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High strain rate characteristics of rubber modified syntactic foams

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HIGH STRAIN RATE CHARACTERISTICS OF RUBBER MODIFIED SYNTACTIC FOAMS

A Thesis

Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering

in

The Department of Mechanical Engineering

by

Paul Wehmer
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This thesis deals with the characterization of hybrid syntactic foams under high strain rates (HSR) ranging from 450/s to 1000/s. The foams studied are comprised of epoxy resin matrix filled with 63% volume fraction glass microspheres and 2% volume fraction of ground rubber fragments. The focus of this study is to compare the strength, ultimate strain, and modulus of these composite materials at high strain rates and quasi-static conditions and to find out the effects of HSR on the failure mode and fracture behavior of these materials.

Split Hopkinson Pressure Bar (SHPB) apparatus is used for the HSR testing. Foams of four different densities were fabricated by using four different microspheres in order to observe the density effect on the HSR properties. The microspheres have the same average outer diameter of 40 \( \mu m \), but different wall thickness leading to a difference in their density. Rubber particles with an average size of 40 \( \mu m \) were also added in these four samples to study the effect of the rubber on their properties. Fracture surfaces were observed under a scanning electron microscope to understand the fracture behavior of these materials and the influence of the rubber particles.

The peak stress was found to increase as the strain rate increased for all types of foam. For the hybrid foams fabricated with the lightest density (S22 and S32 types) microspheres with the 40 \( \mu m \) rubber particles the modulus values had an nearly constant value as the strain rate increased. For these foams, the fracture surfaces showed damage to the microspheres.
The heavier density foams that used the S38 and K46 microspheres with the 40 µm rubber particles had an increasing modulus as the strain rate increased. The fracture surfaces showed failure of the epoxy matrix as the principal failure mode.

A further two samples were fabricated that utilized rubber particles that were 75 µm in diameter. Compared to the smaller rubber particles in the same density of foam, the testing showed that the larger rubber particles resulted in a 20% increase in peak stress and the modulus increased.
1 INTRODUCTION

We use both synthetic and naturally occurring composite materials every day. Composite materials can be broadly defined as the resultant material from combining two or more materials, while maintaining an interface between the constituents. There are two categories of constituent materials: matrix and reinforcement. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements impart their special mechanical and physical properties to enhance the matrix properties. A synergism produces material properties unavailable from the individual constituent materials, while the wide variety of matrix and strengthening materials allows the designer of the product or structure to choose an optimum combination. Based on the type of reinforcement material used, most composite materials are classified into one of these three types: fibrous, particulate, and sandwich. For this study, a particulate type composite is used.

A wide variety of fillers has been used as reinforcements in particulate composites. The reinforcement filler could impart a specific property of the composite such as higher strength or lower weight. In this study, a composite is fabricated that uses hollow glass microspheres as the primary filler. Use of hollow particles to make a cellular structure greatly reduces the weight of the composite.

Cellular materials, due to their high strength to weight ratio and low cost, are commonly available. Cellular materials have a large percentage of open space comprised of small gas filled voids. Two general classes exist for cellular materials. They either have open or closed cells. The open cell type has openings that connect to each other and do not have a consistent size opening. The interconnected
openings allow for lower density but results in an absorbent material. The open structure also results in lower strength. The closed cell type typically has a consistent sized opening that is sealed to liquids. Closed cell offers more strength, better insulation, and a higher density compared to a similar open cell structure. An example of a closed cell composite is syntactic foam.

Syntactic foams are closed cell materials characterized by using a reinforcement of manufactured spheres. The spheres can be made from a variety of materials- metal, carbon, glass, or plastic. The spheres are held in a matrix, which can be made of epoxy resins, plastics, concrete, rubber, or urethanes. For this research, syntactic foam made of an epoxy matrix with silica glass spheres is used. This material offers a high strength to weight ratio. The glass spheres are inexpensive readily available material. These silica dioxide microspheres are available in different wall thicknesses depending on the application.

Typical syntactic foams are two component materials. In this research, a second filler addition of a low percentage of small, solid, rubber particles forms a hybrid syntactic foam. The concept behind addition of the rubber particles is an effort to decrease the brittle nature of the polymer epoxy while having little increase on the composite density. A tougher composite material would be advantageous in reducing notch sensitivity and improving damage resistance. The elastomer crumb rubber utilized in this research came in two different sizes. The sizes are 40 and 75 μm. The shape of the rubber particles is highly irregular and offers a significant amount of surface area for their volume. The grinding of waste tires forms these rubber particles. Developing uses of a recycled material is desirable due to the large amount of waste
generated annually and for cost reductions. Presently the greatest consumer of crumb rubber is the asphalt industry.

The hybrid composite being tested is composed of a polymer epoxy matrix that contains borosilicate glass microspheres and elastomer rubber particles. All three of these materials have widely differing properties. Ideally, this composite would be considered a three-phase material. During mixing of the constituent materials, capturing of small air bubbles occurs. These air bubbles have some effect on the material properties. This also results in this composite being a four-phase construction.

Material engineering has long recognized the differences between static and dynamic reactions from various materials. High strain rate testing is used to characterize the abilities of materials to handle short rise time impacts and loadings. Composite materials due to their ability to be readily modified have been studied extensively for their ability to handle high strain rates. This thesis paper will present the results of high strain rate testing of a hybrid composite syntactic foam.

1.1 Composite Materials

A composite material is a construction material that consists of two or more different materials; the goal is hereby to combine the various advantages of the single materials in the resulting material, and to eliminate their disadvantages. Current design work has resulted in specific parameters for materials of construction. Sometimes it is not possible for an existing material to have all the required properties specified which has led to development of new or improved materials. Use of synthetic composite materials to meet these needs has increased a large amount in
the past few decades. Composite materials play a big role with regard to reducing weight by improving the strength to weight ratio. An example is a fibrous composite material that has anisotropic strength parameters. By optimizing the directional orientation of the reinforcement, the part weight is usually minimized. Some other advantages composite materials may have are high bending stiffness, corrosion resistance, ability to be molded to near finished part dimensions, and good thermal insulation properties. Currently the primary areas of application of composite materials are aerospace, motor sports, automobile industry, military, and sports equipment. The primary reason for these applications is the requirement of high strength to weight ratio while having the manufacturer’s ability to absorb the higher costs of the composite development and construction.

1.2 Particulate Composites

In this class of composite materials, small particles are embedded in the matrix material. Particles used for reinforcing include ceramics and glasses, small mineral particles, clays, metal particles such as aluminum, and amorphous materials including polymers and carbon black. They are widely used in construction and commodity industries for their improved performance over the matrix material. An example of some of the possible improvements includes thermal and electrical conductivities, friction and wear resistance, machinability, surface hardness, and cost effectiveness.

