of relational models.

![Microscopic Image Used to Develop a Morphological Model](image)

Figure 3-3  Microscopic Image Used to Develop a Morphological Model

Using the experimental data collected, the study team developed predictive models that establish relationships between physical, chemical, morphological, and mechanical properties. Taken together, these relational models allow the study team to predict properties and performance characteristics of ReCrete based on properties and characteristics that can be controlled during development or measured in a non-destructive manner. The predicted properties and characteristics may not be immediately measurable, such as performance of aged foam, or measurable only by destructive means, such as absorption energy under dynamic impact. Figure 3-4 illustrates a predictive model that the study team developed using non-linear least-squares regression that quantifies the relationship between ReCrete foam density and its compressive modulus as a quadratic equation.
**Figure 3-4  Non-Linear Least-Squares Regression Model**

Similarly, Fig. 3-5 illustrates a predictive model that the study team developed using multivariate non-linear least-squares regression. The model quantifies the relationship between a representative ReCreate foam mechanical property and the mold size and processing temperature that can be controlled during molded foam fabrication. In the case of this representative example, the relationship would be quantified as a third-order multi-variate polynomial equation. The x-axis represents temperature in degrees Celsius. The x-axis scale is limited to 0-100 degrees because the blowing agent in ReCreate is water. The y-axis represents the mold size in non-dimensional units. The y-axis scale cannot go below zero, but is otherwise unbounded.
Over the course of the study, the UNLV High Pressure Science and Engineering Center (HiPsec) has focused on installing capabilities for measuring thermal, mechanical, and chemical properties of the same sample of fresh and aged foams. This study furthered this capability and capitalized on it to produce scientifically sound results by using a more efficient and more robust scientific methodology to produce consistent data from material samples.

The results of this work will be submitted for publication in appropriate scholarly journals. This dissertation is written in accordance with UNLV format and ASME style guidelines.
3.4 Foam Formulation and Manufacture

ReCrete foam is formulated from five chemicals: Voranol 490, DC 193 Surfactant, Polycat 17 Amine Catalyst, distilled water, and Rubinate 1680. Typically, the mass of each component is selected to produce a desired foam density of 0.1 g/cc. The final foam density was determined by combining these materials with a specific mix ratio. Table 3-1 shows the mass and percentage of chemicals used to make a batch of foam.

3.4.1 Manual Formulation

When the study team made foam manually, the chemicals were mixed in small amounts and used immediately to form foam in the molds. The first four chemicals were weighed in a 1-liter plastic container using a digital scale. The chemicals were then mixed by hand with a spatula for 2 minutes before adding the Rubinate. The completed formulation was then mixed with an overhead mixer for 90 seconds. Although a batch weighed over 140 grams, only 100 grams was usable. The remainder was lost to the manual mixing process. A 100-gram batch made ~1000cc of free-rise foam.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Mass (grams)</th>
<th>Percentage by Mass (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voranol 490</td>
<td>56.16</td>
<td>38.95</td>
</tr>
<tr>
<td>DC 193 Surfactant</td>
<td>1.42</td>
<td>0.98</td>
</tr>
<tr>
<td>Polycat 17 Amine Catalyst</td>
<td>0.33</td>
<td>0.22</td>
</tr>
<tr>
<td>Distilled Water</td>
<td>0.65</td>
<td>0.45</td>
</tr>
<tr>
<td>Rubinate 1680</td>
<td>85.67</td>
<td>59.40</td>
</tr>
<tr>
<td>Total</td>
<td>144.23</td>
<td>100.00</td>
</tr>
</tbody>
</table>

Rubinate 1680 is a hazardous material. Therefore, all chemicals were used within the confines of a fume hood and were properly disposed. As shown in Fig. 3-6, gloves,
masks, and lab coats are worn throughout the mixing process and during cleaning. Caution is taken when handling chemicals as well as when cleaning chemical spills.

Figure 3-6 Mixing the Foam Formula Under the Fume Hood

3.4.2 Automated Formulation

The mechanical properties of polyurethane foam are primarily functions of the density of the foam [74]. The chemistry of the foam also has a smaller secondary effect on the mechanical properties [75]. The density of polyurethane foam is a function of the factors listed in Table 3-2. The factors in Table 3-2 are ordered by the degree of their impact on foam density. The factors at the top of the table have the greatest impact on density, while the factors at the bottom of the table have the least impact.
Table 3-2 Density Factors

<table>
<thead>
<tr>
<th>Foam Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure (foam packing factor)</td>
</tr>
<tr>
<td>Edge Effects (skin)</td>
</tr>
<tr>
<td>Rise Temperature (which also affects chemistry)</td>
</tr>
<tr>
<td>Cure Temperature</td>
</tr>
<tr>
<td>Rise Time</td>
</tr>
<tr>
<td>Vertical Position (within a mold)</td>
</tr>
<tr>
<td>Radial Position (within a mold)</td>
</tr>
<tr>
<td>Humidity</td>
</tr>
</tbody>
</table>

Because the study team was attempting to measure and characterize the impact on density, and the resultant mechanical properties of factors such as the rise temperature and radial position, it became essential that all of the factors above them, which have a greater impact on density, be tightly controlled during the team's experiments. Rise time affects the density when the foam is mixed and begins to rise before being poured into a mold. Because manual formulation of the foam can be inconsistent, the team found it necessary to develop an automated formulation capability. The team acquired a Decker Reaction Injection Machine (RIM), shown in Fig. 3-7, and developed new procedures for formulating and producing ReCrete polyurethane foam. The Decker RIM is designed for 2-component formulas, so the team developed and tested an “A” component, known as the “POLY” component, from an appropriate mixture of Voranol, surfactant, catalyst, and water.
Because the POLY storage tank holds sufficient POLY component for the equivalent of thirty manually mixed batches, the team was able to mix a large volume of the first four components in a more precise ratio. The large volume of POLY component also ensured consistent formula between batches. Because of its instability, Rubinate was used by itself as the “B” component, or “ISO” component. The Decker RIM has an inert gas system that stores the POLY and ISO components in a stable, reduced oxygen environment for extended time periods. The mix head of the Decker RIM has a precise metering system that allowed the study team to make precise mixtures of the POLY and ISO components for a great degree of control over the ReCrete formula. The high speed mix head meters the appropriate amount of polyurethane for a specific mold, so that only the correct amount of formula is mixed as it is injected into the mold. This allowed the study team to closely control the rise time and temperature of each molded specimen. The greater storage capacity and high mix speed of the Decker RIM allowed the study
team to produce larger numbers of foam specimens in less time, adding to the scientific rigor of their experiments. The faster injection process also simplifies the procedures for foam packing to achieve greater foam densities, if needed.

The study team did discover that the short shelf life of Rubinate 1680 (3 months) adversely impacted foam manufacturing capability. Rubinate quickly crystallizes and solidifies when exposed to air. If the Rubinate was not used within 4-6 weeks, this effect routinely blocked the lines, valves, and injectors of the reaction injection machine, resulting in a significant amount of effort required to maintain the machine in working order. Because of the level of maintenance overhead involved, the study team does not recommend the use of Rubinate for commercial foam production applications. The team encountered no problems with any of the chemicals in the POLY A component.

3.4.3 Molded Foam Manufacture

Once the ReCrete was formulated, either manually or automatically, the chemical mix was poured into the appropriate molds. The study team developed many different molds for different tests and experiments. Figures 3-8 and 3-9 show some of the molds used for manufacturing foam samples.

Figure 3-8 Box Molds

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The molds shown are all open on the top so the foam can freely rise. Sufficient foam mix was poured into each mold to produce a small mushroom cap on the top of each cylinder. Mold release (PTM&W Industries Inc. #PA0801) was applied to the inside of each mold to aid foam removal once it was cured. O-rings and Teflon tape were used to prevent leakage between the cylinders and caps for molds processed in a water bath.

A packing factor was used to increase the density of the foam beyond the expected density prescribed by the mix ratio of all the ingredients. During free rise, the foam is only constricted on its sides and bottom while a foam specimen with a packing factor is restricted to a finite volume. This project studied foams with packing factors between 1 and 2, with 1 equating to free rise. Increasing the density by more than a factor of 2 should only be done by changing the mix ratio of the formula and not the packing factor. One type of mold, the small aluminum cylinder, is threaded for a screw-on cap on the top.
to form a fully-enclosed mold for the manufacture of packed foam. The maximum packing factor permitted is 2, so the study team could manufacture ReCrete foam up to a maximum density of 0.2g/cc. The remaining mold types were all open-topped, for the manufacture of free rise foam only.

Once the formula was poured into the mold(s), the foam was allowed to rise for 30 minutes under controlled temperature conditions. Temperature effects were examined by regulating the temperature of the molds at various temperatures during the first 30 minutes of the foaming process. A water bath or a convection oven was used to regulate mold temperature in most cases. Temperatures of foam batches made in the water bath were 25°C, 40°C, and 90°C. Buckets filled with ice were used to bring the temperature of the molds down to 0°C. Manually formulated batches and reference batches in large 1-liter and 4-liter molds were also made in room temperature air at approximately 25°C. When the study team manufactured foam using the RIM, they regulated mold temperature with a convection oven. The aluminum molds were pre-heated overnight to the desired rise temperature to ensure they were heat-soaked. Rise temperatures for foam made in the oven were 25°C, 45°C, 65°C, and 85°C. Figure 3-10 depicts each of the four temperature control mechanisms. The ice bath in Fig. 3-10(a) is used to control the temperature at 0°C. The water bath in Fig. 3-10(b) and the convection oven in Fig. 3-10(d) are used to control the temperature between 25°C and 90°C. The molds in Fig. 3-10(c) are at room temperature. After the 30 minute rise period, the foam was then placed in an oven at 66°C (150°F) for 4 hours. Once the foam finished curing, it was cooled and then extracted from the mold(s).
3.4.4 Aluminum Molds

Figure 3-9 depicts representative samples of foam manufactured by the study team. The reference samples, made in the 1-liter and 4-liter molds, were typically used for baseline data. This is because the skin formed at the edge of the sample, where the foam meets the mold surface, did not have a significant effect on the sample's properties closer to the core of the sample. All of the other molds are significantly smaller, so the foam sample's skin was a significant element of the sample. The two smaller samples shown in Fig. 3-11 are from the smallest molds used by the study team. The molds are 100-mm to 150-mm (4” to 6”) high, and 28.66-mm (1.13”) in diameter. This diameter produces
an area of 645-mm$^2$ (1 square inch) and a volume of 64,500-mm$^3$ to 96,750-mm$^3$
(4-6 cubic inches). The study team developed eleven of these small aluminum molds for
producing large quantities of small 16.39-cm$^3$ (1 cubic inch) foam samples.

Figure 3-12 shows the relative size of the small, medium, and large aluminum molds,
in comparison to a 4-liter mold. The medium molds are 150-mm (6") tall, and 40.53-mm
(1.60") in diameter. This diameter produces an area of 1290-mm$^2$ (2 square inches) and a
volume of 193,500-mm$^3$ (12 cubic inches). The study team developed six of these
medium aluminum molds for producing medium 32.78-cm$^3$ (2 cubic inch) foam samples.
The large molds are 150-mm (6") tall, and 49.64-mm (1.95") in diameter. This diameter
produces an area of 1935-mm$^2$ (3 square inches) and a volume of 290,250-mm$^3$ (18 cubic
inches). The study team developed six of these medium aluminum molds for producing
medium 49.17-cm$^3$ (3 cubic inch) foam samples.

