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# QUICK REACTION EVALUATIONS OF MATERIALS AND PROCESSES

## Delivery Order 0001: AFRL/ML Carbon Foam Round Robin Test Method Development

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A carbon foam round robin test and evaluation study was performed whereby several foam manufacturers provided material to be tested. Test methods used included compression (block and dogbone), shear (plate and torsional), tension, thermal conductivity, crushing with constrained piston, and compression modulus. Test specimen preparation procedures were discussed. Relevant definitions and equations related to test parameters were provided. This report allows one skilled in the art to independently replicate the test and evaluation procedures on a variety of carbon and graphitic foam materials.

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## 1.0 INTRODUCTION

Mechanical and Thermal Management applications are the inevitable future of carbon structures. Carbon's intrinsic thermal properties will enable many future industries with tools to perform lightweight and highly efficient thermal management for a wide variety of applications. Specific application areas include sandwich core materials, heat transfer management applications, and fire protection systems.[1] Designing suitable applications for carbon foam is difficult due to the relatively unpredictable mechanical behavior in relation to the three dimensional cellular structure of the foam.[2] Carbon foam is difficult to characterize without a common standard detailing the mechanical testing and evaluation of such an emerging and unique material. This report will explain current methods used by the Carbon Foam Round Robin participants. An explanation of each test procedure used to determine the Compressive, Shear, Tensile, and Thermal behavior of the various carbon foam materials follow.

## 2.0 **BACKGROUND**

Carbon foam is created from a wide variety of natural and synthetic precursor materials. The behavior of carbon foam is dependant upon several interdependent variables, i.e. carbon pitch type, chemical binder type, temperature, pressure, chemical foaming agent type, and the manufacturing process used. Each variable has a significant role in the resultant thermal and physical properties of the resultant carbon foam material. Research into characterization and manufacturing of cellular solids will allow for improved means to predict material behavior. Previous research and testing conducted by Gibson and Ashby[2] and A. Roy[3,4] and Alan Landis[5] have allowed the Round Robin participants to develop logical approaches for the mechanical testing and analysis of carbon foam. Few improvements were made during the previous round of testing in 2002, for testing in compression and shear.

Characterization of material behavior, when exposed to external forces, is necessary for designing to develop confidence in an emerging material. Accurate and repeatable testing allows a qualitative to quantitative comparison of the five carbon foam manufacturers' materials. The five manufacturers that were involved in the Carbon Foam Round Robin testing were Fiber Materials, Inc. (Biddeford, ME), Materials and Electrochemical Research Corp. (Tucson, AZ), Poco, Inc. (Greenwood, TX), Touchstone Research Laboratories, Ltd. ( Triadelphia, WV), and Ultra-met (Pacoima, CA). Each of these manufacturers has many applications for carbon foam that range from structural components to missile insulation to thermal components. Therefore mechanical characterization of carbon foam has become a focal area of MLBC and MLSC. This report will highlight new developments related to characterizing foams, and explain the rationale in testing and evaluating the carbon foam on a macroscopic scale.

## 3.0 TEST METHODS

A test method is a systematic plan designed to explain to others the correct and accepted approach for acquiring accurate data for the determination of material properties (e.g., elastic modulus and shear strength). The following descriptions of mechanical and thermal testing of carbon foam are intended to convey a clear understanding of the procedures for each test as well as discuss possible sources of error that could influence the accuracy of any acquired data.

Because the creation of foam test specimens can become an arduous task for individuals who are inexperienced in working with the delicate ligament microstructure of the foams, one should refer to the machining portion in this reports appendix (sections A.1 and A.2) for more detailed information on fabricating test specimens from carbon foam.

## 3.1 BLOCK COMPRESSION

Each block compression specimen was machined to the required tolerance, individually labeled and bagged, and sent to the Materials Test and Evaluation Team (MLSC). Upon arrival the specimens were washed in distilled water and dried at 130°F under vacuum for approximately twenty-four hours.

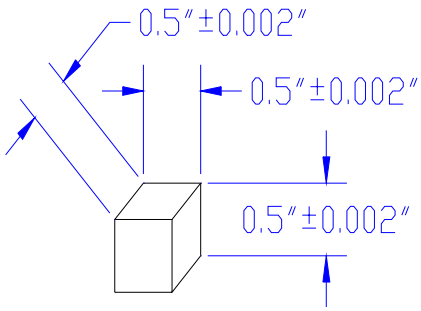



Figure 3.1.1
Block Compression Specimen

American Society for Testing and Materials (ASTM) method C365 was used as a basis for the block compression portion of testing.[6] This test method was used as a reference due to similarities between carbon foams and sandwich core foams. The majority of the ASTM C365 test method was used, with minor adjustments made due to the brittle nature of carbon foam. The testing machine used is a Materials Testing Systems (MTS) screw driven load frame equipped with a 458 MTS controller that was calibrated within the specifications of the test standard. The spherical bearing block is shown in the experimental setup in figure 3.1.2. The compression platens are standard MTS compression platens. The platens should be parallel to ensure accurate loading conditions that reduce the chance for eccentric loading. It is prudent to check the platens using an accurately machined or calibrated gage block and a light to gage if the platens are aligned correctly. The MTS deflectometer shown in figure 3.1.2, is aligned with axial travel of the platens and will accurately judge the movement of the crosshead while it is depressed against the bottom compression platen. A deflectometer was used because

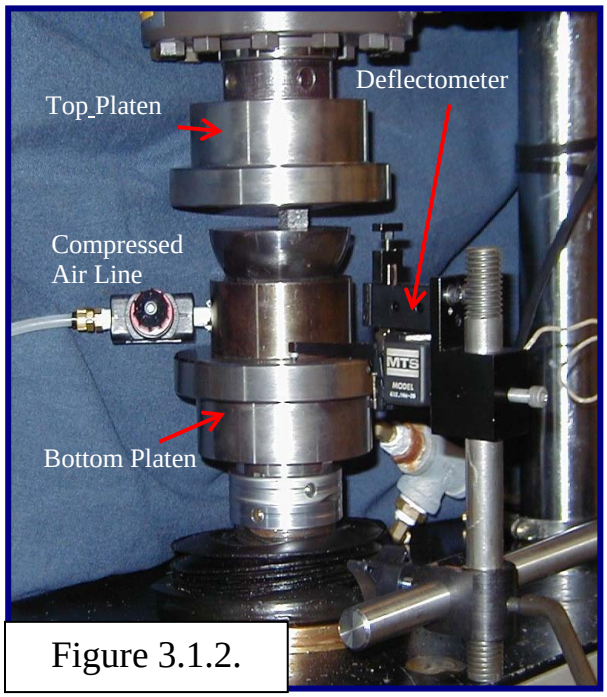



Figure 3.1.2.

the surface of most foams are easily damaged which rules out contact strain measurement for this material.