Particulate composites are most typically isotropic in their macroscopic properties since the reinforcement materials do not have a directional orientation. On the microscopic scale, highly reinforced polymeric materials are nonhomogeneous
materials. When these materials are deformed, the different sizes and distribution of filler particles, the different crosslink density of polymeric chains, and the variation of the bond strength between the particles and the matrix can produce highly nonhomogeneous local stresses and deformation fields.[1]

1.3 Syntactic Foams

The term "syntactic foam" simply means the foam construction resulting from mixing spheres or other lightweight aggregate within a resin system.[2] These hollow spheres typically range from 10 to 200 microns in diameter and are available in several materials. Applications for syntactic foams include underwater buoyancy applications, structural core for aerospace components, tooling pattern-making applications, small arms grips, rifle stocks, acoustics, oil well operations, and deep-sea operations. Syntactic foam is also easily moldable and machinable, making it a viable choice for many applications.[3]

This type of system has been found to have a high compressive strength-to-weight ratio. Syntactic foams have also shown to be useful in a number of applications due to their energy absorption properties. A particularly useful property of them is their ability to continue to absorb energy over a large strain range. As strain increases the glass spheres collapse in a gradually increasing proportion. This continued collapse of spheres results in considerable energy absorption. In addition, the foams show a large amount of strain before total failure. While this is not an elastic strain, the foams provide a good source of sacrificial energy absorption in an application such as an automotive bumper system.
1.4 Structure of Hybrid Syntactic Foams

Neat epoxies are brittle with low resistance to crack formation. In the region near the crack tip where the material is highly stressed, microcracks or microvoids are developed. As the material is further stretched, the microvoids may coalesce forming a large void that promotes the rapid growth of the main crack. Tensile strength is a function of the worst flaw that is present in the material. The macroscopic crack will develop at this flaw until it separates the specimen. A tougher epoxy matrix would allow higher strain rates in a composite material. Higher fracture energy would lead to higher damage tolerances and structures that are more durable.

The function of incorporating rubber particles as a filler material to modify the matrix increases the fracture toughness and fracture strain of the epoxy matrix material. This matrix modification affects the crack propagation. The rubber particles blunt the energy at the crack formation tip.[4]
2 LITERATURE SURVEY

A number of studies have been done on syntactic foams made with hollow glass fillers.[5,6] Other studies of syntactic foams looked at using hollow polymer fillers.[7,8] Studies of epoxy matrix syntactic foams with glass microspheres filler for compressive [2,9-12] and other mechanical properties [13-16] have been published previously in the literature. Elastic behavior of syntactic foams showed that the Young's modulus of syntactic foams with very thin wall microspheres is lower than that of the matrix material, while thicker wall microspheres stiffen the matrix material.[14,17,18]

Studies of strain rate effects on plain epoxy resin matrix syntactic foams have shown that with increasing strain rate the syntactic foam increased in modulus.[19,20] Also testing has shown that that the failure strength for the syntactic epoxy foam linearly increased with the logarithm of strain rate.[21] These papers utilized the Split Hopkinson Pressure Bar (SHPB) device that will be used in this study.

Besides the reinforcing material, characteristic properties of the matrix material affect the fracture strain of syntactic foams as observed clearly in previous studies. The damage tolerance of syntactic foams can be increased further if the epoxy matrix material can be toughened to reduce the brittleness. For damage tolerant applications it is desired to reduce the modulus of syntactic foams in order to reduce premature crack formation in specimens, and increase the fracture strain without a considerable change in the strength. The approach of using small solid
rubber particles for matrix toughening is adopted in the present study. The elastomer rubber particles used in the present study have the name of crumb rubber.

A number of studies have been performed where liquid rubber is mixed with the epoxy to change the characteristics of the matrix. However, these approaches were based on using reactive liquid elastomers.[22-28] The reactive liquid rubber used in this method is dissolved in the epoxy first. After adding a curing agent, the resin begins to cure and the molecular weight rises. The rubber precipitates out and forms the second phase particles. The volume fraction and size of the rubber domains are controlled by the degree of compatibility of the two phases and the kinetics of gelation. Butadiene acrylonitrile based rubbers are the principal liquid elastomer used for toughening of epoxies. In reactive liquid rubber toughening, especially for concentrations of more than five volume percent, these studies indicated that decreasing the size of the rubber domains down to a few microns in the epoxy matrix increased the effectiveness of the toughening process. For instance, in the work of Hwang[28], two different reactive liquid rubbers used with a concentration of 15 phr (parts per 100 parts of resin) leading to a particle size range of 1 to 20\(\mu\)m, increased impact strength, plane strain fracture toughness and fracture energy.

The effects of the addition of crumb rubber to a neat epoxy resin were examined along with the interface bonding strength. Results of the fractographic examination indicated that the main toughening mechanism was crack deflection due to better adhesion at the interface, while the secondary mechanisms were shear deformations and some debonding. It also showed that incorporation of rubber increased the fracture strain of the epoxy polymer.[4,29] Crumb rubber particles have
shown to modify the matrix of syntactic foams to achieve better impact and fracture toughness properties under quasi-static testing. In testing of a hybrid syntactic foam with 2% crumb rubber results showed a 50% decrease in modulus while only having a 10% decrease in peak strength.[30] Testing of a series of hybrid syntactic glass microspheres based composites that used zero, 10%, and 20% of crumb rubber in addition to 1.6% nanoclay and 0.8% 1.6mm long fibers showed that increasing the rubber content reduced impact damage and prevented microcracks from forming.[31]
3 PROPOSED RESEARCH

The effect of crumb rubber on high strain rate compressive strength and modulus of a syntactic foam constructed with glass microspheres needs study. A high volume percentage of glass microspheres is employed to minimize the weight of the syntactic foam. For this study, 63% of the volume is glass microspheres. Addition of a volume fraction of 2% rubber particles as secondary filler creates the hybrid syntactic foam. The objective is to fabricate hybrid syntactic foams of higher toughness and damage tolerance under high strain rate compressive loading conditions compared to plain syntactic foams. Even though the volume fraction of the rubber particles in the composite is 2%, the volume ratio of rubber to polymer matrix is nearly 5.7%, which is sufficient for changing the properties of the matrix.

The high strain rate compressive properties of glass microsphere–rubber hybrid foams are studied. This is to gain understanding of the mechanical properties and fracture behavior of such materials during rapid rises in loading. Fabrication of the hybrid foams will use four different types of microspheres. The types of microspheres all have similar diameters but the wall thickness varies. For each sample type, strain rates ranged from approximately 450 to 1000/sec. Three duplicate tests are made for each material type and conducted at each strain rate level to account for variations in specimen density and microstructure. Comparison of earlier quasi-static and dynamic compression test studies with the properties of hybrid foams will give understanding of the roles of rubber particles and strain rate in syntactic foams.
3.1 High Strain Rate Testing System

For the high strain rate testing the Split Hopkinson Pressure Bar (SHPB) system will be used. This is a commonly used device, also known as the Kolsky apparatus, for this type of testing. This device is a popular tool for study of the strain rate sensitivity of materials. It is mostly useful for testing in the range of strain rates of $100<\dot{\epsilon}<3000$/s. A cylindrical sample specimen is inserted between two elastic bars, called the incident and transmitter bars. A third bar, called the striker bar, impacts the incident bar with a velocity that results in a compressive pulse wave. The incident wave travels along the bar and is partially transmitted through the sample as a compressive force. The remaining wave is reflected from the specimen-incident bar interface and is now tensile in nature. The transmitted wave travels down the transmitter bar. It is desired that the transmitter bar is long enough that wave reflection does not occur during the testing time of the sample. A reflection would interfere with the state of stress in the sample. All three stress waves (incident, reflected, and transmitted) are converted to signals from strain gauges. Records of these signals enable the determination of the elastic stress-strain response of that sample at high strain rates.