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The aluminum molds are designed to produce cylindrical foam samples with cross-sectional areas of 645-mm$^2$, 1290-mm$^2$, and 1935-mm$^2$, respectively. Cylindrical foam samples are used for compression testing. Figure 3-13 depicts small, medium, and large foam specimens manufactured by the study team using the molds shown. The tops are removed and discarded during the process of preparing foam samples for the various types of testing. Using cylinders such as ones shown in Fig. 3-13, the study team could prepare four to six samples for testing.
Once the study team began to experiment with the flexural properties of ReCrete foam under bending loads, it was necessary to develop a new set of molds to produce new shapes, specifically beams. The aluminum box molds are designed to produce foam beam samples with cross-sectional areas of 323-mm$^2$, 645-mm$^2$, 968-mm$^2$, and 1290-mm$^2$, respectively. The study team developed four molds of each size for producing moderate quantities of foam samples. Previously, Fig. 3-8 showed the relative size of the aluminum box molds. Table 3-3 lists the dimensions of the aluminum box molds.

![Table 3-3 Aluminum Box Mold Dimensions (Inner)](image)

<table>
<thead>
<tr>
<th>Dimension</th>
<th>Mold 1</th>
<th>Mold 2</th>
<th>Mold 3</th>
<th>Mold 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width</td>
<td>177.8-mm (7 inches)</td>
<td>177.8-mm (7 inches)</td>
<td>177.8-mm (7 inches)</td>
<td>177.8-mm (7 inches)</td>
</tr>
<tr>
<td>Depth</td>
<td>12.7-mm (0.5 inches)</td>
<td>25.4-mm (1.0 inch)</td>
<td>38.1-mm (1.5 inches)</td>
<td>50.8-mm (2.0 inches)</td>
</tr>
<tr>
<td>Height</td>
<td>177.8-mm (7 inches)</td>
<td>177.8-mm (7 inches)</td>
<td>177.8-mm (7 inches)</td>
<td>177.8-mm (7 inches)</td>
</tr>
<tr>
<td>Area (cross-section)</td>
<td>2258-mm$^2$ (3.5 sq. in.)</td>
<td>4516-mm$^2$ (7.0 sq. in.)</td>
<td>6774-mm$^2$ (10.5 sq. in.)</td>
<td>9032-mm$^2$ (14.0 sq. in.)</td>
</tr>
<tr>
<td>Volume</td>
<td>401.5-cm$^2$ (24.5 cu. in.)</td>
<td>803.0-cm$^2$ (49.0 cu. in.)</td>
<td>1204.5-cm$^2$ (73.5 cu. in.)</td>
<td>1606.0-cm$^2$ (98.0 cu. in.)</td>
</tr>
</tbody>
</table>

Figure 3-14 depicts the four sizes of foam specimens manufactured by the study team using the molds shown in Fig. 3-8 above. The tops were removed and discarded during the process of preparing foam samples for the various types of testing. Using box molds such as those shown in Fig. 3-8, the study team could prepare four to six foam beam samples for testing.
3.5 Measurements and Tests

After the study team manufactured foam specimens, they cut each specimen into samples and prepared the samples for testing. Preparation processes were dependent on the type of testing to be done and are described below. Prior to any chemical or destructive mechanical testing, the study team measured the density of every sample.

3.5.1 Preparation of Foam Samples - Cylinder

After the foam was extracted from the cylindrical molds, it was cut into four to five levels using a band saw, as shown in Fig. 3-15. The cylindrical samples were then sanded on their tops and bottoms to achieve a 25.4-mm (1") height. This process assured sample consistency. To ensure accurate density calculations, extra foam dust was removed from each specimen.
As shown in Fig. 3-16, four to five 25.4-mm (1") tall samples were made from each cylindrical mold, for up to forty samples from a typical batch of foam, which filled eight molds. Depending on the objectives of this study, the samples were used for either static or dynamic compression mechanical testing.

3.5.2 Preparation of Foam Samples – Beam

After the foam was extracted from the box molds, it was cut into five to six levels using a band saw, similar to the cylindrical samples. The team used a cutting jig on the
band saw to achieve a consistent 25.4-mm (1") beam width. This process assured sample consistency without the need for sanding the samples. To ensure accurate density calculations, extra foam dust was removed from each sample after it was cut from the molded specimen.

3.5.3 Preparation of Foam Samples – Morphology

The study team prepared a limited number of morphology samples for Scanning Electron Microscopy (SEM). The study team followed the SEM foam sample preparation procedures described by Nelson in 2004 [76]. Figure 3-17 shows a foam sample on the right prepared with gold sputtering for SEM, and a foam specimen on the left embedded in epoxy. The sample was cut from a vertical slice of the specimen. The epoxy base was used to firmly hold the specimen while the sample was cut from it.

![Figure 3-17 Foam Sample for Scanning Electron Microscopy](image)

3.5.4 Mechanical Testing

The study team used four types of mechanical tests for ReCrete foam. The team conducted quasi-static compression tests, quasi-static flexure tests, dynamic compression tests, and dynamic flexure tests.
3.5.5 Mechanical Testing – Quasi-Static Compression Tests

Quasi-static compression tests were performed according to American Society for Testing and Materials (ASTM) Standard D-1621 [77] on the right circular cylinders described above. An equal number of samples from each vertical position in the cylinder specimens were compression tested to minimize sample variance. All compression tests were conducted with axial compression tests in the rise direction of the foam.

Modulus, peak yield strength, and collapse strength were determined from quasi-static compression tests using a United Testing Model SSTM-1 frame with a 4.4-kN load cell. Strain was measured with a United Testing Model EXT-62-LOE laser extensometer, as shown in Fig. 3-18. Reflective markers were placed on the sample with an initial gage length of approximately 19-mm. The two reflective markers were placed approximately 3-mm from the top and bottom edges of the sample. Datum27 software was used to run the experiments and collect the raw data. Samples were compressed until a strain of greater than 10% was reached. At 10%-20%, the strain, modulus, yield stress, and collapse stress could be accurately measured without encountering densification.

Figure 3-18 United Testing Machine with a Laser Extensometer

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The UTM SSTM-1 frame periodically measures instantaneous force and instantaneous relative displacement over time when compression tests are run. The Datum 27 software application logs and displays the force and displacement measurements. The frame operator entered the sample height and diameter data into the software application, as depicted in Fig. 3-19.

![Figure 3-19 Compression Sample Dimensions](image)

At the conclusion of each test, the operator exported the data from the Datum 27 file to an Excel file and converted the force and displacement data into stress and strain data. The operator then plotted the stress-strain curve. The modulus was calculated from the slope of the elastic range of the stress-strain curve. The yield stress was calculated from the peak of the stress-strain curve. The collapse stress was calculated from the mean value of the steady-state range of the stress-strain curve. Stress and strain were calculated using the following equations:
\[ \sigma = \frac{P}{A} \quad (1) \]
\[ \varepsilon = \frac{\delta}{H} \quad (2) \]

Where:
- \( \sigma \) = stress
- \( \varepsilon \) = strain
- \( P \) = load
- \( A \) = area
- \( \delta \) = displacement
- \( H \) = height

Representative calculations are depicted on the stress-strain curve shown in Fig. 3-20.

![Stress-Strain Curve](image)

Figure 3-20 Representative Stress-Strain Curve from a Compression Test

It has been shown that foam modulus and strength have a predictable dependency on density [78,79]. The study team observed fairly large density variations in the molded samples. Therefore, the experimentally determined modulus and strength for a given
sample were normalized to the target foam density of 0.1 g/cc. The normalized modulus and strength were computed using the following equations:

\[
E_{\text{norm}} = E_{\text{exp}} \left( \frac{0.1}{\rho} \right)^{1.7}
\]

\[
\sigma_{\text{norm}} = \sigma_{\text{exp}} \left( \frac{0.1}{\rho} \right)^{2.1}
\]

Where:

- \( E_{\text{norm}} \) = normalized modulus
- \( \sigma_{\text{norm}} \) = normalized strength
- \( E_{\text{exp}} \) = measured modulus
- \( \sigma_{\text{exp}} \) = measured strength
- \( \rho \) = measured sample density

3.5.6 Mechanical Testing – Quasi-Static Flexure Tests

Flexure tests were performed according to ASTM Standard D-790 [80] on the beam samples described above. The foam beams had thicknesses of 12.7-mm, 25.4-mm, 38.1-mm, or 50.8-mm (0.5 in., 1.0 in., 1.5 in., or 2.0 in.), a suspended span of 152.4-mm (6 in.) and a width of 25.4-mm (1 in.). A 12.7-mm thick foam beam is shown in Fig. 3-21. The test specimens were prepared according to ASTM Standard C-393 [81]. For this experiment, the beams were positioned with the skinned sides on the top and bottom, making the beams similar to sandwich structures. An equal number of samples from each vertical position in the box specimen were flexure tested to minimize sample variance. No flexure tests of beams in other orientations were conducted to explore the effect of anisotropy.
Modulus and fracture strength were determined from quasi-static flexure tests using a United Testing Model SSTM-1 frame with a 4.4-kN load cell. Deflection was measured with a United Testing Linear Displacement Variable Transducer (LDVT) extensometer, as shown in Fig. 3-22.
Datum27 software was used to run the experiments and collect the raw data. Samples were flexed in a 3-point bend jig until failure. At failure, modulus and fracture strength could be accurately measured. The UTM SSTM-1 frame periodically measures instantaneous force and instantaneous relative displacement over time when flexure tests are run. The Datum 27 software application logs and displays the force and displacement measurements. The frame operator entered the sample span, width, and thickness data into the software application, as depicted in Fig. 3-23.

![Figure 3-23 Flexure Sample Dimensions](image)

The study team configured the Datum 27 software to record the failure load and automatically convert load and deflection data into stress and strain data. The team also set up the software to determine the flexural modulus from the stress-strain curve by linear regression.

The modulus was calculated from the slope of the elastic range of the stress-strain curve. The fracture strength was calculated from the peak of the stress-strain curve at failure. Flexure calculations were made with the following equations:
Elastic Modulus

\[ E = \frac{\sigma}{\varepsilon} \]  \hspace{1cm} (5)

Where:

- \( E \) = elastic modulus
- \( \sigma \) = stress
- \( \varepsilon \) = strain

Bending Deflection

\[ \delta = \frac{P \cdot L^3}{48 \cdot E \cdot I} \]  \hspace{1cm} (6)

Where:

- \( \delta \) = deflection
- \( P \) = load
- \( L \) = span
- \( E \) = elastic modulus
- \( I \) = moment of inertia

Flexural Stress

\[ \sigma = \frac{3 \cdot P \cdot L}{2 \cdot B \cdot H^2} \]  \hspace{1cm} (7)

Where:

- \( \sigma \) = flexural stress
- \( P \) = load
- \( L \) = span
- \( B \) = thickness
- \( H \) = height

Flexural Strain

\[ \varepsilon = \frac{6 \cdot H \cdot \delta}{L^2} \]  \hspace{1cm} (8)

Where:

- \( \varepsilon \) = flexural strain
- \( H \) = height
- \( \delta \) = deflection
- \( L \) = span
Representative calculations are depicted on the stress-strain curve shown in Fig. 3-24. Similar to the compression data values, the experimentally determined modulus and fracture strength for a given sample were normalized to the target foam density of 0.1 g/cc. The normalized flexural modulus and fracture strength were computed using the same normalizing equations shown in the previous section.