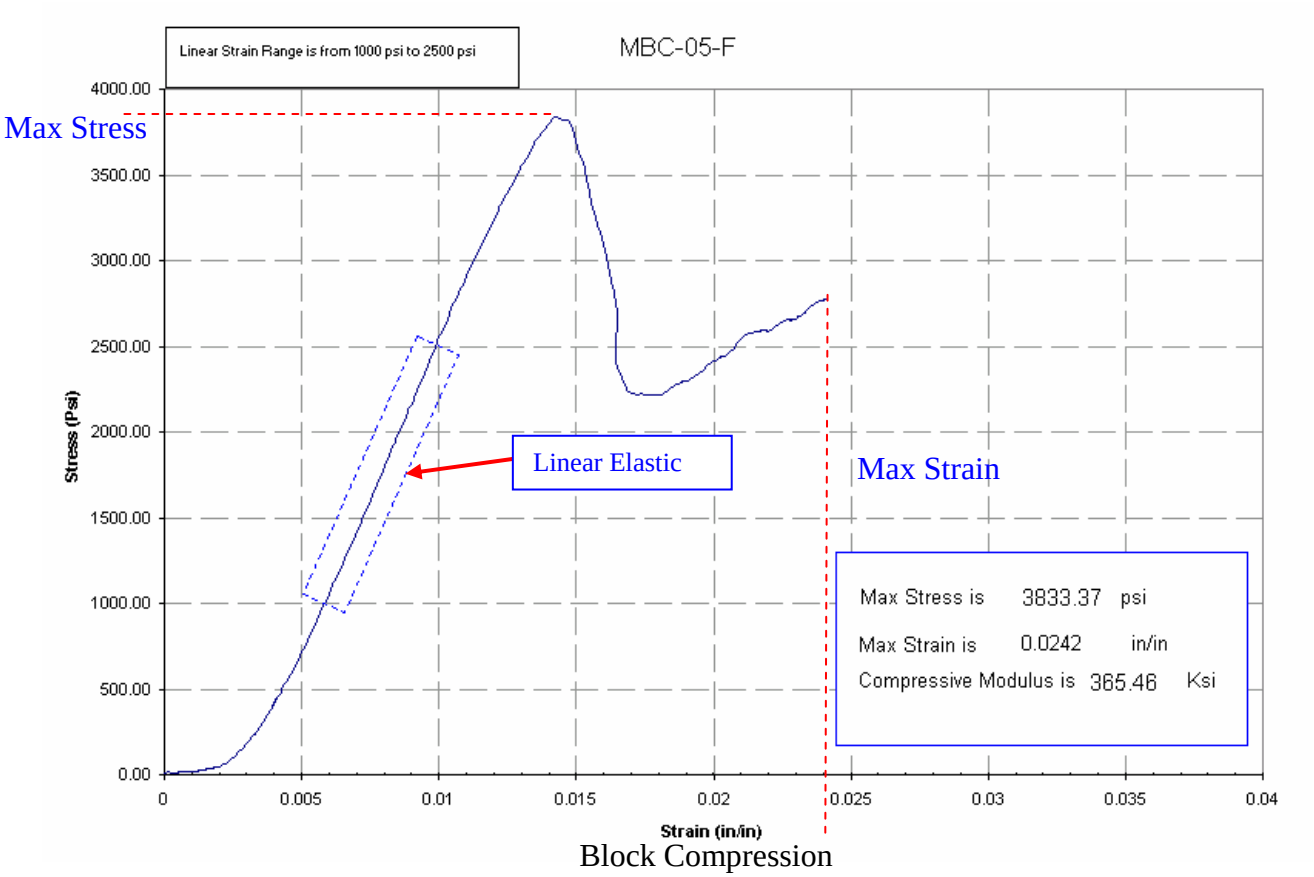
Each transducer that was used for this test method was calibrated prior to testing. The deflectometer was calibrated using an MTS calibration standard and a conditioning amplifier where 1 volt was equivalent to 0.002 inches of displacement. The load frame was calibrated at two kips or five kips full scale depending upon the amount of load generated by the foam specimens. For all block compression specimens, the rate of displacement for the cross head was 0.2 inches per minute. The crosshead speed was measured using a calibrated dial indicator for one minute to determine the inches per minute of cross head travel. The test was programmed into a MTS Micro-Profiler enabling a preset program to control the load frame.

Specimen density was measured and recorded prior to testing by means of Computer-Aided Tomography (CT). The CT images revealed a scale of densities and a direct correlation of the densities to actual values in (grams/cubic centimeter) x $10^{-3}$. The basic behavior and accuracy of CT imaging are within the ASTM specification E1441[7].

Testing consisted of eight specimens for each direction of foam (-x, -y and –z), relative to where the foam was removed from the initial carbon foam block. The data was collected entirely with LabView 7.0 express software. This data capture program also functions as a real-time XYY-plotter and is capable of recording the data and saving it in excel format. A plot of Stress vs. Strain for the MER foam specimen number 5 that is oriented in the X-direction follows in figure 3.1.3. Figure 3.1.3 was created in Excel from the digital data collected with a LabView data acquisition system.

Evaluation of the Stress-Strain plot is based upon the linear elastic portion of the curve; the effective compressive modulus is also obtained from this portion of the curve shown in figure 3.1.3. Refer to the appendix (section A.3) of this report for equation details.

Each block compression sample was loaded until the maximum load of the material was obtained. During the testing, you can often discern the beginning of failure by listening for audible popping within the material. Block compression mechanical test on open celled carbon foam resulted in questionable failures that could possibly make the data less accurate. The top and bottom surfaces of the block compression specimens are parallel and when exposed to a load via the platens, failure first starts directly against the platens. An accurate response from the entire sample is nearly impossible due to the failure occurring so close to the materials edge. The failures are normally manifested as collapsing cells of carbon foam that fracture primarily at the surface of the foam.




Block Compression
Stress vs. Strain Sample Curve
Figure 3.1.3

## 3.2 DOGBONE COMPRESSION

The dogbone compression specimens are machined as detailed in figure 3.2.1. Machining the reduced section of the dogbone compression specimens results in very little stress concentration within the reduced section; therefore, failure is intentionally forced at the center of the specimen. The failure of the reduced section is intended to be a more accurate representation of the behavior of carbon foam in compression.

Each Dogbone Compression specimens was machined to the required tolerance, individually labeled and bagged, and sent to the Materials Test and Evaluation Team for analysis. Upon arrival the specimens were washed in distilled water and dried at 130°F under vacuum. The dimensions for the dogbone specimens are shown in figure 3.2.1.