The equations and theory behind the SHPB are well established. With the use of this device the stress, strain and modulus values can all be determined with reasonable accuracy.
For a SHPB test to be considered valid, it is required that several conditions concerning the specimen be approximately fulfilled: [32]

(a) Equilibrium should prevail

(b) The states of stress and strain should be uniform

(c) The state of stress should be uni-axial

(d) The effects of friction at the bar/specimen interfaces should be negligible.

These conditions are interdependent and sometimes in conflict when it comes to sample dimensions. Thus, (a) requires low whereas (c) and (d) require high aspect ratios (length “l” to diameter “d”). For example, a low ratio would make the interface frictional terms significant. The frictional effects occur from the sample expanding in diameter as it is compressed. Longer samples would have a higher ratio and would reduce the relative amount of friction effects at the interfaces. However, this would result in wave propagation considerations due to the longer time
to establish equilibrium and axial inertia effects. As a practical guide, the following equation serves to guide the optimum sample dimensions.[33]

\[ L = \sqrt[3]{v_s d^2} \]  

(1)

Where in this equation \( v_s \) equals the Poisson Ratio. For epoxy and microsphere foams, the Poisson’s ratio has been shown to be 0.3 for high volume fraction loadings.[34] For equation (1) the friction at the interfaces is neglected.

Friction at the bar-sample interface has a detrimental effect on the accuracy of the testing. If the friction level is high then equilibrium will not be present. The sample would form a barrel shape and the stresses would not be uniform and uniaxial. An estimation of the effect of friction can be given by equation (2).[35]

\[ P = \left(1 + \mu \frac{D}{3L}\right)\sigma_\varepsilon \]  

(2)

Where:

\( P \) equals applied mean pressure

\( \mu \) equals coefficient of friction

\( D \) and \( L \) are the sample diameter and length

\( \sigma_\varepsilon \) is the material flow stress

The material flow stress is a strain rate dependant value. For metallic materials, this factor typically increases as the strain rate increases. A study showed that with the use of a lubricated surface and polishing the interface that errors would be about 1.3% - 4%.[36]

The interface between the samples is lubricated to minimize the friction effects. The friction between the surface of the sample and the bars has been shown
to affect the peak strain readings of the sample. Little effect on the stress readings was noted.[37] In this testing, a thin layer of molybdenum disulfide grease reduces the coefficient of friction for both static and kinetic friction. This lubricant has been shown to be effective in ambient temperature SHPB testing.[38]

Coefficients of friction for hardened steel and hybrid syntactic foam are not readily available. For a lubricated steel surface the typical range of \( \mu \) values range from 0.1 to 0.2 depending on the mating material.[39]

For calculating the stress and strain in the sample, Kolsky developed the following relations.[40] For calculating the specimen stress, the following equation applies.

\[
\sigma_s(t) = E \left( \frac{A_o}{A} \right) \varepsilon_T(t)
\]

(3)

Where:

\( E \) = output pressure bar’s elastic modulus
\( A_o \) = output bars cross sectional area
\( A \) = sample’s cross sectional area
\( \varepsilon_T(t) \) = transmitted strain history

For the samples strain, the following relationship is established.

\[
\varepsilon_s(t) = -2 \left( \frac{C_o}{L} \right) \int_0^t \varepsilon_R(t) dt
\]

(4)

Where:

\( C_o \) = wave velocity in the input pressure bar
\( L \) = initial length of the sample
\[ \varepsilon_r(t) = \text{Strain history in the reflected pressure bar} \]

The wave velocity \( C_0 \) may be calculated by use of this equation,

\[ C_0 = \sqrt{\frac{E}{\rho}} \]  \hspace{1cm} (5)

Where the parameters \( E \) and \( \rho \) are equal to the elastic modulus and density of the pressure bar respectively.

Determination of strain rate is by the following equation:

\[ \frac{d\varepsilon_s(t)}{dt} = -2C_0\varepsilon_r(t) \]  \hspace{1cm} (6)

The striker impacts the free end of the incident bar with a velocity \( v_1 \) and generates a compression pulse that travels through the incident bar towards the specimen. The incident pulse wavelength can be adjusted by using striker bars of different lengths. The impact of two collinear rods results in a square wave with a length twice that of the striker bar.[41] The perfect impact of similar bars results in a square shaped pulse of magnitude \( \sigma = \frac{v_1\rho C_0}{2} \) and of duration \( t = \frac{2l}{C_0} \). The parameters \( v_1, \rho, C_0, \) and \( l \) are the striker bar's velocity just before impact, density, wave velocity, and length, respectively. These equations suggest that for a given impact bar, the stress generated is proportional to its impact velocity and its duration is only related to physical properties of the impact bar. Assuming that there is perfect contact between the striker and the incident bar and that the pulse propagates without dispersion, the rectangular square wave pulse is maintained and a one-dimensional stress state is induced in the specimen. It is desired that the input pulse
length (=C₀t₀) is much longer than the specimen length, where t₀ is the pulse duration and C₀ is the elastic wave velocity of the input/output bars.

Upon arrival at the specimen interface, the wave partially reflects back towards the impact end. The remainder of the wave transmits through the specimen and into the second bar, causing deformation in the specimen. The reflected and transmitted waves measured at the strain gauges are proportional to the specimen’s strain rate and stress, respectively. Specimen strain can be determined by integrating the strain rate. By monitoring the strains in the two bars, specimen stress-strain properties are calculated.[42]

The high speed dynamic testing does require care to be taken in the evaluation and consistency of the results. Testing of low mechanical impedance materials can result in variations and accuracy issues due to the low amplitude of the transmitted strain and excessive deformation of the sample.[43]

To perform data collection strain gauges are bonded to the bars an equal distance from the sample. Location of the gauges is important. To avoid interference from reflections the sensor position is farther from the bar-specimen interface then the length of the striker bar. In this testing, they are 185mm from the respective interface surfaces while the striker bar is 152mm long. On the incident pressure bar this allows the complete incident pulse to be recorded as well as the reflected pulse.

The voltage readings transmitted from the strain gauges connect to an oscilloscope. The data recording system stores the information for later processing. The strain rate and the strain in the specimen can be determined from the reflected pulse only, and the stress can be determined from the transmitted pulse only. By
eliminating time “t” in Equations 3, 4, & 6 by synchronizing the acquired signals, it is possible to calculate the stress–strain curve realized by the specimen material.

![Graph](image)

**Figure 2  Typical Output Graph from SHPB**

The basis of the foregoing calculations has the assumptions that the specimen undergoes homogeneous deformation and that the incident and transmitter bars are made of the same material with a uniform sectional area. The strain relationship assumes that the sample is of a fixed length and cross section. Note that the strain rate is not perfectly constant during the test. In addition, that the computed stress and strain values are the engineering (or nominal) ones. This is because the evaluation uses the initial specimen length and cross-sectional area. An important assumption in these equations is that the total sample material volume stays constant.