![Flexural Stress-Strain](image)

Figure 3-24 Representative Stress-Strain Curve from a Flexure Test

3.5.7 Mechanical Testing – Dynamic Compression Tests

Dynamic compression tests were performed according to ASTM Standard D-5024 [82] on the right circular cylinders described above. The foam cylinder samples were prepared according to ASTM Standard D-1621 [83]. An equal number of samples from each vertical position in the cylinder specimens were compression tested to minimize sample variance.

Modulus, peak yield strength, collapse strength, absorption energy, and densification were determined from dynamic compression tests using an Instron Dynatup 8250 impact...
tower with a 25-kN load cell. Strain was measured with a Microstrain Model S-DVRT-38 Differential Variable Reluctance Transducer (DVRT), as shown in Fig.3-25. Dyn930 software was used to run the experiments and collect the raw data. Samples were compressed until a strain of greater than 70% was reached. At 70%+, the strain, modulus, yield stress, collapse stress, absorption energy, and densification could be accurately measured.

![Figure 3-25 Instron Dynatup with a DVRT Extensometer](image)

The Instron Dynatup 8250 rapidly measures instantaneous force and instantaneous relative displacement 2048 times over a 100-microsecond period when impact tests are triggered. The Dyn930 software application logs and displays the force and displacement measurements, as shown in Fig. 3-26.
The test operator entered the sample height and diameter data into the software application, as depicted in Fig. 3-27.

At the conclusion of each test, the operator exported the data from the Dyn930 file to an Excel file and converted the force and displacement data into stress, strain, energy,
velocity, acceleration, and densification data. The operator then plotted the stress-strain, 
energy, velocity, acceleration, and densification curves. The modulus was calculated 
from the slope of the elastic range of the stress-strain curve. The yield stress was 
calculated from the peak of the stress-strain curve, using the same equations shown for 
static compression. The collapse stress was calculated from the mean value of the 
steady-state range of the stress-strain curve up to the point of densification. The 
absorption energy was calculated from the area under the stress-strain curve. The 
velocity, acceleration, and densification were derived from data using these equations:

**Velocity**

\[
\nu = \frac{d\delta}{dt}
\]  \( \text{(9)} \)

Where:

\[
\begin{align*}
\nu & = \text{velocity} \\
\delta & = \text{displacement} \\
t & = \text{time}
\end{align*}
\]

**Acceleration**

\[
\alpha = \frac{dv}{dt}
\]  \( \text{(10)} \)

Where:

\[
\begin{align*}
\alpha & = \text{acceleration} \\
\delta & = \text{displacement} \\
t & = \text{time}
\end{align*}
\]

**Acceleration**

\[
\alpha = \frac{F}{m}
\]  \( \text{(11)} \)

Where:

\[
\begin{align*}
\alpha & = \text{acceleration} \\
F & = \text{force} \\
m & = \text{mass}
\end{align*}
\]
Absorption Energy

\[ W = \int \sigma(\varepsilon) d\varepsilon \]  \hspace{1cm} (12)

Where:

- \( W \) = absorption energy
- \( \sigma \) = stress
- \( \varepsilon \) = strain

Representative calculations are depicted on the stress-strain curve shown in Fig. 3-28. Similar to the quasi-static compression data values, the experimentally determined modulus, yield strength, and collapse strength for a given sample were normalized to the target foam density of 0.1 g/cc. The normalized compressive modulus, yield strength, and collapse strength were computed using the same equations shown in the quasi-static compression section.

![Stress vs. Strain](image)

Figure 3-28 Representative Stress-Strain Curve from a Dynamic Compression Test

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3.5.8 Mechanical Testing – Dynamic Flexure Tests

Dynamic flexure tests were performed according to ASTM Standard F-736 [84] on the foam rectangular beams described above. The foam beam samples were prepared according to ASTM Standard D-790 [85]. An equal number of samples from each vertical position in the box specimens were flexure tested to minimize sample variance.

Modulus, fracture strength, and absorption energy were determined from dynamic flexure tests using an Instron Dynatup 8250 impact tower with a 25-kN load cell. Strain was measured with a Microstrain Model S-DVRT-38 DVRT, as shown in Fig.3-29. Dyn930 software was used to run the experiments and collect the raw data. Samples were flexed to bending failure. At failure, the strain, modulus, fracture stress, and absorption energy could be accurately measured.

![Figure 3-29 Instron Dynatup with a Flexure Jig](image.jpg)

Figure 3-29 Instron Dynatup with a Flexure Jig
The Instron Dynatup 8250 rapidly measures instantaneous force and instantaneous relative displacement 2048 times over a 100-microsecond period when impact tests are triggered. The Dyn930 software application logs and displays the force and displacement measurements, as shown in Fig. 3-30.

![Load & Displacement vs. Time](image)

Figure 3-30 Dynamic Flexure Load-Displacement Curve

The test operator entered the sample length, width, and thickness data into the software application, as depicted in Fig. 3-23. At the conclusion of each test, the operator exported the data from the Dyn930 file to an Excel file and converted the force and displacement data into stress, strain, energy, velocity, and acceleration data. The operator then plotted the stress-strain, energy, velocity, and acceleration curves. The modulus was calculated from the slope of the elastic range of the stress-strain curve. The fracture stress was calculated from the peak of the stress-strain curve. The absorption energy was calculated from the area under the stress-strain curve. The velocity and acceleration curves were derived from time and displacement data. All calculations were done with appropriate equations shown previously. Representative calculations are depicted on the
stress-strain curve shown in Fig. 3-31. Similar to the dynamic flexure data values, the experimentally determined modulus and fracture strength for a given sample were normalized to the target foam density of 0.1 g/cc. The normalized flexural modulus and fracture strength were computed using the same equations shown in the quasi-static compression section.

![Stress vs. Strain](image)

Figure 3-31 Representative Stress-Strain Curve from a Dynamic Flexure Test

3.5.9 Mechanical Testing – Test Cell Matrix

Table 3-4 depicts the test cell matrix for this study. The test conditions are the two primary variables of the series of experiments: mold size and processing temperature. There were four different box mold sizes for flexure experiments and three different cylinder mold sizes for compression experiments, for a total of seven different molds. There were four different processing temperatures. Experiments with foam samples manufactured in all combinations of mold size and processing temperatures were
repeated for dynamic testing. The number listed in each test cell in the matrix represents the number of samples manufactured under those conditions and tested. The four shaded quadrants represent the grouping of test cells for the four primary experiments. The upper left shaded quadrant represents the static compression experiment. There were a total of 632 samples tested in this experiment. The upper right shaded quadrant represents the static flexure experiment. There were a total of 382 samples tested in this experiment. The lower left shaded quadrant represents the dynamic compression experiment. There were a total of 410 samples tested in this experiment. The lower right shaded quadrant represents the dynamic flexure experiment. There were a total of 272 samples tested in this experiment. In all, the study team manufactured and tested 1696 foam samples under 56 different sets of conditions.

Table 3-4 Test Cell Matrix

<table>
<thead>
<tr>
<th>Test Conditions</th>
<th>28.7mm Cylinder</th>
<th>40.5mm Cylinder</th>
<th>49.6mm Cylinder</th>
<th>12.7mm Box</th>
<th>25.4mm Box</th>
<th>38.1mm Box</th>
<th>50.8mm Box</th>
</tr>
</thead>
<tbody>
<tr>
<td>25°C Static</td>
<td>132</td>
<td>30</td>
<td>48</td>
<td>20</td>
<td>28</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>45°C Static</td>
<td>64</td>
<td>30</td>
<td>30</td>
<td>24</td>
<td>24</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>65°C Static</td>
<td>100</td>
<td>30</td>
<td>30</td>
<td>23</td>
<td>24</td>
<td>24</td>
<td>23</td>
</tr>
<tr>
<td>85°C Static</td>
<td>78</td>
<td>30</td>
<td>30</td>
<td>24</td>
<td>24</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>25°C Dynamic</td>
<td>31</td>
<td>30</td>
<td>30</td>
<td>17</td>
<td>15</td>
<td>20</td>
<td>15</td>
</tr>
<tr>
<td>45°C Dynamic</td>
<td>40</td>
<td>34</td>
<td>32</td>
<td>16</td>
<td>17</td>
<td>13</td>
<td>21</td>
</tr>
<tr>
<td>65°C Dynamic</td>
<td>40</td>
<td>31</td>
<td>31</td>
<td>12</td>
<td>22</td>
<td>24</td>
<td>18</td>
</tr>
<tr>
<td>85°C Dynamic</td>
<td>40</td>
<td>35</td>
<td>36</td>
<td>16</td>
<td>15</td>
<td>17</td>
<td>14</td>
</tr>
</tbody>
</table>

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3.5.10 Mechanical Testing – Experimental Accuracy

The study team used the same experimental instrumentation for compressive and flexural testing. There study team found significant differences in the scale of data collected from these experiments. For example, the mean static compressive modulus, \( E = 77.25 \) MPa, while the mean dynamic flexural modulus, \( E = 0.065 \) MPa. There is a difference greater than three orders of magnitude between them. Similarly, the mean dynamic compressive absorption energy = 60.22 J, while the dynamic flexural absorption energy = 0.05 J. There is a difference greater than three orders of magnitude between them, as well. Experimental accuracy was limited by the following factors: the sampling rate of the instrumentation software and the signal conditioner; the minimum resolution of the impact load cell; the maximum travel of the DVRT; and signal oscillations from the impact load cell.

Figure 3-32 compares the mean modulus and modulus variance for the empirical data collected from each of the four experiments in this study.

![Modulus Comparison](image)

Figure 3-32 Comparison of Experimental Modulus Data
It is clear from the figure that the measurable dynamic flexural modulus is very small compared to the measurable quasi-static compressive modulus. The dynamic flexural modulus values are about 1% of the maximum load cell rating of the load cell used to measure the data. Because of this, the study team placed a low confidence value on this data. Also, the figure shows that the mean dynamic compressive modulus was less than the quasi-static compressive modulus. This does not correspond to the findings of other studies that report that the modulus increases as the strain rate increases. The study team determined that the sampling rate of the instrumentation was insufficient to measure the dynamic compressive modulus.

Figure 3-33 compares various mean strength and strength variance for the empirical data collected from the experiments in this study. It is clear from the figure that the measurable static strengths and dynamic fracture strength compare well in scale. Because of this, the study team placed a high confidence value on this data.

![Strength Comparison](image-url)

Figure 3-33 Comparison of Experimental Strength Data
Figure 3-34 compares the mean strength and strength variance for the empirical data collected from the two dynamic experiments in this study. It is clear from the figure that the measurable dynamic fracture strength is very small compared to the measurable dynamic yield strength and collapse strength.

![Dynamic Strength Comparison](image)

Figure 3-34 Comparison of Experimental Dynamic Strength Data

The dynamic flexural strength values are about 1% of the maximum load cell rating of the load cell used to measure the data. Because of this, the study team placed a low confidence value on this data. Also, the figure shows that the mean dynamic yield strength and collapse strength increased as the strain rate increased. Also, the mean dynamic yield strength and collapse strength values are an order of magnitude greater than the quasi-static yield strength and collapse strength values shown in Fig. 3-33. This corresponds to the findings of other studies that report that the strength increases as the strain rate increases.
Figure 3-35 compares the mean absorption energy and absorption energy variance for the empirical data collected from the experiments in this study. It is clear from the figure that the measurable dynamic flexure absorption energy is very small compared to the measurable static and dynamic compression absorption energy.

![Absorption Energy Comparison](image)

Figure 3-35 Comparison of Experimental Absorption Energy Data

The dynamic flexure absorption values are about 1% of the maximum load cell rating of the load cell used to measure the data. Because of this, the study team placed a low confidence value on this data. Also, the figure shows that the mean compression absorption energy (per unit volume) remained constant as the strain rate increased.