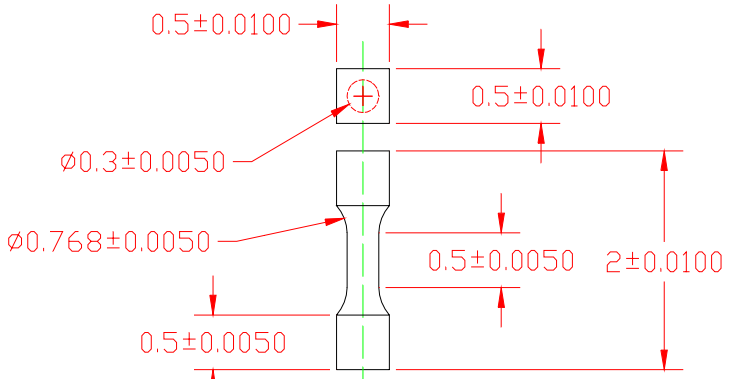



Figure 3.2.1
Dogbone Compression Specimen

Additionally, the dogbone compression specimens were bonded to aluminum to distribute compressive loads and promote failure inside the reduced gage section of the specimen instead of at the ends. EA9394 and EA9396 *Loctite* adhesive was used to adhere 0.5” x 0.5” x 0.125” aluminum stiffeners to the top and bottom of each dogbone specimen tested. *Flashbreaker* tape manufactured by *Patco Tape Inc*. was used to prevent excess epoxy from entering the foam anyplace except the bond-line near the aluminum stiffeners. Further explanation of the application of adhesive is located in the appendix.

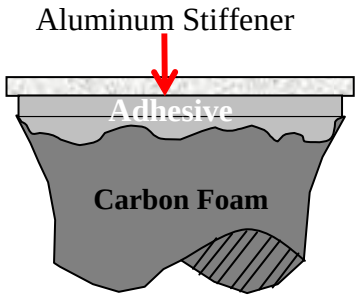
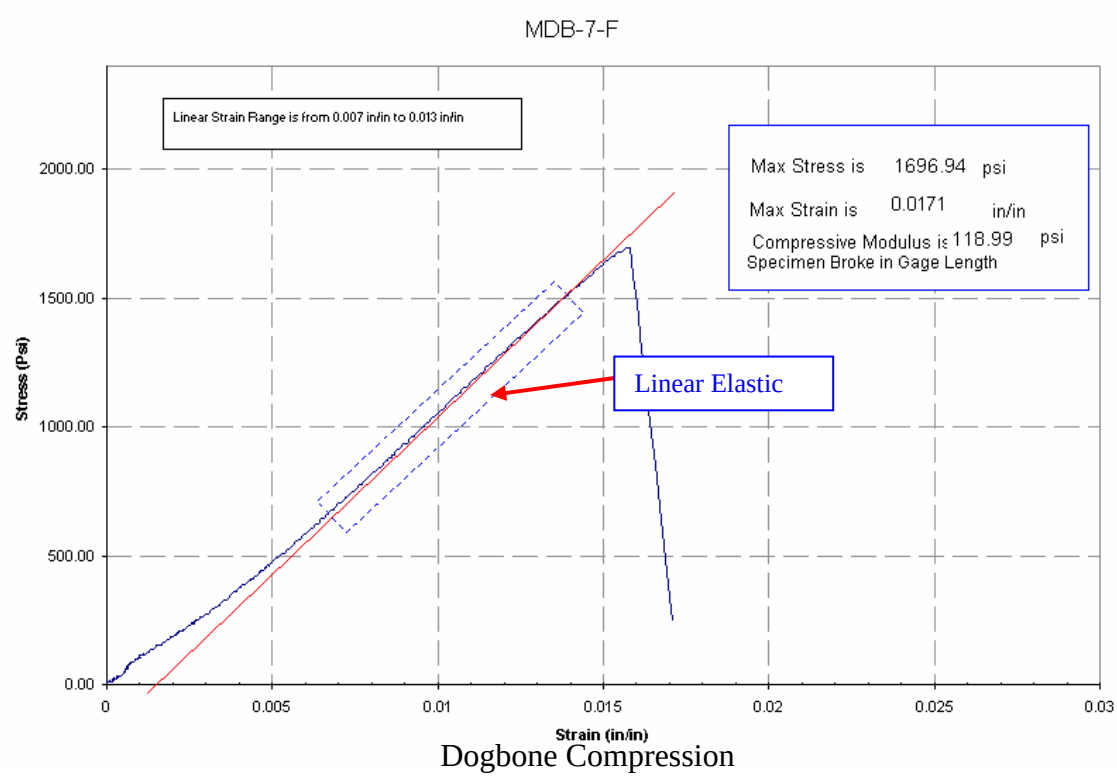



Figure 3.2.2
Bonding Aluminum Stiffeners to Dogbone Compression Specimens

The spherical bearing block was pre-loaded to 150 lbs to ensure accurate end loading conditions before each dogbone compression specimen was tested.

The experimental setup and procedure for the dogbone compression samples are identical to the block compression setup and procedure. The setup and procedure were identical to allow for a comparison of the block compression testing and the dogbone compression testing. This would allow a determination of which method would display a




Dogbone Compression
Stress vs. Strain Sample Curve
Figure 3.2.3

better representation of the mechanical behavior of the respective foam. Figure 3.2.3 shows a sample of a dogbone compression stress vs. strain plot. The stress vs. strain curve shows the linear elastic portion that is used to determine the compressive modulus. The equations that were used to determine the mechanical properties are detailed in the appendix.

## SHEAR TESTING

Determination of shear properties for carbon foam is based upon two common mechanical testing methods. Plate Shear and Torsion specimens have been used in previous testing by Alan Landis[5] and have been found to be useful in measuring the effective shear modulus of a material. A plate shear test is preferred due to the ease of machining test specimens, yet the test specimens are difficult to test. On the other hand, torsion specimens are more difficult to machine; however, torsion specimens are easier to test due to the torsional test machine[5].

## 3.3 PLATE SHEAR

Plate shear specimens were machined to the required tolerance, labeled and bagged, and sent in individually labeled bags to the Materials Test and Evaluation Team. Upon arrival the specimens were washed in distilled water and dried at 130°F under vacuum. The required dimensions for each plate shear specimen are shown in figure 3.3.1.