A SHPB test setup used at Louisiana State University is capable of a range of strain rates. In this equipment, the system used to develop the input pulse is
composed of a pneumatic cylinder. Control of the cylinder is with a manual valve, which fires the striker bar. Variation of the firing pressure achieves different impact speeds to change the strain rate. The incident and transmitted pressure bars and a striker bar get support from linear slide bearings. A basis of the equations stated previously is the operation of the incident pressure bar in the elastic strain region. So long as the pressures in the bars remain under their elastic limits, specimen stress, strain, and strain rate may be calculated from the recorded strain histories. Therefore, consideration to what material properties used for the pressure bars is required for proper set up of the testing.

Length of the incident bar is twice the length of the transmitter bar in this type of testing. The length of the bars must satisfy the one-dimensional wave propagation theory. Selection of the lengths of the bars is such that the length of the bar is significantly longer then the pulse length. The length, L, and diameter, D, of the pressure bars are chosen to meet a number of criteria for test validity as well as the maximum strain rate and strain level desired in the sample. The L/D ratio for the pressure bars is 128:1 and 64:1 for the incident and transmitter bars respectively. To allow separation of the incident and reflected waves for data reduction, each bar should exceed a length-to-diameter (L/D) ratio of ~20.[44] The other consideration affecting the selection of the bar length is the amount of total strain desired to be imparted into the specimen; the absolute magnitude of this strain is related to the time period of the incident wave.

Impedance is a important property since it determines the ratio between the magnitudes of the reflected and transmitted waves at the interface. For testing of
low impedance specimens, the majority of the incident wave would be reflected at the interface. The calculation of the impedance of the pressure bars was performed using the following equation.\[42\]

\[ V_b = s \rho C_o \] (7)

Where the variable “s” equals the cross sectional area of the rod.

The pressure bars available at the Composites Laboratory at Louisiana State University have the following properties. Diameters of all the bars are 9.5mm.

The testing performed here utilized the 18% Ni-Mo-Co Maraging Steel material for the striker and pressure bars. This ultra high strength steel is able to operate at high impact velocities required in this testing without any deformation.

For an ideal SHPB bar experiment, the sample should be in dynamic stress equilibrium and deform at a nearly constant strain rate over most of the test duration.\[45,46\] When the incident pulse in the incident bar arrives at the front surface of the specimen, the interface between the incident bar and the specimen is compressed and a stress wave is generated and starts to propagate in the specimen. At this moment, the interface between the specimen and the transmitter bar is still unloaded. A finite amount of time is required to get equilibrium of stress in the specimen.\[47\] The stress wave in the specimen then propagates to the back end of the specimen. Some of this wave is reflected back into the specimen and a portion of it transmits to the transmitter bar. Because the mechanical impedance of a specimen is usually different from that of the bars, the stress in the specimen initially increases from zero and reaches a homogenous state after a certain number of loading waves.
Table 1 Properties of Pressure Bars

<table>
<thead>
<tr>
<th>Type</th>
<th>Yield Strength (MPa)</th>
<th>Wave Speed (m/s)</th>
<th>Length of Striker Bar (m)</th>
<th>Length of Incident Bar (m)</th>
<th>Length of Transmitter Bar (m)</th>
<th>Impedance V_b</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maraging AISI Grade 18Ni (350)</td>
<td>2363</td>
<td>4980</td>
<td>0.152</td>
<td>1.22</td>
<td>0.61</td>
<td>0.0227</td>
</tr>
<tr>
<td>Aluminum 6061</td>
<td>455</td>
<td>5060</td>
<td>0.152</td>
<td>1.22</td>
<td>0.61</td>
<td>0.0027</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>1100</td>
<td>4940</td>
<td>0.152</td>
<td>1.22</td>
<td>0.61</td>
<td>0.02321</td>
</tr>
</tbody>
</table>

Back and forth within the specimen. During this period of loading, the specimen is not at equilibrium. The stresses at the interface with incident bar, the front-end stress, are different from those at interface with transmitted bar, the back-end stress. It is important to know how many transits the wave makes in the specimen. With the information an assessment can be made of how accurate the measured data is and which part of the data is unreliable.

The time increment for a stress wave to travel through the sample is:

\[ \Delta t = \frac{L}{v} \]  

(8)

Where \( v \) is the velocity of the wave in this material.

To achieve equilibrium the wave will have to traverse the sample a set number of times, which will take a finite amount of time. With “\( \eta \)” being the number of cycles, the calculation of this amount of time “\( t_b \)“ by use of:
The ratio of impedance \( \beta \) between the steel bars \( (V_b) \) and sample \( (V_s) \) being tested is given by:

\[
\beta = \frac{V_s}{V_b}
\]  

(10)

The stress increment \( \Delta \sigma_k = \sigma_k - \sigma_{k-1} \) describes the incremental change of stress in the sample after the incident wave travels “k” times from one end of the sample to the other would be given by:[48]

\[
\Delta \sigma_k = - \left( \frac{1 - \beta}{1 + \beta} \right)^{k-1} \frac{\rho C v_0}{1 + \beta}
\]  

(11)

In addition, the sample stress \( \sigma_k \) could be found by summation while considering the impedances from:

\[
\sigma_k = \sum_{i=1}^{k} \Delta \sigma_i = -\frac{1}{2} \left( \rho_o C_o v_o \right) \left\{ 1 - \left( \frac{1 - \beta}{1 + \beta} \right)^k \right\}
\]  

(12)

With the square wave transversing the sample “k” number of times the stress equilibrium of the sample can be quantified by the parameter \( \alpha_k \) and is defined by:

\[
\alpha_k = \frac{\Delta \sigma_k}{\sigma_k} = \frac{2 \beta (1 - \beta)^{k-1}}{(1 + \beta^k - (1 - \beta)^k)}
\]  

(13)

In this testing, it is desirable to have as uniform a stress state as possible. As the number of cycles of the wave \( (k) \) increases, a lower value of \( \alpha_k \) occurs since the incremental change in stress in the sample would decrease. With use of the
impedance values and other material properties, the amount of time to nearly reach equilibrium can be quantified.

Estimation of the impedance of the samples utilizes a modulus value from previous quasi-static testing of this fabrication of syntactic foam.[30] Table 2 gives the approximate impedance values for the samples. The modulus values will likely change under high strain rates but the ratio of the sample impedance to the maraging steel rod impedance is helpful in validating the testing procedures used.

Using the testing parameters already determined a graphical example of the stress equilibrium for this sample testing is determined through use of equation (13) and is shown in Figure 3. This figure shows how the dynamic equilibrium improves as the number of transversals (k) of the strain wave increases. For clarity, only the two extremes of impedance ratios are graphed.