3.5.11 Mechanical Testing – Density Tests

The study team measured the mean density of every foam sample and the vertical density gradient of every foam specimen.
3.5.12 Mechanical Testing – Mean Density Measurements

The study team measured mean density of foam samples by traditional mass-volume methods. To calculate mean densities, all samples were weighed on a precision balance, as shown in Fig. 3-36, and the height and diameter of the cylinders were measured with digital calipers, as shown in Fig. 3-16. For the beams, the length, width, and thickness were measured. When measurements were taken, four readings from each dimension were averaged and then recorded.

Figure 3-36 Weighing Foam Samples

3.5.13 Mechanical Testing – Vertical Density Measurements

Samples for vertical density analysis of the cylinder specimens were identified according to the taxonomy shown in Fig. 3-37.
Samples for vertical density analysis of the box specimens were identified according to the taxonomy shown in Fig. 3-38. Specimens from circular and box mold types were cut into four to six horizontal sections using a band saw. Density was calculated for each sample as described in the section above.
Endnotes – Chapter 3


[76] Nelson, M.C., O’Toole, B.J., Jackovich, D.J., 2004, “Cell Morphology of Rigid Polyurethane Foam,” unpublished paper, University of Nevada, Las Vegas, NV, p. 8. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.


CHAPTER 4

ANALYSIS AND FINDINGS

4.1 Foam Density

This section summarizes the analysis of the density of the ReCrete foam specimens produced by the study team for mechanical testing. Although the foam specimens were produced in different molds and different quantities for different testing purposes, the study team produced all foam specimens using the same automated formulation process described in Chapter 2. This section also includes a comparison of the manual formulation process to the automated formulation process with regard to the density of the specimens produced.

4.2 Density of Compression Samples

Figure 4-1 summarizes the average density of compression foam samples. The reference density is plotted as a dashed line, with a density of 0.10 g/cc. The chart shows that the mean densities of most of the samples were above the reference density, consistent with molded foam specimens. The average density is indicated by the top of each bar. The first and second standard deviations are indicated by the rectangular boxes and vertical lines, respectively, centered on the top of each bar. Figure 4-1 clearly shows that the density variations between samples were nominal despite the known vertical density gradients found in the cylinder specimens. The variance of the first data set
(29-mm diameter, 25°C) is unusually large because of a much larger sample count. The study team produced more specimens under those conditions to support additional chemistry and morphology experiments. The chart shows that the density gradually decreases as the mold size increases and as the processing temperature increases.

![Density vs. Cylinder Mold Size & Processing Temperature](image)

**Figure 4-1 Density of Compression Foam Samples**

The last bar on the chart (labeled “ALL”) represents the mean and variance of all of the samples, made under all conditions. The large variance of that data set provides further evidence that there is a correlation between changing formulation conditions and changing mean densities.
Figure 4-2 summarizes the average densities of static compression foam samples made by Jackovich and Nelson in 2002 for earlier experiments. These ReCrete foam specimens were made by hand as described in Chapter Three. The Taxonomy used on the X-axis is S, M, and L; for small (29-mm diameter), medium (41-mm diameter), and large (50-mm diameter) cylinder samples, respectively. The density of the reference batch is plotted as a dashed line, with a density of 0.101 g/cc. The chart shows that the average densities of the samples under all conditions were above the reference density.

![Average Density vs Processing Temperature and Mold Size](image)

Figure 4-2  Density of Manually-Formulated Compression Foam Samples

The average density is indicated by the top of each bar. The first and second standard deviations are indicated by the rectangular boxes and vertical lines, respectively, centered on the top of each bar. Figure 4-3 clearly shows that the density variations between
samples were minimal despite the known vertical density gradients found in the cylinder specimens. Also, the chart clearly shows that the density decreases as the mold size increases and as the processing temperature increases. Radial density gradients are thoroughly analyzed and discussed by Jackovich [86].

4.3 Density of Flexure Samples

The foam specimens molded in boxes for flexure samples were substantially larger in volume than the specimens molded in cylinders for compression samples. The largest cylinder mold is 290 cm$^3$ in volume, while the smallest box mold is 402 cm$^3$ in volume.

Figure 4-3 summarizes the average densities of flexure foam samples. The reference density is plotted as a dashed line, with a density of 0.10 g/cc. The chart shows that the mean densities of most of the samples under all conditions were above the reference density, consistent with molded foam specimens. The average density is indicated by the top of each bar. The first and second standard deviations are indicated by the rectangular boxes and vertical lines, respectively, centered on the top of each bar. Figure 4-3 clearly shows that the density variations between samples were nominal despite the known vertical density gradients found in the molded box specimens. The chart shows that the density clearly decreases as the mold size increases. The decrease is most noticeable in the two smallest mold sizes. These molds have significantly higher surface-to-volume ratios. The increased surface area can exhibit mechanical drag on the foam as it rises, slowing the rise process and consequently increasing the resulting density.
4.4 Foam Density Findings

Over the course of five experiments, the study team found consistent trends in the mean density of molded foam samples. Molded foam specimens have a higher mean density than un-molded reference samples for a given formulation. Mallick suggests that the skin formed at the edge of the foam against the mold surface is a possible explanation for the increase in density [87]. The mean density of the foam samples decreases as the mold size increases, as a result of the drag of the mold surface on the rising foam. The mean density of the foam samples slightly decreases as the processing temperature increases. The variance of sample sets for common conditions is low, despite vertical density gradients, indicating the gradients have limited effect relative to the mold size and processing temperature. Comparison of densities between foam specimens manually
formulated and automatically formulated indicates that both processes achieve similar consistency of sample density.

The study team developed a statistical model of foam density that is useful for predictive simulations of foam production. A histogram constructed from all of the density data collected during this study is depicted in Fig. 4-4. The histogram includes 1614 data points from all molded specimens, all seven cylinder and box mold sizes, and all four processing temperatures. The histogram assists in visualizing the mean density of approximately 0.117 g/cc and the upward skew of the density data.

![Density Distribution](image)

**Figure 4-4 Density Distribution of Foam Samples**

Using a computer statistical application, the study team test fitted a number of continuous probability distribution functions to the histogram, using maximum likelihood estimators. From the functions fitted, the study team discovered that the log-logistic
distribution function \((0, 13.5, 0.117)\) produced the best fit. The log-logistic statistical model is overlaid on the histogram in Fig. 4-5.

4.5 Foam Mechanical Properties

The following four sections on mechanical properties present the results of the core tasks of this study. The study team conducted four significant experiments to characterize and quantify the mechanical response of ReCrete foam to quasi-static compression, quasi-static flexure, dynamic compression, and dynamic flexure. Within each of the four experiments, the study team varied the ReCrete samples by molding them in multiple mold sizes (three compression molds and four flexure molds) and processing them at four different temperatures. The study team produced and tested a statistically significant set of samples under each combination of experimental conditions (mold size and processing temperature).
4.6 Quasi-Static Compression

Quasi-static compression testing of polyurethane foams is very common and has been well-documented in the literature for a great number of materials. Prior to this specific effort, the study team had already conducted quasi-static compression testing on manually-formulated ReCrete specimens at UNLV and at Sandia Laboratories. The study team was able to compare the results of quasi-static compression testing for manual and automated foam formulations. The strain rate used for quasi-static compression was 0.0008/second.

The following three figures depict the range of shapes and values that the study team found in the quasi-static compression stress-strain curves during the experiment. Figure 4-6 shows the stress-strain curves for the high, median, and low density samples. The figure shows that at low density, the stress-strain response appears significantly different, with no clear transition from elastic to plastic collapse.

![Static Compression Stress-Strain Curves](image)

Figure 4-6 Static Compression Stress-Strain Curves for High, Median, and Low Density Samples

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Figure 4-7 shows the stress-strain curves for the samples with the high, median, and low elastic modulus values. The figure shows that there is minimal difference between the upper half of all the samples in the experiment between the high and median samples. The figure also shows that samples with correspondingly low densities can have significantly lower modulus values.

Figure 4-7  Static Compression Stress-Strain Curves for High, Median, and Low Modulus Values

Figure 4-8 shows the stress-strain curves for the samples with the high, median, and low peak yield strength values. The figure shows that there is minimal difference between the upper half of all the samples in the experiment between the high and median samples. The figure also shows that the peak yield point becomes more pronounced in relation to the collapse plateau as the peak yield strength increases.
4.6.1 Compressive Modulus

Figure 4-9 displays the mean elastic modulus for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. Figure 4-9 indicates that there was significant variance between data sets without a clear correlation to either mold size or processing temperature. Most of the data sets fall within 1 standard deviation of a median elastic modulus of about 74 MPa, as indicated by the horizontal dashed line. The mean elastic modulus for all samples is 77.3 MPa. The mean density of the 41 mm, 25°C sample set is below average, so the compressive modulus is also correspondingly low.
Figure 4-9 Compressive Modulus vs. Mold Size and Processing Temperature

4.6.2 Normalized Compressive Modulus

Figure 4-10 displays the mean normalized elastic modulus for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. With the exception of the small molds processed at 85°C, there is a moderate trend of the normalized modulus increasing as the mold size increases. Most of the data sets fall within 1 standard deviation of a median elastic modulus of about 68,000 kPa, as indicated by the horizontal dashed line. The normalized mean elastic modulus for all samples is 63,250 kPa.
Figure 4-10 displays the mean normalized elastic modulus for each set of foam samples manually formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. As with the foam samples formulated using the automated process, there is a moderate trend of the normalized modulus increasing as the mold size increases. All of the mean modulus values fall within a range of 50-90 MPa, similar to those in Fig. 4-10.
4.6.3 Peak Yield Stress

Figure 4-12 displays the mean peak yield stress for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ± 1 standard deviation and values inside the vertical lines are within ± 2 standard deviations. With the exception of the medium molds processed at 25°C, Fig. 4-9 shows there is a moderate trend of the peak yield stress decreasing as the mold size increases. There is significantly less variance indicated in the mean peak yield stress than there is in the mean elastic modulus.
Figure 4-12 Peak Yield Stress vs. Mold Size and Processing Temperature

4.6.4 Normalized Peak Yield Stress

Figure 4-13 displays the mean normalized peak yield stress for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ± 1 standard deviation and values inside the vertical lines are within ± 2 standard deviations. Figure 4-13 shows the normalized peak yield reaches a local maximum at medium mold sizes and middle processing temperatures. There continues to be significantly less variance indicated in the mean peak yield stress than there is in the mean elastic modulus.
Figure 4-13 Normalized Peak Yield Stress vs. Mold Size and Processing Temperature

Figure 4-14 displays the mean normalized peak yield stress for each set of foam samples manually formulated under all combinations of mold size and processing temperatures, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. As with the foam samples formulated using the automated process, the normalized peak yield reaches a local maximum at middle processing temperatures. All of the mean peak yield stress values fall within a range of 1.0-1.2 MPa, similar to those in Fig. 4-13.
4.6.5 Collapse Stress

Figure 4-15 displays the mean collapse stress for each set of foam samples formulated under all combinations of mold size and processing temperatures, as well as the variance of the data sets. Values inside the box are within ± 1 standard deviation and values inside the vertical lines are within ± 2 standard deviations. As with the peak yield stress, Fig. 4-15 shows there is a moderate trend of the collapse stress decreasing as the mold size increases. There is significantly less variance indicated in the mean collapse stress than there is in the mean elastic modulus.
Figure 4-15 Collapse Stress vs. Mold Size and Processing Temperature

4.6.6 Normalized Collapse Stress

Figure 4-16 displays the mean normalized collapse stress for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. As with the peak yield stress, Fig. 4-16 shows the normalized collapse stress reaches a local maximum at medium mold sizes and middle processing temperatures. There continues to be significantly less variance indicated in the mean collapse stress than there is in the mean elastic modulus.
Figure 4-16 Normalized Collapse Stress vs. Mold Size and Processing Temperature

Figure 4-17 displays the mean normalized collapse stress for each set of foam samples manually formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ± 1 standard deviation and values inside the vertical lines are within ± 2 standard deviations. Unlike the recent samples tested, the normalized collapse stress does not show a clear correlation to mold size or processing temperature. All but one of the mean collapse stress values fall within 1 standard deviation of 1.05 MPa, and all of the mean collapse stress values fall within 2 standard deviations of 1.05 MPa, suggesting the quasi-static collapse stress may be independent of mold size and processing temperature.