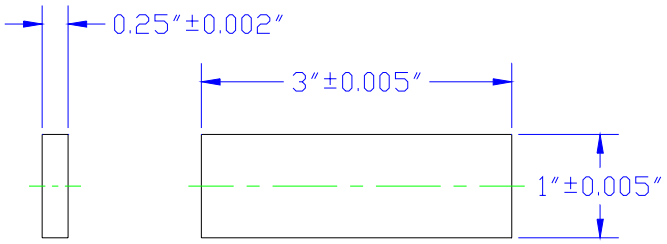
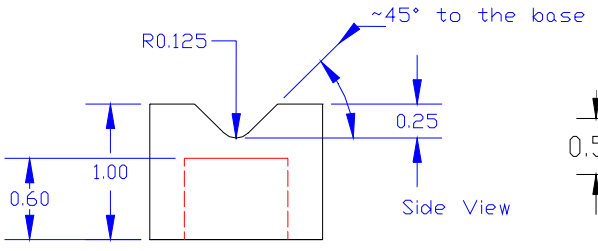
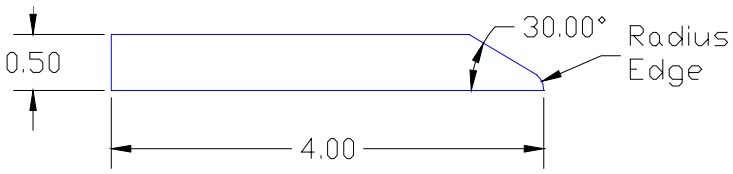



Plate Shear Specimens
Figure 3.3.1

The American Society for Testing and Materials (ASTM) specification for testing core materials in shear is ASTM C273.[8] Previous testing by Alan Landis was based upon the ASTM C273 test method and this round of testing is based upon his findings from the previous round robin. The aluminum plates are bonded to the carbon foam using



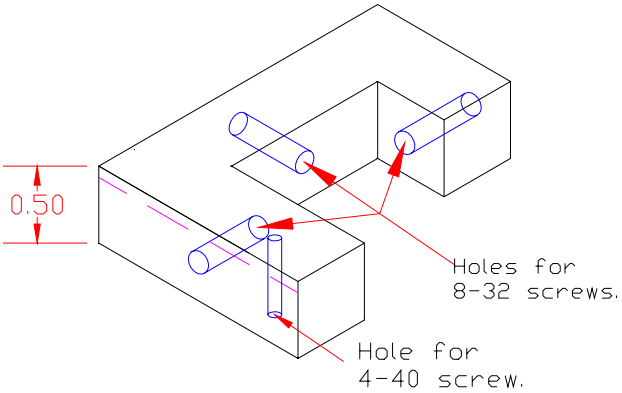

V-Block Base Plate
Figure 3.3.2




Aluminum Shear Plates
Figure 3.3.3

aerospace epoxy, detailed in the appendix. Each aluminum plate rests upon a v-block base plate; the blocks are detailed in figure 3.3.2. The shear specimens are tested using the compression setup shown in ASTM C273. The compression setup allows for the technician to have a similar setup to the block compression setup shown in section 3.1 and also in ASTM C365. Two parallel compression platens are used; however the spherical bearing block is not used for this portion of testing. The aluminum shear plate dimensions are shown in figure 3.3.3. The extensometer fixture was designed in the previous round of testing carried out by Alan Landis[5]; this fixture was necessary for strain monitoring of the carbon foam. The drawing for the extensometer fixture is detailed in figure 3.3.4. The plate shear experimental setup is detailed in figure 3.3.5.

The test speed was doubled to 0.4 inches of displacement per minute for the plate shear testing. The extensometer was recalibrated, using a Materials Testing Systems (MTS) calibration standard; 0.1 inches of displacement were made equivalent to 10 volts. The load was set to a 5000 lb full scale. Each test was terminated after a noticeable drop in the load on the specimen.




Extensometer Support Fixture
Figure 3.3.4

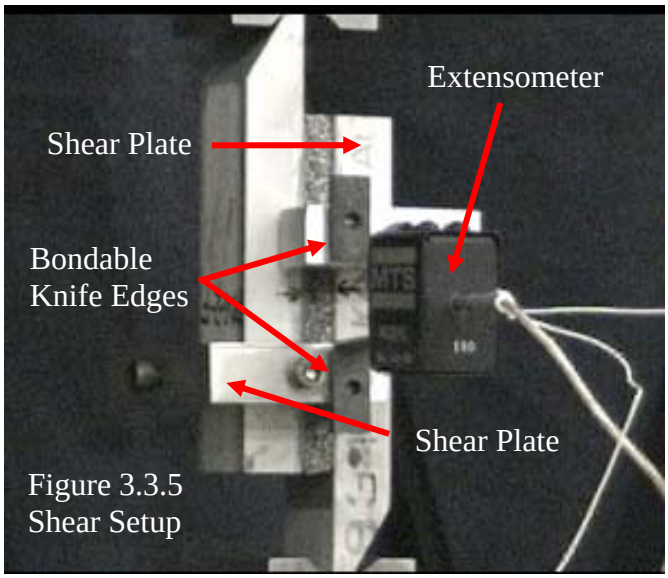



Figure 3.3.5
Shear Setup

The epoxy was a noticeable source of error during this test. It was possible for the epoxy to permeate into the first 0.20” of the carbon foam during bonding, shown in figure 3.3.6. This caused reinforcement in the carbon foam microstructure to occur. While reinforcement makes most materials stronger, it can also influence the mechanical behavior of any specimen tested. This influence increased the strength of certain foam specimens that were capable of absorbing epoxy into the existing cellular structure.

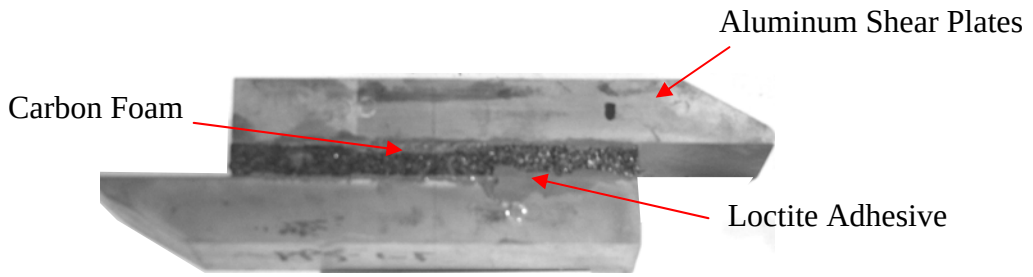
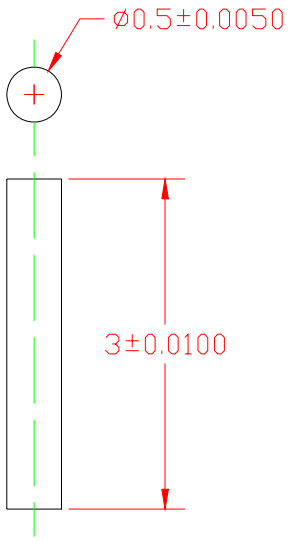



Plate Shear Specimen with Adhered Aluminum Shear Plates
Figure 3.3.6

## 3.4 TORSIONAL SHEAR

Cylindrical torsion specimens were fabricated according to figure 3.4.1. Specimens were tested by AFRL/MLBC.