As can be seen from Figure 3 it takes between 11 and 13 cycles for stress equilibrium difference to become less then 5% when the input pulse is a perfect square wave. However, for SHPB testing in the real world the leading edge of the incident wave is not squared off but has a slope to it. The slope on the leading edge of the incident wave gives a rise time to the wave. Deliberate wave shaping and dispersion are the causes of this rise time.
Table 2 Impedance Values of Samples

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Modulus (E) (GPa)</th>
<th>Density (g/cm³)</th>
<th>Impedance (Vₛ)</th>
<th>Percentage Ratio (B)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S22R40</td>
<td>870</td>
<td>0.504</td>
<td>0.000729</td>
<td>3.2%</td>
</tr>
<tr>
<td>S32R40</td>
<td>1025</td>
<td>0.563</td>
<td>0.000935</td>
<td>4.1%</td>
</tr>
<tr>
<td>S38R40</td>
<td>1300</td>
<td>0.606</td>
<td>0.001175</td>
<td>5.2%</td>
</tr>
<tr>
<td>K46R40</td>
<td>1400</td>
<td>0.601</td>
<td>0.001205</td>
<td>5.3%</td>
</tr>
<tr>
<td>S32R75</td>
<td>850</td>
<td>0.516</td>
<td>0.000747</td>
<td>3.3%</td>
</tr>
<tr>
<td>S38R75</td>
<td>1225</td>
<td>0.605</td>
<td>0.001138</td>
<td>5.0%</td>
</tr>
</tbody>
</table>

Figure 3 Wave Cycles to Reach Equilibrium
Deliberate wave shaping of the input pulse by use of a pulse shaper is often added to the SHPB apparatus. The rise rate of the input pulse is lowered by placing a thin metal disk section on the impact surface of the incident bar. After impact by the striker bar, the metal disk pulse shaper deforms elastically and spreads the rise of the pulse in the incident bar over a time period. A mild steel pulse shaper that had a diameter of 6.73 mm and a length of 3.29 mm is used in this testing. In addition, selection of the pulse shaper can affect the harmonics of the pulse itself. The pulse graph shown in Figure 2 does not show any overshooting as it transitions from the sloped section to the generally constant amplitude incident pulse.

The second cause of a slope to the leading edge of the input pulse is dispersion. The impulse is comprised of a number of different frequencies. The higher frequencies travel at a slower speed. The dispersion of the wave acts to smear the waveforms. As the waveform travels down the bar, the dispersion also results in ringing effects that distorts the shape.

The rise time of the input pulse helps establish dynamic equilibrium by allowing the pressure wave to traverse the length of the sample. Examination of the incident pulse shows that regardless of the strain rate involved it takes on average 9 $\mu$s for the pulse to increase in strength to its nearly constant strain value. A calculation of the time for a traverse of the wave through the samples shows that it ranges from 0.5 $\mu$s for the densest foam to 0.7 $\mu$s for the lightest foam. Previously it was shown that to achieve less then 5% difference in the incremental stress increase would take 11 cycles of the input wave for the lightest density foam. For the lightest foam, the rise time of the pulse corresponds to $\sim$13 cycles. From this, it can be seen
that the rise time of the pulse is longer then the number of cycles required to reach equilibrium. The rise time of the incident pulse will be used in the modulus calculation to insure that the measurement is done while the sample is near equilibrium.

3.2 Experimental

The samples are cut to the following dimensions. The diameter was core drilled to 9.38mm and the length was 14.0 to 14.6mm. The diameter of the samples was slightly less than that of the bars (0.965 cm) and due to the small Poisson's ratio the specimen diameter during deformation never exceeds the bar diameter within the strain levels carried out. Samples are carefully centered on the bars by the use of an alignment fixture.

Strain gauges manufactured by “Vishay” with resistance a 350 Ohms and Gauge Factor of 2.10 with a gauge length of 6.35mm are used. They are bonded to the surface of the incident and transmitter pressure bars at the distance of 18.5 cm from the junction ends of both pressure bars. Signals from the strain gauges are fed to an amplification signal processor and then to the data display and logging system.

3.2.1 Matrix Resin

Based on previous studies of syntactic foams of similar construction, D.E.R. 332, a di-epoxy resin, manufactured by DOW Chemical Company is selected for the study. This resin is called diglycidyl ether of bisphenol A (DGEBA). The chemical name of this resin is 2,2- bis[4-((2'3' epoxy propoxy) phenyl) propane. Manufacturer’s properties are given in table 3.[49]
Table 3 Properties of Epoxy Resin

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
<th>Testing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxide Equivalent Weight (g/eq)</td>
<td>171-175</td>
<td>ASTM D-1652</td>
</tr>
<tr>
<td>Epoxide Percentage (%)</td>
<td>24.6-25.1</td>
<td>ASTM D-1652</td>
</tr>
<tr>
<td>Viscosity @ 25°C (mPa/s)</td>
<td>4000-6000</td>
<td>ASTM D-445</td>
</tr>
<tr>
<td>Density @ 25°C (g/ml)</td>
<td>1.16</td>
<td>ASTM D-4052</td>
</tr>
</tbody>
</table>

3.2.2 Hardener

For a curing agent for the epoxy resin the chemical commercially known as D.E.H. 24 and manufactured by DOW Chemical Company was utilized.[50]

Table 4 Properties of Epoxy Hardener

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amine Hydrogen Equivalent Weight (g/eq)</td>
<td>≈ 24</td>
</tr>
<tr>
<td>Viscosity @ 25°C (mPa/s)</td>
<td>19.5-22.5</td>
</tr>
<tr>
<td>Density @ 20°C (g/ml)</td>
<td>0.981</td>
</tr>
</tbody>
</table>

Molecular weight of this hardener is 146.4. For the selected combination of epoxy resin and hardener, the curing schedule is to gel at room temperature and then post cure at 100°C for three hrs.

3.2.3 Neat Epoxy Resin

For this epoxy and hardener combination, the following property data is provided.[51]
Table 5 Properties of Neat Epoxy

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average Epoxide Equivalent Weight (g(eq))</td>
<td>174</td>
</tr>
<tr>
<td>Parts per Hundred Parts Resin (weight)</td>
<td>14.0</td>
</tr>
<tr>
<td>Formulation Viscosity (cps at 25°C)</td>
<td>900</td>
</tr>
<tr>
<td>Flexural Strength (MPa)</td>
<td>107.5</td>
</tr>
<tr>
<td>Flexural Modulus (MPa)</td>
<td>2800</td>
</tr>
<tr>
<td>Yield Compressive Strength (MPa)</td>
<td>109</td>
</tr>
<tr>
<td>Compressive Modulus @ 10% Deformation or less (MPa)</td>
<td>1813</td>
</tr>
<tr>
<td>Tensile Strength (MPa)</td>
<td>66.3</td>
</tr>
</tbody>
</table>

3.2.4 Diluent

A diluent is added to lower the viscosity of the resin mix. Effective mixing is difficult due to the high volume fraction of microspheres. Addition of the diluent permits higher filler loading and gives better wetting and impregnation.

Adding 5% by volume of the diluent that is commercially known as ERISYS GE-8, brings down the viscosity of the resin from about 4000-6000 cps at 25°C to about 2000 cps at the same temperature. The diluent was supplied by CVC Specialty Chemicals. This material is C_{12-14} aliphatic glycidyl ether.

Table 6 Properties of Diluent [52]

<table>
<thead>
<tr>
<th>Typical Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxide Equivalent Weight</td>
<td>275-300</td>
</tr>
<tr>
<td>Viscosity @ 25°C</td>
<td>5-10</td>
</tr>
<tr>
<td>Density @ 25°C</td>
<td>0.88-0.90</td>
</tr>
</tbody>
</table>
Additional effects are shown in the manufacturer’s data that the diluent addition results in lowering tensile strength by about 10%. In addition, the modulus of the epoxy decreases about 15% with a corresponding increase in ductility. For a 95 wt% resin and 5 wt% diluent mixture, the EEW is calculated to be 17.75.