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4.7 Quasi-Static Compression Findings

Between the two quasi-static compression experiments, the study team found consistent trends in the mean mechanical responses of molded foam samples. The mean normalized modulus increases as mold size increases. The mean peak yield stress and collapse stress decrease as mold size increases. The normalized peak yield stress and collapse stress reach local maxima at middle mold sizes and processing temperatures. As with the density analysis, comparison of quasi-static compression responses between foam specimens manually formulated and automatically formulated indicates that both processes produce specimens with similar quasi-static mechanical properties.
4.8 Quasi-Static Flexure

Foam is widely and commonly used for shock absorption, vibration dampening, impact absorption, and other compression-related applications. Consequently, foam compression response is also widely studied. Because foam is well known for its limited tensile strength, its response to tension and bending is studied less frequently. Prior to this specific effort, the study team had conducted limited quasi-static uni-axial tensile testing on manually-formulated ReCrete specimens at Sandia Laboratories. The strain rate used for quasi-static flexure was 0.0008/second.

4.8.1 Flexural Modulus

The study team found no clear correlation between the mean flexural modulus of ReCrete foam beams flexed in a 3-point bend and mold size or processing temperature. See Appendix II for detailed results.

4.8.2 Normalized Flexural Modulus

Once the mean flexural modulus data were normalized to a density value of 0.1 g/cm$^3$, there was a clear relationship to mold size. Figure 4-18 displays the mean normalized flexural modulus for each set of foam samples formulated under all combinations of mold size and processing temperatures, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. The mean normalized flexural modulus clearly increases as the mold size increases.
4.8.3 Fracture Stress

Figure 4-19 displays the mean fracture stress for each set of foam samples formulated under all combinations of mold size and processing temperatures, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. The mean fracture stress clearly decreases as the mold size increases.
4.8.4 Normalized Fracture Stress

Once the mean fracture stress data were normalized to a density value of 0.1 g/cc, there was no longer a clear relationship to mold size. The study team found no clear correlation between the mean fracture stress of ReCrete foam beams flexed in a 3-point bend and mold size or processing temperature. See Appendix II for detailed results.

4.9 Quasi-Static Flexure Findings

Through the quasi-static flexure experiment, the study team found clear relationships to mold size in the mean mechanical responses of molded foam samples. The mean normalized flexural modulus increases as mold size increases. The mean fracture stress decreases as mold size increases.
4.10 Dynamic Compression

Like quasi-static compression testing, dynamic compression testing of polyurethane foams is common and has been well-documented in the literature for many polymer materials. Foam is widely and commonly used for shock absorption, vibration dampening, impact absorption, and other dynamic compression-related applications. Consequently, foam compression response is also widely studied.

Prior to this specific effort, the study team had conducted moderate dynamic impact testing on manually formulated ReCrete specimens at Sandia Laboratories. A key factor in this experiment was the corresponding increase in drop height and initial strain rate with the increase in mold size. The study team used a constant impact mass of 25.14 kg. For the 29-mm foam samples, the drop height was 100-mm, the impact velocity was 1.4 m/second, the impact energy was 24.66 Joules, and the initial strain rate was 55/second. The drop height was increased with the mold size in order to maintain constant impact energy (in Joules/unit volume). For the 41-mm foam samples, the drop height was 200-mm, the impact velocity was 2.0 m/second, the impact energy was 49.32 Joules, and the initial strain rate was 80/second. For the 50-mm foam samples, the drop height was 300-mm, the impact velocity was 2.4 m/second, the impact energy was 73.99 Joules, and the initial strain rate was 95/second. Because the samples were compressed completely to the point of densification, the final strain rate was zero as the impact mass came to rest.

The following three figures depict the range of shapes and values the study team found in the dynamic compression stress-strain curves during the experiment. Figure 4-20 shows the stress-strain curves for the high, median, and low density samples. The figure shows that the mechanical properties increase as the density increases. Unlike the
static compression experiment, the densities are more evenly distributed between the high and low density samples.

Figure 4-20 Dynamic Compression Stress-Strain Curves

Figure 4-21 shows the stress-strain curves for the samples with the high, median, and low elastic modulus values. The figure shows that there is minimal difference between the lower half of all the samples in the experiment between the low and median samples. The figure also shows that samples with correspondingly high densities can have significantly higher modulus values.
Figure 4-22 shows the stress-strain curves for the samples with the high, median, and low peak yield strength values. Unlike the static compression experiment, the peak yields are more evenly distributed between the high and low samples. The figure shows that the peak yield point becomes more pronounced in relation to the collapse plateau as the peak yield strength increases. The figure also shows that the oscillations in the collapse plateau become more pronounced as the peak yield and collapse strengths increase.
4.10.1 Compressive Modulus

The study team found no clear correlation between the mean compressive modulus of ReCrete foam beams dynamically flexed in a 3-point bend and mold size or processing temperature. See Appendix III for detailed results.

4.10.2 Normalized Compressive Modulus

After the mean flexural modulus data were normalized to a density value of 0.1 g/cc, the study team still found no correlation between the mean normalized compressive modulus and the mold size or processing temperature. See Appendix III for detailed results.
4.10.3 Peak Yield Stress

Figure 4-23 displays the mean peak yield stress for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. Figure 4-23 shows that peak yield stress increases as the strain rate and mold size increase.

![Yield Stress vs. Mold Size & Processing Temperature](image)

Figure 4-23 Peak Yield Stress vs. Mold Size and Processing Temperature

4.10.4 Normalized Peak Yield Stress

After the mean peak yield stress data were normalized to a density value of 0.1 g/cc, the study team found the same correlation between the mean normalized peak yield stress, the strain rate, and the mold size. See Appendix III for detailed results.
4.10.5 Collapse Stress

Figure 4-24 displays the mean collapse stress for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ± 1 standard deviation and values inside the vertical lines are within ± 2 standard deviations. Figure 4-24 shows that collapse stress increases as the strain rate and mold size increase.

![Figure 4-24 Collapse Stress vs. Mold Size & Processing Temperature](image)

Figure 4-24 Collapse Stress vs. Mold Size and Processing Temperature

4.10.6 Normalized Collapse Stress

After the mean collapse stress data were normalized to a density value of 0.1 g/cc, the study team found the same correlation between the mean normalized collapse stress, the strain rate, and the mold size. See Appendix III for detailed results.
4.10.7 Absorption Energy

Figure 4-25 displays the mean absorption energy for each set of foam samples formulated under all combinations of mold size and processing temperatures, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. Unlike the other dynamic compressive responses, Fig. 4-25 reflects the ReCrete foam response to only the increasing mold size rather than the increasing strain rate. The leftmost bar, labeled "BASELINE", represents 100% of the impact energy available for absorption. The rightmost bar, labeled "ALL", represents the mean and variance for the entire experiment, including all samples. The overall mean absorption energy is about 18 J/unit volume, or about 72% of the impact energy available. With the exception of the samples from the medium molds processed at 25°C, Fig. 4-25 shows that absorption energy decreases slightly with increasing mold size. Absorption energy was measured to the densification point for all samples. Densification is discussed in the next section.

![Energy per unit volume vs. Mold Size & Processing Temperature](image)

Figure 4-25 Absorption Energy vs. Mold Size and Processing Temperature
4.10.8 Densification

Figure 4-26 displays the mean densification for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ± 1 standard deviation and values inside the vertical lines are within ± 2 standard deviations. Like the absorption energy, Fig. 4-26 reflects the ReCrete foam response to increasing mold size. The leftmost bar, labeled “BASELINE”, represents 100% densification. The rightmost bar, labeled “ALL”, represents the mean and variance for the entire experiment, including all samples. The overall mean densification is about 70% of the theoretical limit. With the exception of the samples from the small molds processed at 25°C, Fig. 4-26 shows that densification decreases slightly with increasing mold size.

![Densification vs. Mold Size & Processing Temperature](image)

Figure 4-26 Densification vs. Mold Size and Processing Temperature
4.11 Dynamic Compression Findings

Through the dynamic compression experiment, the study team found clear relationships to strain rate and mold size in the mean mechanical responses of molded foam samples. The mean peak yield and collapse stresses and the mean normalized peak yield and collapse stresses increase exponentially (∼e^0.034) as strain rate increases. These relationships and a comparison to quasi-static mechanical properties are depicted in Fig. 4-27. Initial data indicates an exponential relationship between strain rate and strength. Further testing at strain rates between 0.1/second and 10/second are needed. The mean absorption energy and mean densification decrease as strain rate and mold size increase.

![Dynamic Compression vs. Strain Rate](image)

**Figure 4-27 Dynamic Compression vs. Strain Rate**

4.12 Dynamic Flexure

Compression testing of polyurethane foams is common and has been well-documented in the literature for many polymer materials. Foam is widely and commonly used for shock absorption, vibration dampening, impact absorption, and other dynamic
compression-related applications. Consequently, foam compression response is also widely studied. Because foam is well known for its limited tensile strength, its response to tension and bending is studied less frequently. Prior to this specific effort, the study team had not conducted any dynamic flexure testing on ReCrete specimens. A key factor in this experiment is the constant drop height and constant impact energy for all samples and all experiment conditions. For all samples in this experiment, the impact mass was 23.32 kg, the drop height was 50-mm, the impact velocity was 0.99 m/second, the impact energy was 11.44 Joules, and the initial strain rate was 40/second. Unlike the dynamic compression experiments, the dynamic flexure experiments experienced very little decrease in the strain rate, as the samples failed at low impact loads and the impact mass continued to pass through the failed beam. The mass did not come to rest on the sample.

4.12.1 Flexural Modulus

The study team found no clear correlation between the mean flexural modulus of ReCrete foam beams dynamically flexed in a 3-point bend and mold size or processing temperature. See Appendix IV for detailed results.

4.12.2 Normalized Flexural Modulus

After the mean flexural modulus data were normalized to a density value of 0.1 g/cm$^3$, the study team still found no correlation between the mean normalized flexural modulus and the mold size or processing temperature. See Appendix IV for detailed results.