Torsion Specimen
Figure 3.4.1

The difficulty in generating a desirable failure mode in Iosipescu shear specimens may be attributed to excessive stress concentrations of the foam ligaments in the vicinity of loading rollers. Thus, a cylindrical specimen configuration (for example, circular rods) subjected to an axial torsional load is considered to be a preferred test configuration for this material to measure its shear properties. The schematic configuration of the specimen is shown in figure 3.4.2, as well as, the foam specimen with slotted end tab is shown in figure 3.4.3. The cylindrical specimen configuration of applying torque at the specimen ends, in contrast to Iosipescu, Double rail and Single rail shear tests, significantly reduces the stress concentration at the loading locations, i.e., the specimen ends. A special miniature torsion fixture was designed to measure the shear stiffness and strength of the material, as shown in figure 3.4.4 [3]. Due to the low bulk modulus of carbon foam (generally, one order of magnitude lower than that of PVC plastic), a careful attention was given to the low load requirement in designing the test fixture. Since strain gages could not be installed on the specimen surface, shear strain was determined from specimen end rotation (with a special design) to obtain an angular rotational resolution of 0.005 degrees. Further, to relieve the axial constraint (i.e., zero end load) while twisting (applying torque) the specimen, the specimen-gripping end attached to the load cell is mounted on a linear roller. In addition to testing foam, this miniature torsion tester is also designed to allow the convenient and accurate torsional testing of small samples of various low stiffness materials. The design configuration and calibration of the fixture, as well as the data reduction of the test data to calculate the shear modulus, developed by Roy and Camping [3], is described below.

General Configuration: The test machine consists of a torsion drive section and a reaction section that are both mounted on an aluminum base. The torsion drive section uses a

computer-controlled stepper motor to apply a torsional displacement to the material under test. The reaction section contains a torque load cell that measures the applied torque (see figure 3.4.5(a) and figure 3.4.5(b)).

Support Base: The base is made of a 76.2 x 44.5 x 6.35 mm (3" x 1.75" x 0.25") aluminum channel with 12.7 mm (0.5") thick aluminum end plates. The overall size of the base is 127 x 343 x 76.2 mm (5" x 13.5" x 3"). The main consideration in constructing the base is that it be able to resist the torque produced by the machine without significant deflection. The top surface of the base is machined flat to provide a stable platform for the drive and reaction sections.

Drive Section: The drive section is constructed on an 89 x 165 x 12.7 mm (3.5" x 6.5" x 0.5") aluminum plate, which is bolted to one end of the base. The drive itself consists of a Frame 23-stepper motor with a 30 oz capacity geared to the output shaft through two sets of gear reduction. The first gear reduction is a 3:1 timing belt drive that drives a 60:1 worm gear reducer to the output shaft for a total reduction of 180:1. All elements of the torsion drive are mounted in precision ball bearings to reduce friction in the system as much as possible. The worm drive consists of a hardened and ground steel worm driving a brass worm gear. The output shaft is a 6.35 mm (0.25") diameter steel shaft aligned parallel to the long axis of the base. All of the mechanical parts of the drive section are protected by an extruded aluminum cover.

Angular Measurements: Provisions have been made in the drive section for either a potentiometer or a shaft encoder to be added to the drive unit to measure the angular deflection applied to the specimen. The current version accomplishes this measurement in the control software by simply counting the number of steps applied to the motor. The capacity of the motor coupled with the high torque multiplication due to the gear reduction ensures that the motor will never "stall" so the software calculation of the applied angle will always be accurate to within one step. The stepper motor is a standard 200 step per revolution motor so the final resolution of the drive is:

$$360 / (180 \times 200) = 100 \text{ Steps/Degree (or 0.01 Degree/Step)}$$

Since the control software operates the motor in "half-step" mode, this resolution doubles to 200 Steps/Degree or 0.005 Degree/Step step (or $8.726 \times 10^{-5}$ Radian/Step). This resolution was found satisfactory for the preliminary testing.

Reaction Section: The reaction section consists of a reaction base, which contains two commercial ball slide assemblies that are aligned parallel to the test axis of the machine (the long axis of the base) and the mounting plate for the reaction torque cell. The purpose of the ball slides is to allow the torque cell the freedom to move along the torque axis of the machine while resisting the applied torque. This ensures that no axial loads are applied to the specimen. The total range of motion of the torque cell is approximately 1 inch (25.4 mm). This allows the operator sufficient room to insert each specimen into the gripping fixtures of the machine. The location of the reaction section on the base is also adjustable over about a 4 inch (101 mm) range. Once the reaction section has been located in a suitable position for the specimen being tested, it can be locked in place using

two locking screws. This allows specimens to be tested in a range from less than 0.5 inch (12.7 mm) to about 3.5 inch (76 mm) in length.

Torque Measurement: The torque cell is a commercial unit with a 100 lb capacity (Transducer Techniques, Inc., TRT-100). The signal from the torque cell is amplified by a commercial strain gage signal conditioning system and fed to a 16-bit resolution Data Translation data acquisition card in an IBM PC-compatible computer. The torque cell is calibrated before each set of tests using a 10.00 inch (254 mm) lever arm and a set of standard laboratory gram weights.

Specimen Attachment: The carbon foam specimens for this program were limit in size by approximately 0.3 inch (7.6 mm) diameter by 2 to 3 inch (50 to 75 mm) length due to the small size of the carbon foam preforms. Carbon foam specimens were fitted with slotted end ring or solid tabs (figure 3.4.2 shown with slotted ring tab) for easy gripping to the torsion tester. Due to this limitation, the torsion tester was fitted with grooved collar adapters to grip the specimens through the slotted end tabs. These adapters can be easily modified or replaced to accommodate specimens of different diameters.

Control Software: The control software was written in Microsoft Quick Basic, Version 4.5. The program allows the operator to enter specific information about the specimen and designate a file name for the data. The output data file contains the torque and angular displacement values in engineering units for each step of the motor (every 0.005°). This data is in ASCII format and may be imported into a spreadsheet program, such as Excel, for further analysis or plotted using any of the commercially available plotting programs.

Calibration: The fixture was calibrated in two stages. First, the response of the load cell was calibrated to a known applied torque and then the whole torsion fixture by measuring shear moduli of two plastics, PVC and Urethane. The modulus of Urethane is the same order of magnitude as that of low-density carbon foam, whereas the modulus of PVC is about five times that of Urethane. Thus, calibrating the test fixture with these two materials is expected to provide a good range for measuring shear properties of carbon foam. The details of the calibration are described in Development of a Portable Shear Test Fixture for Low Modulus Porous (Foam) Materials [3].