3.2.5 Microspheres

Four types of borosilicate glass microspheres are used for the fabrication of hybrid syntactic foam specimens. The commercial names of these four types are S-22, S-32, S-38 and K-46. The number they are represented by indicates the density of the microspheres in g/cc. The microspheres were manufactured and supplied by 3M under the trade name "Scotchlite". Microspheres wall thickness can be related to a parameter named Radius Ratio, \( \eta \), which is given by Equation 7.[53]

\[
\eta = \frac{R_i}{R_o} \tag{14}
\]

Where \( R_i \) is the internal radius and \( R_o \) is the outer radius of the microspheres. Increase in \( R_i \) corresponds to a decrease in wall thickness and a corresponding reduction in weight and strength. Four different radius ratio of borosilicate glass microspheres are used for the fabrication of the hybrid syntactic foam specimens. Distribution of outer diameter of all types of microspheres is nearly the same, but the internal diameter is different. Mean particle size, true particle density and radius ratio values of selected microspheres supplied by the manufacturer.
Table 7 Properties of Microspheres

<table>
<thead>
<tr>
<th>Type</th>
<th>10th Percentile</th>
<th>50th Percentile</th>
<th>90th Percentile</th>
<th>Average True Density (Kg/m³)</th>
<th>Average Wall Thickness (µm)</th>
<th>Pressure for Min. 80% Survival (Mpa)</th>
<th>Radius Ratio &quot;n&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>S22</td>
<td>20</td>
<td>35</td>
<td>60</td>
<td>220</td>
<td>1.26</td>
<td>2.76</td>
<td>0.9703</td>
</tr>
<tr>
<td>S32</td>
<td>20</td>
<td>40</td>
<td>75</td>
<td>320</td>
<td>1.86</td>
<td>13.79</td>
<td>0.9561</td>
</tr>
<tr>
<td>S38</td>
<td>15</td>
<td>40</td>
<td>75</td>
<td>380</td>
<td>2.23</td>
<td>27.58</td>
<td>0.9474</td>
</tr>
<tr>
<td>K46</td>
<td>15</td>
<td>40</td>
<td>70</td>
<td>460</td>
<td>2.74</td>
<td>41.37</td>
<td>0.9356</td>
</tr>
</tbody>
</table>

3.2.6 Rubber Particles

The rubber particles are commercially supplied by Rouse Polymerics under the trade name GF-80 and GF-170. The 80 and 170 notations refer to the grid size used in size separation. These have a mean particle size of 75 and 40 µm, respectively, and are referred to as R75 and R40 in this paper. The specific gravity of rubber particles is in the range of 1.12–1.15. The rubber particles are shown to have a highly irregular surface area. This is due to the shearing generated in micro milling used in the manufacturing process.

3.2.7 Mold

Stainless steel molds having inner dimensions of 228 x 228 x 13 mm are used for casting the syntactic foams. No degassing or positive pressure is applied during the casting or curing of syntactic foam slabs.

3.2.8 Mold Release Agent

Dow Corning 111 Sealant and Lubricant is used as a release agent in the molds. This lubricant is a silicone-based compound, heavy consistency white translucent grease. Selection of this release agent is based on its service
temperature range of –40 to 204°C and bleed characteristics, 0.5% in 24 hrs at 200°C. Specific gravity of this release agent is 1.0.

3.3 Hybrid Syntactic Foam Fabrication

Volume fraction of microspheres is maintained at 63% in all types of syntactic foams. A high volume of microspheres is desired to minimize the density of the foams. The random close packing factor of spheres of equal size is given approximately as 64%. The volume fraction of the rubber particles is 2% for both the 40 μm and 75 μm particles. If the rubber particles are assumed to be spherical in shape, this size difference results in approximately 6 times as many 40 μm rubber particles as 75 μm rubber particles. Fabrication of syntactic foams is carried out in a step process, mixing and casting. First, the resin and diluent are mixed and heated to 50°C to further reduce the viscosity of the mix and insure that none of the epoxy is in a crystalline state. The lower viscosity facilitates higher filler loadings and aids in deaerating the mixture.

Hardener is then added and stirred thoroughly, followed by filler additions. The mixture is stirred gently to minimize the damage to microspheres during the mixing process and to entrap as small amount of air as possible. The slurry like mixture is then cast. This is done in stainless steel molds that are cured for at least 36 hrs at room temperature. After removal from the molds, they are post cured at 100±3°C for three hrs. This resulted in complete cross-linking of the polymer resin.

Even with care, these fabricated syntactic foams have some entrapped air due to mechanical mixing being the fabrication route. Due to the high stiffness of the
mixture air bubbles are inevitably produced and retained during incorporation. This entrapped air is termed as voids. Density and void volume fraction values of fabricated syntactic foams are presented in Table 8. It is also noted that due to the small size of the filler materials and the high loading percentage that settling is not observed during the curing process, therefore, the materials are considered macroscopically homogenous in properties throughout the sample.

Table 8 Density & Void Fraction of Samples

<table>
<thead>
<tr>
<th>Microsphere Type</th>
<th>Rubber Particle Size (um)</th>
<th>Corresponding Foam Nomenclature</th>
<th>Syntactic Foam Density (kg/m³)</th>
<th>Void Volume Fraction %</th>
</tr>
</thead>
<tbody>
<tr>
<td>S22</td>
<td>40</td>
<td>S 22R40</td>
<td>504</td>
<td>7.50</td>
</tr>
<tr>
<td>S32</td>
<td>40</td>
<td>S 32R40</td>
<td>563</td>
<td>14.83</td>
</tr>
<tr>
<td>S38</td>
<td>40</td>
<td>S 38R40</td>
<td>606</td>
<td>7.75</td>
</tr>
<tr>
<td>K46</td>
<td>40</td>
<td>K46R40</td>
<td>601</td>
<td>11.68</td>
</tr>
<tr>
<td>S32</td>
<td>75</td>
<td>S 32R75</td>
<td>516</td>
<td>7.11</td>
</tr>
<tr>
<td>S38</td>
<td>75</td>
<td>S 38R75</td>
<td>605</td>
<td>7.34</td>
</tr>
</tbody>
</table>

3.4 Specimen Nomenclature

The specimen nomenclature for hybrid foams is a six digit alphanumeric code. The first three letters represents the density of the microspheres, e.g. S22 are used in the nomenclature for microsphere density of 220 kg/m³. The third letter R represents Rubber Modified and last two digits are related to the average rubber particle size. A sample code for hybrid foams is S32R40, which contains rubber particles of 40 um size and microspheres of 320 kg/m³ density. The syntactic foam samples containing only glass microspheres in epoxy matrix have a three digit alphanumeric code such as S22. This nomenclature relates to the true particle density of microspheres as explained for hybrid foams.
4 RESULTS AND DISCUSSION

4.1 High Strain Rate Properties and Density Effects

4.1.1 Stress-strain Behavior

High strain rate compression test results of six types of hybrid syntactic foams are discussed here to establish the effect of strain rate and effect of syntactic foam density on the dynamic compressive properties of hybrid syntactic foams. The objective is to establish the effects of high strain rates and different filler materials have on the dynamic compressive properties of syntactic foams. Four foams of increasing density that used the 40 μm rubber particles as a matrix modifier will be tested using HSR. These foams used the S22, S32, S38, and K46 microspheres as the reinforcement. In addition, a comparison of the HSR properties of foams that use 40 or 75 diameter rubber filler materials will be done for the foams constructed with the S32 and S38 microspheres.