4.12.3 Fracture Stress

Figure 4-28 displays the mean fracture stress for each set of foam samples formulated under all combinations of mold size and processing temperatures, as well as the variance
of the data sets. Values inside the box are within ± 1 standard deviation and values inside
the vertical lines are within ± 2 standard deviations. Similar to the findings from quasi-
static flexural testing, the mean fracture stress clearly decreases as the mold size
increases. Compared to the mechanical response to quasi-static flexural testing, the mean
fracture strength of ReCrete is doubled under dynamic strain rates for all sets of
formulation conditions in this experiment. Also, the variance remains low relative to the
mean values, making this a high confidence predictor. The decreasing fracture strength is
a result of the decreasing influence of the foam skin. For this experiment, the beams
were oriented for the 3-point bend test such that the skin acted as the top and bottom
layers of a sandwich-like structure. The data suggests that the bottom layer of skin was
able to provide a significant portion of tensile strength to the smallest beams. [88]

![Fracture Stress vs. Mold Size & Processing Temperature](image)

Figure 4-28 Fracture Stress vs. Mold Size and Processing Temperature
4.12.4 Normalized Fracture Stress

After the mean fracture stress data were normalized to a density value of 0.1 g/cm$^3$, the study team found the same correlation between the mean normalized fracture stress and the strain rate. Figure 4-29 displays the mean normalized fracture stress for each set of foam samples formulated under all combinations of mold size and processing temperature, as well as the variance of the data sets. Values inside the box are within ±1 standard deviation and values inside the vertical lines are within ±2 standard deviations. Unlike the findings from quasi-static flexural testing, that showed no correlation, the mean normalized fracture stress clearly decreases as the mold size increases.

![Normalized Fracture Stress vs. Mold Size & Processing Temperature](image)

Figure 4-29 Normalized Fracture Stress vs. Mold Size and Processing Temperature

Compared to the mechanical response to quasi-static flexural testing, the mean normalized fracture strength of ReCrete is nearly quadrupled under dynamic strain rates.
for all sets of formulation conditions in this experiment. In addition, the normalization process revealed a significant increase in fracture strength in the samples processed at 85°C. This correlates well with the predicted response of the chemistry changes and the morphology findings for compression samples processed at 85°C, as discussed in Chapter 2.

4.12.5 Fracture Energy

The study team found no clear correlation between the mean fracture energy of ReCrete foam beams dynamically flexed in a 3-point bend and mold size or processing temperature. See Appendix IV for detailed results.

4.13 Dynamic Flexure Findings

Through the dynamic flexure experiment, the study team found clear relationships to processing temperature and mold size in the mean fracture strength of molded foam samples. The mean fracture strength and the mean normalized fracture strength decrease as mold size increases. The mean normalized fracture strength increases as processing temperature increases. The mean fracture strength and the mean normalized fracture strength increase significantly under dynamic strain rates.

4.14 Numerical Models

Using the experimental data collected on the densities and mechanical properties of foam samples made in different size molds and at different rise temperatures, the study team developed different numerical models to identify the relationships between the independent and dependent variables. Like the example models in the previous chapter,
these models were developed using least squares regression. The study team began by modeling the relationships with linear least squares regression. For relationships that could not be accurately modeled with a linear model, the study team progressed to modeling the relationships with quadratic least squares regression, and finally, third-order least squares regression. The results are shown in the following figures.

Figure 4-30 shows the normalized yield stress model developed using quadratic least squares regression of quasi-static compression data. Figure 4-30 shows how the fitted curve follows the general trend of the data. The dome shape of the curve presents a visualization of the local maxima of the normalized peak yield strength at middle mold sizes and processing temperatures. This shape correlates well with the data displayed in Fig. 4-13.

Figure 4-30 Normalized Peak Yield Strength for Quasi-Static Compression
The mathematical model for Fig. 4-30 is as follows:

$$\sigma_{ys} = 240.2 + 19.77(T) - 0.16(T^2) + 387.9(A) - 97.88(A^2)$$

Where:
- \(\sigma_{ys}\) = Yield Stress (kPa)
- \(T\) = Temperature (degrees C)
- \(A\) = Area (non-dimensional)

Figure 4-31 shows the normalized collapse stress model developed using quadratic least squares regression of quasi-static compression data. Figure 4-31 shows how the fitted curve follows the general trend of the data. The dome shape of the curve presents a visualization of the local maxima of the normalized collapse strength at middle mold sizes and processing temperatures. This shape correlates well with the data displayed in Fig. 4-16.

![Figure 4-31 Normalized Collapse Strength for Quasi-Static Compression](image)

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The mathematical model for Fig. 4-31 is as follows:

\[ \sigma_c = 332.6 + 15.79(T) - 0.12(T^2) + 336(A) - 86.75(A^2) \]

Where:

\( \sigma_c \) = Collapse Stress (kPa)

\( T \) = Temperature (degrees C)

\( A \) = Area (non-dimensional)

Figure 4-32 shows the density model developed using quadratic least squares regression of foam specimen density data prepared for quasi-static flexure testing. Figure 4-32 shows how the fitted curve follows the general trend of the data. The nearly flat shape of the curve presents a visualization of the almost linear relationship of the foam sample density to mold size and processing temperature. This shape correlates well with the data displayed in Fig. 4-3. Also, the model does not diverge at the boundaries, making it useful for extrapolation beyond the experimental data set.
The mathematical model for Fig. 4-32 is as follows:

\[ \rho = 0.18 - 1.08 \times 10^{-3} (T) + 8.08 \times 10^{-4} (T^2) - 0.018 (t) + 1.9 \times 10^{-3} (t^2) \]

Where:

\[ \rho \quad \text{Density (g/cc)} \]

\[ T \quad \text{Temperature (degrees C)} \]

\[ t \quad \text{Thickness (non-dimensional)} \]

Figure 4-33 shows the normalized modulus model developed using linear least squares regression of quasi-static flexure data. The flat surface presents a visualization of the linear relationship of the foam sample flexural modulus to mold size and processing temperature. This surface correlates well with the data displayed in Fig. 4-18.

![Normalized Modulus for Quasi-Static Flexure](image-url)
The mathematical model for Fig. 4-33 is as follows:

\[ E = 28.04 - 0.045(T) + 6.94(t) \]

Where:

- \( E \) = Flexural Modulus (MPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)

Figure 4-34 shows the fracture strength model developed using linear least squares regression of quasi-static flexure data. The flat surface presents a visualization of the linear relationship of the foam sample fracture strength to mold size and processing temperature. This surface correlates moderately well with the data displayed in Fig. 4-19.

Figure 4-34 Fracture Strength for Quasi-Static Flexure
The mathematical model for Fig. 4-34 is as follows:

\[ \sigma_{fs} = 1545 + 2.88 (T) - 215 (t) \]

Where:

- \( \sigma_{fs} \) = Fracture Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)

Figure 4-35 shows the yield stress model developed using linear least squares regression of dynamic compression data. The flat surface presents a visualization of the strong linear relationship of the foam sample peak yield strength to strain rate. This shape correlates extremely well with the data displayed in Fig. 4-23.
The mathematical model for Fig. 4-35 is as follows:

$$\sigma_{ys} = 2.25 - 0.018(T) + 13.62(A)$$

Where:

- $\sigma_{ys}$ = Yield Stress (MPa)
- $T$ = Temperature (degrees C)
- $A$ = Area (non-dimensional)

Figure 4-36 shows the collapse stress model developed using linear least squares regression of dynamic compression data. The flat surface presents a visualization of the very strong linear relationship of the foam sample collapse strength to strain rate. This shape correlates well with the data displayed in Fig. 4-24.

![Figure 4-36 Collapse Strength for Dynamic Compression](image)
The mathematical model for Fig. 4-36 is as follows:

\[ \sigma_c = -1.78 + 0.039(T) + 11.68(A) \]

Where:

\(\sigma_c\) = Collapse Stress (MPa)

\(T\) = Temperature (degrees C)

\(A\) = Area (non-dimensional)

Figure 4-37 shows the density model developed using linear least squares regression of foam specimen density data prepared for dynamic flexure testing. The flat shape of the surface presents a visualization of the linear relationship of the foam sample density to mold size and processing temperature. This shape correlates well with the data displayed in Fig. 4-3.

![Figure 4-37 Density of Dynamic Flexure Samples](image-url)
The mathematical model for Fig. 4-37 is as follows:

\[ \rho = 0.167 - 3.8 \times 10^{-3} (T) - 8.88 \times 10^{-3} (t) \]

Where:

\( \rho \) = Density (g/cc)
\( T \) = Temperature (degrees C)
\( t \) = Thickness (non-dimensional)

Figure 4-38 shows the fracture stress model developed using quadratic least squares regression of dynamic flexure data. The nearly flat surface presents a visualization of the almost linear relationship of the foam sample fracture strength to mold size and processing temperature. This surface correlates well with the data displayed in Fig. 4-28. Also, the model does not diverge at the boundaries, making it useful for extrapolation beyond the experimental data set.

![Figure 4-38 Fracture Strength for Quasi-Static Flexure](image-url)
The mathematical model for Fig. 4-38 is as follows:

\[ \sigma_{fs} = 8000 - 55.63(T) + 0.457(T^2) - 2.75 \times 10^3(t) + 363.4(t^2) \]

Where:

\[ \sigma_{fs} = \text{Fracture Stress (kPa)} \]
\[ T = \text{Temperature (degrees C)} \]
\[ t = \text{Thickness (non-dimensional)} \]

Figure 4-39 shows the normalized fracture stress model developed using quadratic least squares regression of dynamic flexure data. The nearly flat surface presents a visualization of the almost linear relationship of the foam sample normalized fracture strength to mold size and processing temperature. This surface correlates moderately well with the data displayed in Fig. 4-29. Also, the surface plot assists in the visualization of the effect of increased processing temperature on the normalized fracture strength.

Figure 4-39 Normalized Fracture Strength for Dynamic Flexure
The mathematical model for Fig. 4-39 is as follows:

\[ \sigma_b = 3200 - 31.51(T) + 0.364(T^2) - 787.4(t) + 90.38(t^2) \]

Where:

- \( \sigma_b \) = Fracture Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)

A complete listing of data and equations for the development of the least squares regression models for foam sample densities and mechanical properties is contained in Appendices I through IV of this paper.

4.15 Chemistry and Morphology

The study team observed the actual physical failures of the cylinder foam samples under dynamic compression, and categorized the dynamic impact responses of the samples into three types of failures. A type I failure, as depicted in Fig. 4-40, is a mild failure where the foam is nearly or fully densified, but the sample remains intact with some bulging and localized splitting.

Figure 4-40 Type I Compression Failure
A type II failure, as depicted in Fig. 4-41, is a moderate failure where the foam is fully densified and portions of the sample separate. There is also significant bulging and splitting.

![Figure 4-41 Type II Compression Failure](image)

A type III failure, as depicted in Fig. 4-42, is a severe failure where the foam is fully densified, and the sample disintegrates, leaving only the core of the sample intact, with significant loss of stiffness.

![Figure 4-42 Type III Compression Failure](image)
Dynamic axial compression testing of foam specimens manufactured with 25°C to 65°C rise temperatures (all mold sizes, all strain rates) yielded 100% of the type III failures. Dynamic axial compression testing of foam specimens manufactured with an 85°C rise temperature (all mold sizes, all strain rates) yielded 100% of the type I failures. Further, the post-impact samples exhibited significant flexibility and loss of rigidity, behaving more like flexible, open-cell foam.

The SEM micrograph depicted in Fig. 4-43 is a ReCrete foam sample from a 29-mm mold processed at 25°C. The micrograph is a perpendicular view of a middle section of the cylindrical sample at 35x magnification. This micrograph is representative of a large sample of micrographs taken by the study team for an earlier morphology study.

![Figure 4-43 Micrograph of ReCrete processed at 25°C](image)

The SEM micrograph depicted in Fig. 4-44 is a ReCrete foam sample from a 29-mm mold processed at 85°C. The micrograph is a perpendicular view of a middle section of the cylindrical sample at 35x magnification. This micrograph is representative of a large sample of micrographs taken by the study team for an earlier morphology study.
section of the cylindrical sample at 18x magnification. This micrograph is representative of a small sample of four (4) micrographs taken by the study team for this specific morphology comparison between processing temperatures.