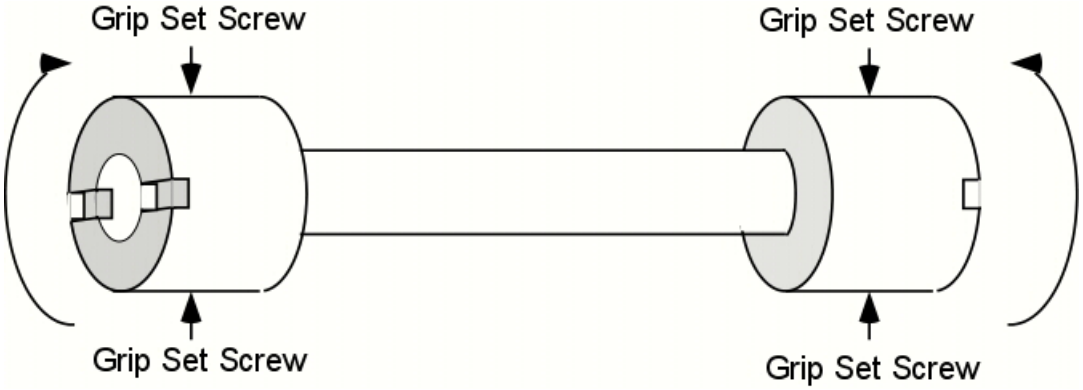



Figure 3.4.2. Schematic specimen configuration (circular rod) of the torsion specimen with slotted end tabs.

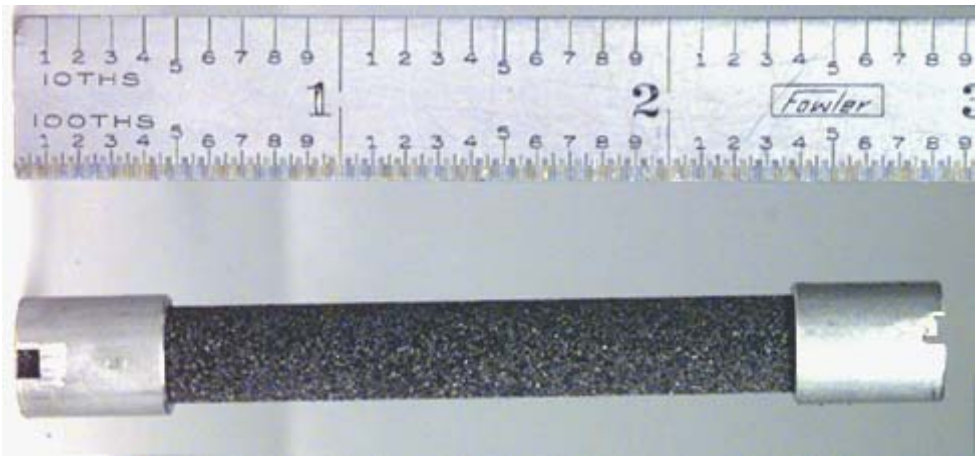



Figure 3.4.3. Carbon foam specimen of circular cross section with slotted end tabs.

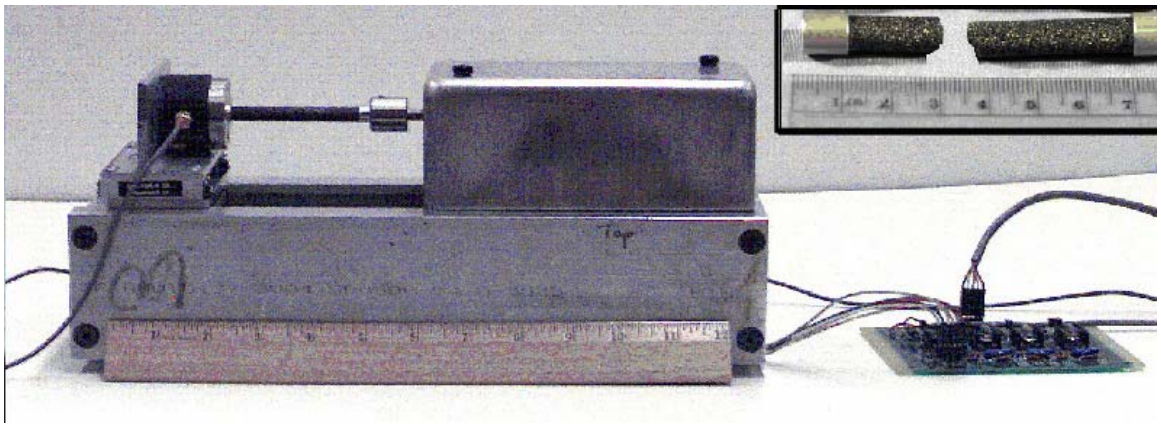


Figure 3.4.4. Miniature torsion fixture for measuring shear modulus and strength of foam. The inset at the top left corner in the figure shows a failed foam specimen.

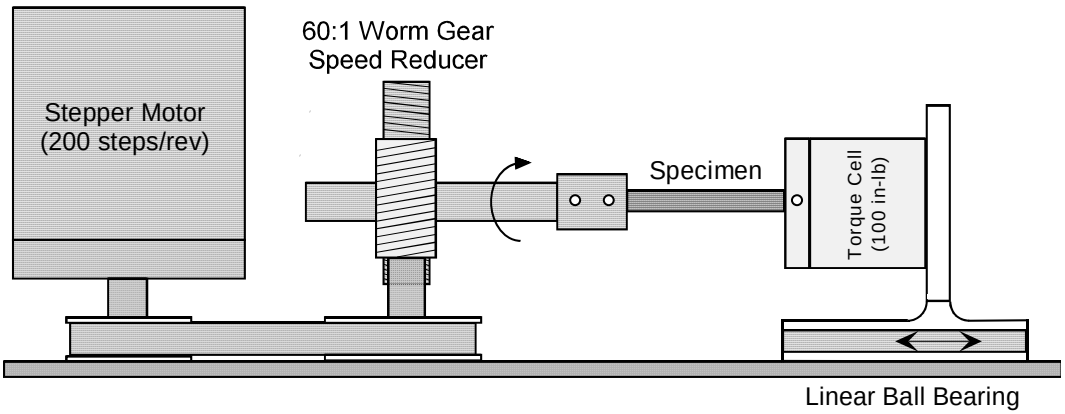



Figure 3.4.5(a). Schematic configuration of the shear test fixture, side view.