Verification of the equilibrium of the testing can be verified by examining the response waves for the incident, reflected, and transmitted waves. Assuming that dynamic forces in the incident and transmitter bars is equal the following equation can be written,

\[ E_R = E_T - E_I \]  

(15)

Careful coordination of the time frames of the signals results in Figure 7. The lines match up reasonably well. Some differences are noted in the first 5-15 μs area. This is not unexpected due to the calculation that ~9 μs is required for 95% equilibrium to establish between the incident bar/specimen and the specimen/transmitter bar.
Figure 4 Verification of Equation of Dynamic Equilibrium

Typical engineering stress-strain curves of the four foams constructed with the 40 μm rubber particles at strain rates of approximately 450/s, 800/s, and 900/s are shown in Figures 6-8. An almost linear initial region is found for each stress-strain curve where stress is directly proportional to strain up to about 1% strain. An observation for each stress-strain curve is that when the compressive strain is between 1.5 to 2.0%, the stress reaches its peak value. The density of the foam does not appreciably change the amount of strain at the peak stress. This suggests that the critical strain at which the peak stress is observed is not dependent on the microspheres but is a property of the matrix.
Figure 5 Stress vs. Strain for 4 hybrid foams at Strain rate of ~450/s

Figure 6 Stress vs. Strain for 4 hybrid foams at Strain rate of ~800/s
Figure 7 Stress vs. Strain for 4 hybrid foams at Strain Rate of ~900/s

The reflected strain wave is proportional to the strain rate history in the specimen. A reflected wave signal for a test of the S22R40 foam is shown in Figure 8. In this figure, the strain rises for a period of ~75 μs. After this rise time the specimen reaches a strain rate of 440/s (SD=10) for ~200 μs. The plateau area on the reflected pulse shows that the sample deformed at a nearly constant rate. 440/s was the lowest strain rate tested and did not result in fracture of this specimen. For all four of the hybrid foams fabricated none fractured in the ~450/s strain rate testing.

A reflected response of an example of the S22R40 foam that did fracture is shown in Figure 10. In this case, the strain rises in ~30 μs. After the rise a period of strain rate of 590/s (SD=25) occurs for ~180 μs. After this time, there is
a sharp rise in the strain rate where the specimen has fractured and there is a rapid increase in the incident bar/specimen interface velocity. The specimen is considered to have failed when a fracture occurs and is no longer able to carry a load.

For the highest strain rate, the S22R40 foam showed a markedly different reflected wave response. The Figure 10 shows the measurements. The rise time shows to be $\sim 25 \mu s$. The strain value only rises to a fraction of the strain seen in 440/s and 590/s strain rates. This signal doubles in value during the testing period of $\sim 125 \mu s$. During this testing period the strain rate is unstable and oscillates in the 700-900/s range. After the testing time, the reading rapidly increases as the specimen begins to crack and eventually fail after 250 $\mu s$ into the test.
Figure 9  Reflected Wave Signal for S22R40 Foam @ 590/s

A probable cause of the unstable readings was that the formation of microcracks occurred during the testing period. This would have resulted in movements of the incident bar during the test period causing the unstable readings.

The S32R40 foam shows very similar results for the reflected wave to the S22R40 foam at all three strain rates tested. An example of this is shown in Figure 12 where the reflected wave for the S32R40 is overlaid with a curve from the S22R40. The similar response suggests that the microspheres have minimal effect on the specimen strain property of the foam.
For the S38R40 and the K46R40 foams, the reflected response changed. An example of this is shown in Figure 12 from the K46R40 foam. The time period of the test was much shorter in duration. The rise time for the K46R40 density foam reduced in time and shows to be only ~5 μs. There is instability in this part of the curve until after ~10 μs due to wave reflections in the specimen and the state of dynamic equilibrium being established. The testing period continues for only ~30 μs during which the specimen has a strain rate plateau of ~860/s. After the testing time the curve rapidly increases for 10 μs after which cracking and fracture of the sample occurs.
The strain rate dependence of peak stress for various types of foams can be observed in Figure 13. It is observed that for the lower density foams (S22R40 and S32R40) only a small change in the peak stress occurs as the strain rate increases. This means that in case of the lower density foams, the strain rate sensitivity of peak stress decreases at higher strain rate values. At high strain rates, microspheres play the primary role in sustaining applied stresses.

For the two heavier density foams (S38R40 and K46R40) the peak stress amount increases linearly as the strain rate increases. In addition, as the density increases the relative amount of change in peak stress increases. The S38R40 foam shows an increase of 29% in peak stress as the strain increased from 440/s to 908/s. The K46 exhibited an even larger increase of 52% over the range of strain rates tested.
For the two heavier density foams, the wall strength of the microspheres stiffens the matrix resulting in higher maximum stress values.

Figure 12 Reflected Wave Signal for K46R40 Foam @ 860/s

Figure 13 Peak Stress values for hybrid syntactic foams vs. strain rate
A comparison of the peak stress for the S32R40/S38R40 and S32R75/S38R75 materials shows that the peak stress increases with the increase in size of the rubber particles. Figure 14 shows the results of the testing. On average a 20% increase in peak stress is measured for hybrid foams fabricated with the R75 rubber particles. These results were unexpected. In quasi-static compression testing the peak stress values were lower for hybrid syntactic foams with R75 rubber particles. For example in quasi-static, the S38R40 foam had a peak stress of 58 MPa while the S38R75 was 52 MPa.[10]

![Figure 14 Peak Stress of hybrid foams containing R75 and R40 rubber particles](image-url)
4.1.2 Effect of Strain Rate on Modulus Values

Figure 16 shows the change in the stress-strain curve of the S38R40 hybrid syntactic foam as the strain rate changes. It is clear that the specimen becomes stiffer as the strain rate increases. For all types of hybrid foams, similar effects are noted in the HSR results. These composite materials show that the effective stiffness changes with the strain rate. This viscoelasticity is a result of the matrix. Cross linked polymers and rubber elastomers both have viscoelastic response that is strain rate sensitive.

![Stress-Strain curves for S38R40 foam specimens](image)

Figure 15 Stress-Strain curves for S38R40 foam specimens

Compressive modulus measurements of all four of the hybrid syntactic foams fabricated R40 rubber particles presented in Figure 16. The modulus increases for all four foams ~60% as the strain rate increased from 450/s to 1050/s. This increase in
stiffness as the strain rate shows that the hybrid syntactic foam has a significant viscoelastic response to strain rate for all types microspheres.

The lightest density foams (the S22R40 and S32R40) showed very little difference in modulus values between these two densities. This small difference between the S22R40 and the S32R40 foams indicates that the matrix is the primary factor affecting the modulus.

The S38R40 foam had a ~70% higher modulus than the S22R40 and S32R40 results at all strain rates. The modulus values of the K46R40 were consistently ~80% higher than the S38R40 modulus values. The heavier wall thickness microspheres results in a synergy that increases the stiffness of the matrix. This effect stays constant in the range of strain rates tested here.