Both micrographs in Fig. 4-43 and Fig. 4-44 are oriented with the rise direction at the top. The figures are sized in order to match magnification scales for comparison purposes. A comparison of the micrographs shows that there are significant microscopic differences in the foam samples processed at different temperatures. ReCrete foam processed at 25°C produces anisotropic cells of consistent size, oriented in the rise direction. There are limited portals in the cell walls between cells, contributing to rigidity. ReCrete foam processed at 85°C produces more isotropic, more spherical cells with greater variance in size, and no particular orientation. The cell walls are significantly thicker, but there are more portals between cells, decreasing cell rigidity by allowing the blowing agent gas to flow freely between cells.
Endnotes – Chapter 4


CONCLUSIONS AND RECOMMENDATIONS

A study of the effects of mold sizes and processing temperatures on density and compressive and flexural mechanical properties was presented. It was shown that the mold size had a significant effect on the density, the quasi-static compressive modulus, the quasi-static flexural modulus, the quasi-static fracture stress, the dynamic compressive densification, the dynamic compressive absorption energy, and the dynamic fracture stress. Research also showed that the processing temperature had a moderate effect on the density, moderate effects on quasi-static and dynamic compressive mechanical properties, and a significant effect on the quasi-static and dynamic fracture strength. These effects are large enough to warrant careful consideration of mold geometry and processing temperature when forming foams in place.

A study of the effects of foam manufacturing processes on density and mechanical properties was presented. It was shown that the foam manufacturing process had a significant positive effect on the density, in terms of both accuracy and consistency. It was shown that the foam manufacturing process also had a significant positive effect on the efficiency of foam production. It was also shown that the foam manufacturing process did not have a significant effect on the mechanical properties. The density effects and production efficiency benefits may be large enough to warrant careful consideration of foam production processes for commercial and government applications.
The study team did determine that Rubinate 1680 MDI require a significant maintenance effort to sustain production. This overhead burden may be large enough to warrant careful consideration of foam component selection for small to mid-size production applications.

5.1 Effect of Mold Size and Processing Temperature on Density

Researchers discovered that, for both the compressive test specimens (cylinders) and the flexural test specimens (beams), the mean density of the samples decreased as the mold size increased, and slightly decreased as the processing temperature increased. Variance of the density among the compressive test specimens (cylinders) was greater than the variance of the density among the flexural test specimens (beams), primarily because the cylinders have significantly smaller volumes than the beams. Density did not vary significantly as a function of position within the foam specimen mold.

5.2 Effect of the Manufacturing Processes on Density

Using the manual formulation and manufacturing process for ReCrete foam, researchers produced 265 foam density test specimens. This sample of 265 specimens had a mean density of 0.120 g/cc, with a standard deviation of 0.0119 g/cc. Using the automated formulation and manufacturing process for ReCrete foam, researchers produced 466 foam density test specimens. This sample of 466 specimens had a mean density of 0.113 g/cc, with a standard deviation of 0.0109 g/cc. Researchers demonstrated that the automated process was more productive (number of specimens...
produced in an experiment period); produced foam specimens closer to the target density of 0.100 g/cc; and produced a foam sample with less variance between specimens.

5.3 Effect of Mold Size and Processing Temperature on Quasi-Static Compressive Properties

The study team found that the normalized quasi-static compressive modulus increased as the mold size increased, and the normalized modulus reached a local maximum between the 45°C and 65°C processing temperatures. The quasi-static yield strength and quasi-static collapse strength decreased as mold size increased. The normalized peak yield strength reached a local maximum at the 65°C processing temperature, and at medium mold sizes. The normalized collapse strength also reached a local maximum at the 65°C processing temperature, and at medium mold sizes. These findings are consistent with previous findings by the study team for manually formulated foam samples.

5.4 Effect of Mold Size and Processing Temperature on Dynamic Compressive Properties

Researchers found that the dynamic compressive responses of the foam samples were primarily influenced by the strain rate of the impact tests. Increasing the strain rate and the mold size of the dynamic compression tests resulted in increases in peak yield stress, normalized peak yield stress, collapse stress, and normalized collapse stress. The normalized peak yield strength increased as the processing temperature increased. The normalized collapse strength also increased as the processing temperature increased.
5.5 Effect of Mold Size and Processing Temperature on Quasi-Static Flexural Properties

The study team determined that the normalized quasi-static flexural modulus increased as the mold size increased, and the flexural modulus was independent of processing temperatures. The fracture strength decreased as mold size increased. The normalized fracture strength reached a local minimum near the 45°C processing temperature.

5.6 Effect of Mold Size and Processing Temperature on Dynamic Flexural Properties

Researchers determined that the dynamic flexural modulus was independent of processing temperatures and mold sizes. The dynamic fracture strength and the normalized dynamic fracture strength decreased as mold size increased. The normalized dynamic fracture strength increased as the processing temperature increased.

5.7 Mechanical Property Differences Between Manufacturing Processes

A comparison of the results of mechanical testing between the manual and automated manufactured samples shows that the relationships between mold size, processing temperature and mechanical properties that were determined in the first experiment continue to hold true with only minor refinements. The quasi-static compressive modulus increases as the mold size increases. The quasi-static compressive modulus reaches a local maximum at a processing temperature of 65°C, which differs from the
first experiment, where researchers concluded that the quasi-static compressive modulus decreased as the processing temperature increased.

5.8 Effects of Chemistry

The chemistry study team found that increases in the processing temperature changed the chemistry of the foam samples, making them less rigid and more flexible. These chemistry changes resulted in decreasing density as the processing temperature increased. The study team also found that the resulting increased flexibility of the dynamic compression samples caused them to resist disintegration after impact, at all measured strain rates. The study team found microscopic morphology changes that correspond to higher processing temperatures. The chemistry changes also produced increased normalized peak yield strength and normalized collapse strength under dynamic compression for foam samples processed at higher temperatures. The normalized dynamic fracture strength increased as the processing temperature increased and the chemistry changes made the samples more flexible.

5.9 Recommendations for Further Study

The study team found a number of areas for further study that would address questions posed by the results of this study. Tensile testing of foam skin and tensile testing of un-skinned foam would aid in explaining the increased fracture strength of small foam beams. Flexure testing of beams rotated 90° from their “sandwich” orientation would also aid in understanding the role of foam skin in flexural responses. The study team could explore multiple dynamic strain rates for flexure testing to
determine the effect of strain rate on flexural response. Researchers could conduct dynamic compression testing under a constant strain rate to more accurately determine the effects of mold size on foam compressive impact response. The study team could conduct further dynamic flexure testing with more precise load instrumentation to better measure and characterize foam absorption energy under flexural failure. The study team could also conduct further dynamic compression and flexure testing with more precise displacement instrumentation to better measure and characterize foam densification, impact velocity, and deceleration under flexural and compressive failure. Researchers could repeat compression and flexure tests on foam samples processed in 10°C increments between the 60°C-100°C range to better characterize the chemistry change and the resultant mechanical property changes.
GLOSSARY

**Absorption Energy** - Energy that is retained without reflection or transmission on passing through a medium. The transmitted energy is absorbed by conversion into energy of another form, usually thermal. Closed-cell, rigid foam absorbs energy through brittle crushing and collapse of cells.

**Alpha Expansion Coefficient** – Used for materials with a glass transition temperature to distinguish the expansion coefficients before and after the glass temperature, where alpha 1 is the thermal expansion coefficient at a temperature lower than T_g and alpha 2 is the thermal expansion coefficient at a temperature higher than T_g. In many calculations, the alpha is used to represent the thermal expansion coefficient.

**Cell Geometry** – Cells are composed of vertices, edges, and faces. Common geometric models include cubes, rhombic dodecahedrons, pentagonal dodecahedrons, and icosahedrons. ReCrete mean cell diameter is 390 um.

**Closed-Cell, Rigid Foam** – Material made up of a network of solid struts and plates which form the edges and faces of cells. Solid faces separate the cells of a closed-cell foam. Cells are formed by a blowing agent (carbon dioxide). Strut bending, face stretching, and compression of the blowing agent are mechanisms of foam strength. Plastic buckling and brittle fracture are mechanisms of compressive strength for rigid foams. Rigid foams have high toughness but low resilience. Rigid foams can exhibit either elastic-brittle behavior.
Collapse Strength – Compressive stress at plateau region of stress-strain curve, associated with the brittle crushing of cell struts in a rigid foam. Euler buckling stress.

Cream Time – Time from start of mixing to start of foam rising (5% of final height).

Densification – Region of rapidly increasing stress, to a slope of $E_s$, as the cell material itself begins to compress. When relative density is above 0.3, there is a transition from cellular material to porous material. When relative density is above 0.5, the material begins to behave as a solid.

Dynamic – Strain rates above 40/second. Initial strain rate for ReCrete testing was 70/second. Dynamic strain rates are achievable with a drop tower with 1 us resolution. Dynamic strain limited to 0.60.

Flexure – The ability of a beam to bend without fracture. Flexural strength is the stress at the outer edge of the beam at the moment of crack or break.

Foam – A lightweight material in cellular form. Foam is made by introducing bubbles (usually gas) during manufacture. Foam typically has 30% or less density of its base material.

Fracture Strength – For rigid foams, stress at which the cells rapidly fail beyond the linear-elastic regime. Point at which cell struts transition from bending to brittle fracture. Tensile failure mechanism is propagation of a single crack. Ultimate Tensile Strength.

Gel Time – Curing reaction time of the polymer.

Impact Failure – Point on stress-strain diagram where impact load drops to 60% of peak load.
**Model** – A representation that defines the key characteristics, attributes, and qualities of an object.

**Plastic Yield Stress** – For rigid foams, stress at which the cells begin to collapse plastically beyond the linear-elastic regime. Point at which cell struts transition from bending to buckling.

**Polyurethane** – Any of various polymers containing the urethane radical. Polyurethane is used to form a wide variety of synthetic plastics, adhesives, paints, and rubber.

**Quasi-Static** – Strain rates at or below 1/second. Initial strain rate for ReCrete testing was 1.7x10^-4/second. The range of strain rates tested was 1.7x10^-5/second to 1.7x10^-2/second. Quasi-static strain was limited to 0.20.

**ReCrete** – A proprietary polyurethane, closed-cell, rigid foam developed by Sandia Laboratory for the U.S. Department of Energy. ReCrete is a second-generation improvement of Crete.

**Relative Density (p*/ps)** – Density of the cellular material divided by the density of the solid from which the cells are made (polyurethane); also referred to as normalized density. Normalizing is necessary in order to compare foam samples of different densities.

**Rise Time** – Time from start of mixing to end of foam rising (95% of final height); also referred to as blowing reaction time.

**Rise Time = Gel Time** – A polymer foam formula is optimized when the rise time equals the gel time. The formula is manipulated by changes in blowing agent and catalyst.
Simulation – The operation of a dynamic model or surrogate process in order to obtain or determine the outcomes that could occur in a real world system.

Structural Anisotropy (R) – Dependency of physical and mechanical properties on orientation of the material, as a result of elongation of cells in the rise direction.

Toughness – Energy absorption per unit volume, calculated by measuring the area under the tensile stress-strain curve. A product of strength and ductility.

Uni-Axial Compression – A load that causes a body to shorten in the direction of the applied load.

Uni-Axial Tension – A load that produces elongation in the direction of the applied load.