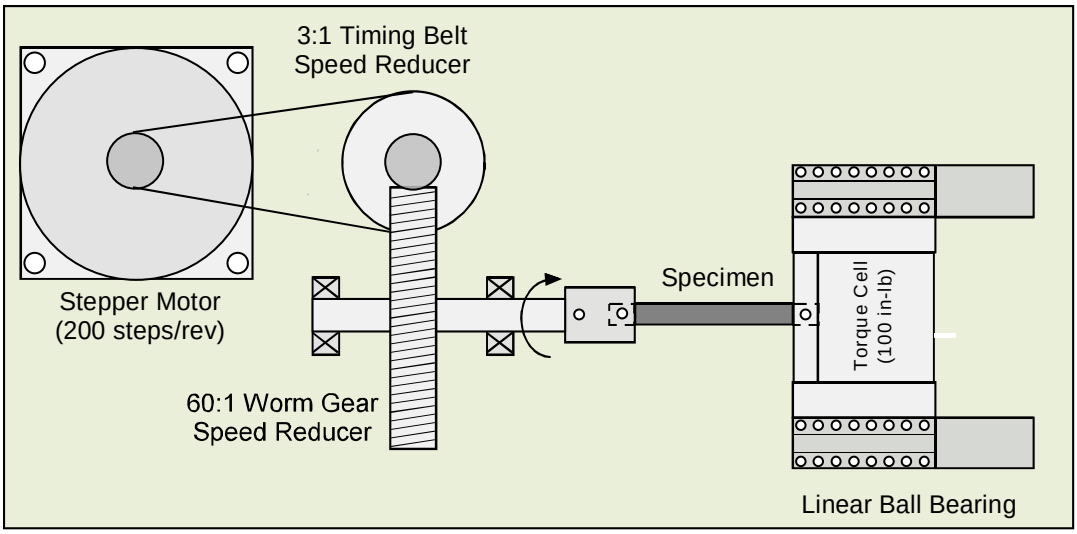
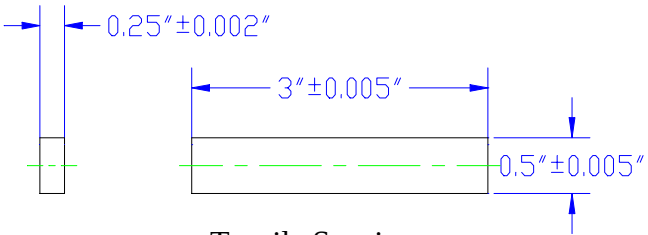



Figure 3.4.5(b). Schematic configuration of the shear test fixture, plan view.

## 3.5 TENSION

Tensile specimens were machined according to figure 3.5.1 [9]. The tensile specimens were tested by AFRL/MLBC.

Due to high porosity and relatively low strength of the material, the tensile coupons prepared from the foam material could not be directly gripped with the testing frame's hydrostatic end grips. Tab materials were bonded to the two ends of the foam specimens in order to apply a uniform displacement on the foam sample subjected to a tensile load. Then the tab materials were pin loaded to apply the tensile load to the foam specimen (figure 3.5.2). Tensile specimens shown in figure 3.5.2 were tested to measure the stress-strain behavior. The strain applied to the specimen was calculated from the machine head displacement, assuming the amount of strain in the bond between the tab and the foam is negligible compared to that of the material in the gage section. Further, all the specimens tested in these two specimen configurations failed in the gage section. Thus, in view of the simplistic specimen configuration, the straight-edge specimen appeared to be the convenient specimen configuration and suitable for the tensile testing for the graphitic foam.




Tensile Specimen
Figure 3.5.1

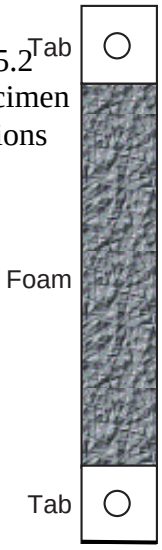



Figure 3.5.2
Tensile Specimen
Tab Locations

## 3.6 THERMAL CONDUCTIVITY

### 3.6.1 Thermal Conductivity Measurement of Carbon Foams using Flash Diffusivity Method

Carbon foam samples were machined to 1 inch cubes and tested in the flash diffusivity instrument. This testing was done at Ohio University and at Oak Ridge National Laboratory. Some samples, which were low conductivity, were machined to a smaller thickness before testing was done. The equipment software carries out the curve fitting for each test, and determines the diffusivity for the samples. The theoretical basis for this measurement is described below.

The laser flash method was proposed by Parker, Butler, Jenkins, and Abbott of the U.S. Navy Radiological Defense Laboratory in 1961.[10] It is the most popular method for measuring the thermal diffusivity of solids. Since its introduction in 1961, the flash method has become a standard testing method for thermal diffusivity measurements of solids.

In this method the front face of a small plane insulated sample (usually disk-shaped) is subjected to a very short burst of radiant energy coming from a lamp or laser. A temperature detector is used to record the temperature change of the rear face of the sample. This temperature history is used to determine the time needed for the temperature to reach half of its maximum value. This "half-max-time" is the basis for the calculation of the thermal diffusivity.

Assuming that the laser beam heats the sample front surface uniformly, the heat source produces a one-dimensional temperature field described by the following differential equation.

$$\frac{\partial T}{\partial t} = \alpha \nabla^2 T \tag{1}$$

Applying the appropriate initial and boundary conditions the solution of the Equation (1) of Parker et al.[10]:

$$T_{end} = \left[ 1 + 2\sum_{n=1}^{\infty} (-1)^n e^{-\frac{n^2\pi^2\alpha}{L^2}t} \cos\left( \frac{n\pi(L-x)}{L} \right) \right] T_{\max} \tag{2}$$

where $T_{end}$ is the sample total temperature variation, $\alpha$ the thermal diffusivity and $L$ the sample thickness.

Normalizing the equation with respect to $T_{max}$, we have:

$$V(\tau) = 1 + 2\sum_{n=1}^{\infty} (-1)^n e^{-n^2\tau} \tag{3}$$

where $\tau = \frac{\pi^2 t\alpha}{L^2}$ is the non-dimensional time

$V = \frac{T_{end}}{T_{\max}}$ is the non-dimensional temperature.

Equation (3) above provides the temperature of the back surface as a function of time. The experimental data is fitted with the expression in equation (3) to determine the thermal diffusivity. The data, plotted in dimensionless form is shown in figure 3.6.1.