Figure 16 Modulus of hybrid syntactic foams vs. strain rate
Table 10 compares the compressive modulus of hybrid syntactic foams constructed with R40 rubber particles and the plain epoxy matrix syntactic foams under quasi-static and HSR testing. The plain syntactic foams shown here had 65% volume microspheres and used the same epoxy resin composition used in the hybrid syntactic foams. The only difference was that in the hybrid syntactic foams 2% vol. of the microspheres was replaced with crumb rubber.

Results show that for the S22R40 and S32R40 hybrid syntactic foams that the modulus decreases in comparison to the plain syntactic foams (S22 and S32) at comparable strain rates. For example at the strain rate of ~870/s the plain foam has approximately twice the compressive modulus compared to the hybrid foam. In the lightest density foams (S22R40 and S32R40) the addition of rubber particles results in a much more ductile composite material in comparison to similar plain syntactic foams.

For the S38R40 and the K46R40 specimens the modulus increases at a higher rate as the strain rate increases than it does in the S38 and K46 foams. The modulus of the S38R40 and K46R40 hybrid syntactic foams is less then the plain foam when the strain rate is less than ~800/s. At strain rates higher than ~800/s the hybrid syntactic is stiffer. Addition of rubber particles in the hybrid foams results in a decrease in stiffness when the strain rate is low to medium compared to plain foams.

The change in compressive modulus in HSR resulting from the use of 75 μm rubber particles rather than 40 μm particles is shown in Figure 17. The modulus of the S32R75 and S38R75 hybrid foam is more than double the identical foam fabricated with 40 μm rubber particles. This shows that the use of smaller rubber particles has a large effect on reducing the stiffness of the composite material.
Table 9 Comparison on modulus of hybrid and plain syntactic foams [10,20,30]

<table>
<thead>
<tr>
<th>Syntactic Foam Type</th>
<th>Strain Rate (/sec)</th>
<th>Modulus E (MPa)</th>
<th>Syntactic Foam Type</th>
<th>Strain Rate (/sec)</th>
<th>Modulus E (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Static</td>
<td>870</td>
<td>S22</td>
<td>Static</td>
<td>1547</td>
</tr>
<tr>
<td></td>
<td>480</td>
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<td></td>
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<tr>
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<td></td>
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<td></td>
<td>1688</td>
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<tr>
<td>S32R40</td>
<td>Static</td>
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<td>S22</td>
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<td>2025</td>
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<tr>
<td></td>
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<tr>
<td></td>
<td>878</td>
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<td></td>
<td>1164</td>
<td>2372</td>
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<tr>
<td></td>
<td>1017</td>
<td>1734 (SD=1.5%)</td>
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<td>2601</td>
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<tr>
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<td>S32</td>
<td>Static</td>
<td>2394</td>
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<td>437</td>
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<td></td>
<td>830</td>
<td>2796</td>
</tr>
<tr>
<td></td>
<td>747</td>
<td>2098 (SD=5.9%)</td>
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<td>1030</td>
<td>2888</td>
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<tr>
<td></td>
<td>909</td>
<td>3127 (SD=11.6%)</td>
<td></td>
<td>1324</td>
<td>1864</td>
</tr>
<tr>
<td>K46R40</td>
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<td>S38</td>
<td>Static</td>
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<td></td>
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<td></td>
<td>649</td>
<td>3396 (SD=23.5%)</td>
<td></td>
<td>1015</td>
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<tr>
<td></td>
<td>835</td>
<td>5282 (SD=12.3%)</td>
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Figure 17 Modulus of foams fabricated with R75 & R40 rubber particles
4.1.3 Modes of Failure

Failure modes of the specimens are evaluated using scanning electron microscopy (SEM). The fracture surfaces of the failed specimens are examined for characteristics of their failure. The samples are observed to have failed due to a crack forming at a shallow angle to the axial centerline. The samples were prepared for the SEM by coating with a thin layer of gold to protect the fracture surfaces from damage and the charge build up.

For the SFS22R40 and SFS32R40 fracture surfaces, large percentages of the visible microspheres were fractured. Example of images taken from the S22R40 and the S32R40 hybrid foam are shown in Figure 18 and 19 respectively. Nearly all the glass spheres at the fracture surface are shattered. The collapse of microspheres indicates that the material failed from compressive failure. In the case of HSR testing for the S22R40 and the S32R40 hybrid foams, the crack propagates directly through the microspheres.

In contrast, SEM images from the S38R40 and K46R40 foams show much less damage to microspheres. Image from the S38R40 fracture plane is shown in Figure 20. The S38R40 surface showed a small amount of debris from broken microspheres. The lack of broken microspheres and debris is an indication that the crack formed from tensile failure. The cause of the tensile strain is the expansion of the sample due to Poissons ratio during the HSR testing. The crossways tensile strain would exceed the capability of the matrix resulting in formation of the fracture. The fracture surface is well illustrated in Figure 21 and 22 of the K46R40. The surface is nearly devoid of any fractured microspheres. The failed surface is comprised of debonded microspheres or
the impression of microspheres from the corresponding surface. The matrix to microsphere interface is shown to have failed which is typical of a brittle like fracture.

![Figure 18 Image of S22R40 fracture surface](image18)

![Figure 19 Image of S32R40 fracture surface](image19)
Figure 20 Image of S38R40 Fracture Surface

Figure 21 Image of K46R40 Fracture Surface
Figure 22 Exposed microspheres in the K46R40
5 CONCLUSIONS

Four hybrid syntactic foams samples were fabricated using 2% volume percentage of solid rubber particles that had an average size of 40 \( \mu m \) as a matrix modifier. The rubber particles modified the epoxy matrix to reduce the brittle nature of the epoxy. The samples used glass microspheres as cellular filler. The microspheres had a common outside diameter but the wall thickness varied. The volume fraction of microspheres is 63%. Two types of samples were fabricated that were identical but used rubber particles that were 40 \( \mu m \) and 75 \( \mu m \) in size.

The high strain rate compressive properties have been tested with the Split Hopkinson Pressure Bar (SHPB). This testing is shown to be effective and reliable in characterizing the properties of hybrid syntactic foams in the strain rate range of 450/s to 1100/s. The conclusions from the SHPB testing are summarized as follows:

1. Increases in the density of the foam resulted in increases in fracture toughness. The synergy between the rubber modified matrix and the S38 and K46 microspheres resulted in a material with a higher viscoelastic response compared to plain syntactic foams.
2. The performance of the S32R40 foams is basically the same as that of the lighter S22R40 foam in HSR testing.
3. The larger 75 \( \mu m \) rubber particles increased the stiffness of the material and increased the strength compared to identical materials made with 40 \( \mu m \) rubber particles.
4. Peak stress for the S22R40 and S32R40 showed minimal increase as strain increased from 450/s to 1000/s. For the S38R40 and the K46R40 foams the peak stress increased as the strain rate increased.
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VITA

Paul Wehmer was born in Memphis, Tennessee in 1965. He graduated from the University of Arkansas at Little Rock in 1992. Paul worked in the industrial plastic packaging industry for 16 years. He worked in engineering and management for an international blow molding company that manufactured packaging for hazardous chemicals and food products. For the past two years, he has worked at a major refinery in the south Louisiana as a project engineer. He started his studies at Louisiana State University in September 2002. His technical interests are corrosion, material science, and fitness for service evaluations.