Young’s Modulus (E) – The initial slope of the stress-strain curve in the elastic region. The modulus of rigid foam is dominated by elastic bending of cell struts.
APPENDIX I

QUASI-STATIC COMPRESSION DATA
Density Gradients of All Molds at Different Processing Temperatures
Density vs. Mold Size & Processing Temperature

Mold Size & Processing Temperature

Density (g/cc)
\[ \rho = 0.183 - 4.5 \times 10^{-4}(T) + 1.7 \times 10^{-6}(T^2) - 0.057 (A) + 0.013 (A^2) \]

Where:
\( \rho = \) Density (g/cc)
\( T = \) Temperature (degrees C)
\( A = \) Area (non-dimensional)
$E = 68.47 - 0.095(T) + 0.011(T^2) - 1.5 \times 10^{-4}(T^3) + 42.71(A) - 35.47(A^2) + 7.51(A^3)$

Where:
- $E$ = Compressive Modulus (MPa)
- $T$ = Temperature (degrees C)
- $A$ = Area (non-dimensional)
Normalized Modulus vs. Mold Size & Processing Temperature

Mold Size & Processing Temperature

Modulus (MPa)

- 29mm 25C
- 41mm 25C
- 50mm 25C
- 29mm 45C
- 41mm 45C
- 50mm 45C
- 29mm 65C
- 41mm 65C
- 50mm 65C
- 29mm 85C
- 41mm 85C
- 50mm 85C
Normalized Static Compressive Modulus – 2nd Order

\[ E = 22.6 + 1.27(T) - 0.012(T^2) + 12.57(A) - 1.97(A^2) \]

Where:
- \( E \) = Compressive Modulus (MPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Static Compressive Yield Stress – 2\textsuperscript{nd} Order

\[ \sigma_{ys} = 1992 + 15.78(T) - 0.16(T^2) - 926.6(A) + 200.4(A^2) \]

Where:
\( \sigma_{ys} \) = Yield Stress (kPa)

\( T \) = Temperature (degrees C)

\( A \) = Area (non-dimensional)
\[ \sigma_{ys} = 240.2 + 19.77(T) - 0.16(T^2) + 387.9(A) - 97.88(A^2) \]

Where:
- \( \sigma_{ys} \) = Yield Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Static Compressive Collapse Stress – 2\textsuperscript{nd} Order

\[ \sigma_c = 1990 + 11.48(T) - 0.123(T^2) - 921.5(A) + 200.8(A^2) \]

Where:
- \( \sigma_c \) = Collapse Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Normalized Static Compressive Collapse Stress – 2nd Order

\[ \sigma_c = 332.6 + 15.79(T) - 0.12(T^2) + 336(A) - 86.75(A^2) \]

Where:
- \( \sigma_c \) = Collapse Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
APPENDIX II

QUASI-STATIC FLEXURE DATA
Static Flexural Density – 2\textsuperscript{nd} Order

\[ \rho = 0.18 - 1.08 \times 10^{-3} (T) + 8.08 \times 10^{-6} (T^2) - 0.018 (t) + 1.9 \times 10^{-3} (t^2) \]

Where:
\( \rho \) = Density (g/cc)
\( T \) = Temperature (degrees C)
\( t \) = Thickness (non-dimensional)
Static Flexural Modulus – 2\textsuperscript{nd} Order

\[ E = 65.71 - 1.21(T) + 9.7 \times 10^{-3}(T^2) + 14.60(t) - 2.57(t^2) \]

Where:
\begin{align*}
E & = \text{Flexural Modulus (MPa)} \\
T & = \text{Temperature (degrees C)} \\
t & = \text{Thickness (non-dimensional)}
\end{align*}
Normalized Flexural Modulus vs. Mold Size & Processing Temperature

Modulus (MPa)

Mold Size & Processing Temperature

13mm 25C 25mm 25C 38mm 25C 51mm 25C 13mm 45C 25mm 45C 38mm 45C 51mm 45C 13mm 65C 25mm 65C 38mm 65C 51mm 65C 13mm 85C 25mm 85C 38mm 85C 51mm 85C
Normalized Static Flexural Modulus – 1st Order

\[ E = 28.04 - 0.045 (T) + 6.94 (t) \]

Where:
- \( E \) = Flexural Modulus (MPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)
Static Flexural Fracture Stress – 1st Order

\[ \sigma_{fs} = 1545 + 2.88 \, (T) - 215 \, (t) \]

Where:
- \( \sigma_{fs} \) = Fracture Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)
Normalized Fracture Stress vs. Mold Size & Processing Temperature

Fracture Stress (kPa)

Mold Size & Processing Temperature

- 13mm 25mm 38mm 51mm
- 45C 45C 45C 45C
- 65C 65C 65C 65C
- 85C 85C 85C 85C
Normalized Static Flexural Fracture Stress – 3rd Order

\[ \sigma_{fs} = 2190 - 98.9 \,(T) + 1.85 \,(T^2) - 0.01 \,(T^3) + 171.6 \,(t) - 65.13 \,(t^2) + 6.5 \,(t^3) \]

Where:
- \( \sigma_{fs} \) = Fracture Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)
APPENDIX III

DYNAMIC COMPRESSION DATA
Dynamic Compressive Density – 2nd Order

\[ \rho = 0.251 - 2.4 \times 10^{-3} (T) + 1.7 \times 10^{-5} (T^2) - 0.05 (A) + 0.011 (A^2) \]

Where:
- \( \rho \) = Density (g/cc)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Dynamic Compressive Modulus – 2nd Order

$$E = -13.86 + 0.372 (T) - 2.6 \times 10^{-3} (T^2) + 14.66 (A) - 1.9 (A^2)$$

Where:
- $E$ = Compressive Modulus (MPa)
- $T$ = Temperature (degrees C)
- $A$ = Area (non-dimensional)
Normalized Dynamic Compressive Modulus – 2nd Order

\[ E = -22.65 + 0.5(T) - 3.4 \times 10^{-3}(T^2) + 15.72(A) - 2.52(A^2) \]

Where:
- \( E \) = Compressive Modulus (MPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Yield Stress vs. Mold Size & Processing Temperature

Yield Stress (MPa)

Mold Size & Processing Temperature

29mm 25C
41mm 25C
50mm 25C
29mm 45C
41mm 45C
50mm 45C
29mm 65C
41mm 65C
50mm 65C
29mm 85C
41mm 85C
50mm 85C
ALL
Dynamic Compressive Yield Stress – 1st Order

\[ \sigma_{ys} = 2.25 - 0.018(T) + 13.62(A) \]

Where:
- \( \sigma_{ys} \) = Yield Stress (MPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
\[ \sigma_{ys} = -8.06 + 0.13(T) + 9.62(A) \]

Where:
- \( \sigma_{ys} \) = Yield Stress (MPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
\[ \sigma_c = -1.78 + 0.039(T) + 11.68(A) \]

Where:
- \( \sigma_c \) = Collapse Stress (MPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Normalized Dynamic Compressive Collapse Stress – 1st Order

\[ \sigma_c = -8.25 + 0.13 \times (T) + 8.12 \times (A) \]

Where:
- \( \sigma_c \) = Collapse Stress (MPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Residual Densification vs. Mold Size & Processing Temperature

Mold Size & Processing Temperature

Densification (%)
Dynamic Compressive Densification – 2nd Order

\[ D = 0.667 + 3.7 \times 10^{-3} (T) - 4.2 \times 10^{-5} (T^2) + 0.019 (A) - 8.8 \times 10^{-3} (A^2) \]

Where:
- \( D \) = Densification (%)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Dynamic Compressive Absorption Energy – 2nd Order

\[ E_A = 22.77 - 0.056(T) + 4.9 \times 10^{-4} (T^2) - 1.71 (A) + 0.31 (A^2) \]

Where:
- \( E_A \) = Absorption Energy (J/unit volume)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
APPENDIX IV

DYNAMIC FLEXURE DATA
Dynamic Flexural Density – 1st Order

\[ \rho = 0.167 - 3.8 \times 10^{-4} T - 8.88 \times 10^{-3} t \]

Where:
- \( \rho \) = Density (g/cc)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)
Modulus vs. Mold Size & Processing Temperature

Modulus (Kpa)

Mold Size & Processing Temperature

12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm
25C 25C 25C 25C 45C 45C 45C 65C 65C 65C 65C 65C 85C 85C 85C 85C
\[ E = 484.5 - 25.65(T) + 0.464(T^2) - 2.7 \times 10^{-3}(T^3) + 152.9(t) - 91.79(t^2) + 13.19(t^3) \]

Where:
- \( E \) = Flexural Modulus (kPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Normalized Modulus vs. Mold Size & Processing Temperature

Normalized Modulus (KPa)

Mold Size & Processing Temperature

12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm
25C 25C 25C 25C 45C 45C 45C 45C 65C 65C 65C 65C 85C 85C 85C 85C

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Normalized Dynamic Flexural Modulus – 3rd Order

\[ E = 177.0 - 14.53(T) + 0.275(T^2) - 1.6 \times 10^{-3}(T^3) + 192.2(t) - 90.89(t^2) + 11.97(t^3) \]

Where:
- \( E \) = Flexural Modulus (kPa)
- \( T \) = Temperature (degrees C)
- \( A \) = Area (non-dimensional)
Fracture Stress vs. Mold Size & Processing Temperature

Mold Size & Processing Temperature

Fracture Stress (kPa)

12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm
25C 25C 25C 25C 45C 45C 45C 45C 65C 65C 65C 65C

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Dynamic Flexure Fracture Stress – 2nd Order

\[ \sigma_{fs} = 8000 - 55.63(T) + 0.457(T^2) - 2.75 \times 10^3(t) + 363.4(t^2) \]

Where:
- \( \sigma_{fs} \) = Fracture Stress (kPa)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)
Normalized Fracture Stress vs. Mold Size & Processing Temperature

Fracture Stress (KPa)

Mold Size & Processing Temperature

12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm 12.7mm 25.4mm 37.1mm 50.8mm
Normalized Dynamic Flexure Fracture Stress – 2\textsuperscript{nd} Order

\[ \sigma_{fs} = 3200 - 31.51(T) + 0.364(T^2) - 787.4(t) + 90.38(t^2) \]

Where:
\[ \sigma_{fs} = \text{Fracture Stress (kPa)} \]
\[ T = \text{Temperature (degrees C)} \]
\[ t = \text{Thickness (non-dimensional)} \]
Dynamic Flexure Fracture Energy – 3rd Order

\[ E_f = 0.469 + 5.3 \times 10^{-3} (T) - 88 \times 10^{-6} (T^2) + 4.7 \times 10^{-7} (T^3) + 0.674 (t) - 0.298 (t^2) + 0.039 (t^3) \]

Where:
- \( E_f \) = Fracture Energy (J)
- \( T \) = Temperature (degrees C)
- \( t \) = Thickness (non-dimensional)
BIBLIOGRAPHY


Hatchett, D.W., Kodipilli, G.C., Kinyanjui, J.M., Benincasa, F., Sapochak, L. 2004. “FTIR Analysis of Thermally Processed PU Foam.” Unpublished Paper. University of Nevada. Las Vegas, NV. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.

Hatchett, D.W., O’Toole, B.J. 2003. “High Pressure Science and Engineering Center Polymer Foams Project Annual Report.” University of Nevada. Las Vegas, NV. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.


Nelson, M.C. 2002. “Annual Review Morphology Presentation”. University of Nevada. Las Vegas, NV. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.

Nelson, M.C., O’Toole, B.J., Jackovich, D.J. 2004. “Cell Morphology of Rigid Polyurethane Foam.” Unpublished Paper. University of Nevada. Las Vegas, NV. A copy of this article is in UNLV possession and may be consulted by contacting the project’s principal investigator at bj@me.unlv.edu.


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