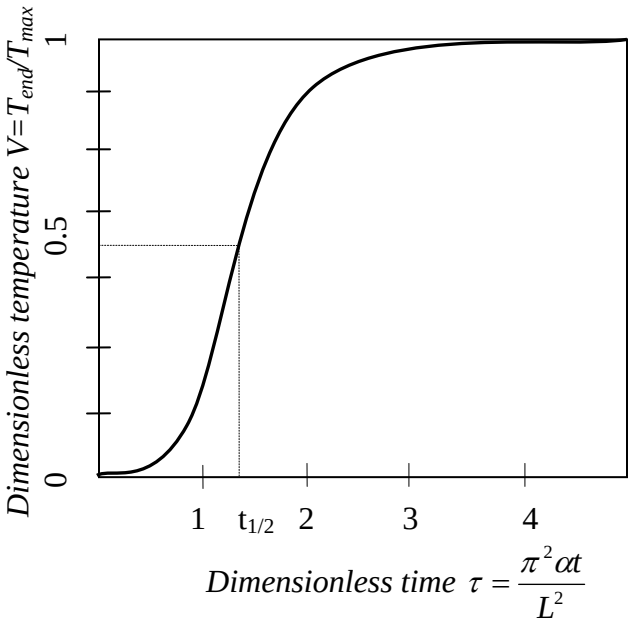



Figure 3.6.1. Dimensionless plot of rear temperature.

The dimensionless temperature ($V$) reaches the value of 0.5 at the dimensionless time ($\tau$) value of 1.37. Therefore the thermal diffusivity $\alpha$ can be calculated from half time ($t_{1/2}$) using the relation:

$$\alpha = \frac{0.1388}{t_{1/2}} L^2 \qquad (4)$$

where $t_{1/2}$ is the time from the initiation of the energy pulse until the rear face temperature reaches one-half of its maximum value (as it can be seen in Fig. 1, above) and $L$ is the sample thickness.

The thermal diffusivity $\alpha$ also offers a convenient and accurate method of finding the thermal conductivity $K$. The relationship between $\alpha$ and $K$ is given by $K = \alpha\rho C_p$ where $C_p$ is the specific heat and $\rho$ is the density. The specific heat and density can be measured or even calculated based on the known values of the constituent

elements. Thus it can be easier to measure $\alpha$, $C_p$, and $\rho$, and calculate $K$, than it is to measure K directly.

The flash diffusivity instrument is schematically presented in Fig.3.6.2, below:

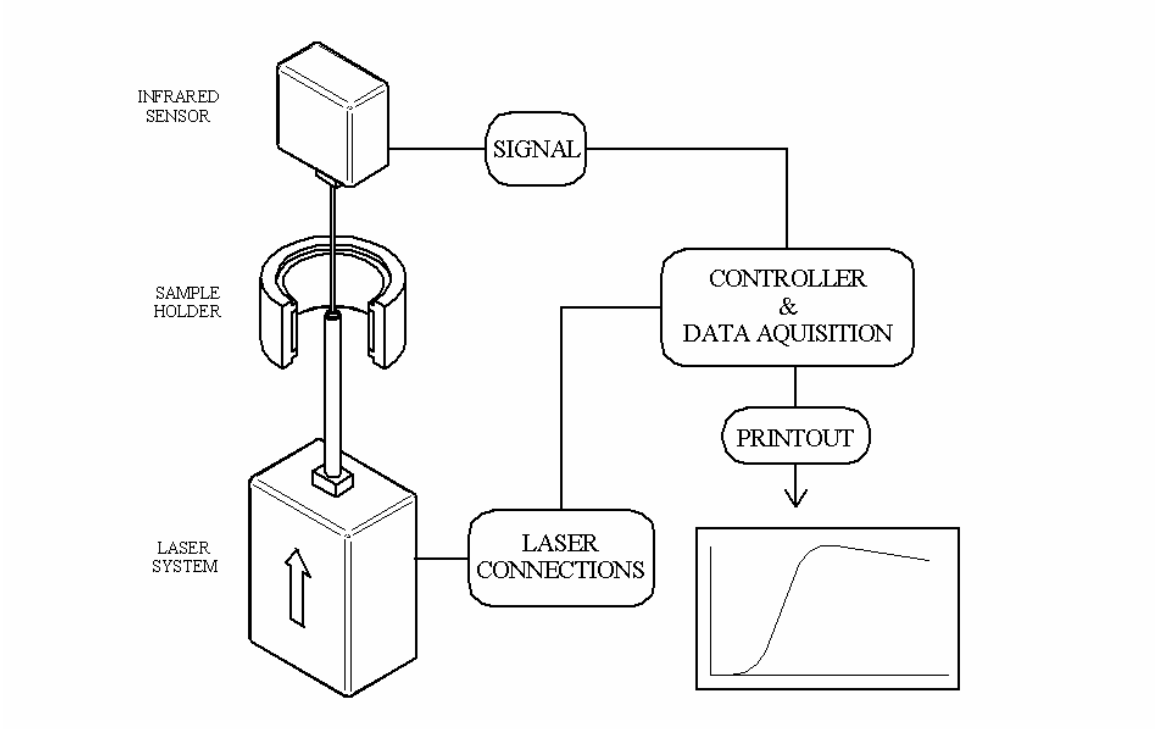



Figure 3.6.2. Laser flash instrument apparatus.

The samples were tested in x, y and z directions. The specimen was tested with the greatest thickness possible so that the effect of pores on the surface is minimized. Each test was repeated at least 5 times and the average value was taken. The conductivity value was then calculated as discussed above.

## 3.7 CRUSHING WITH A CONSTRAINED PISTON

Compression tests on the carbon foam were performed using an Instron 5869 load frame fitted with a 50 kN load cell. This testing was performed at West Virginia University. The two platens compressed the samples at a rate of 1 mm/min. WVU uses an encased-piston assembly constructed out of stainless steel in order to control the sample cross-section during compression testing. This device is useful for measuring strength after initial compression failure occurs. Without the use of an encased piston, large pieces of the sample can fracture, resulting in a poorly defined cross-sectional area of the sample.

When the piston is used, the volume change is essentially one dimensional as the samples are compressed. Thus, a uniform sample might be expected to demonstrate near constant stress as it is compressed from its initial volume to its final packed state, in which essentially all void-space has been removed.

The stainless steel cylinder used for compression measurements has an inner diameter of 1.0 in. A 3.0 in long solid piston and a 0.50 in long solid plug fit inside the cylinder. The plug is employed as a false bottom for the sample chamber cavity to ensure the crushed sample can be removed.

The foam samples are cut out using a regular 1.25 in carbide-tip hole saw on a stationary drill press. This cut results in a specimen with a 1.0 in outer diameter (OD), a tight fit for the sample cylinder. Once the samples are cut out, the ends are cut parallel using a diamond impregnated blade on a wet tile saw. The cut samples are set aside to dry before they are tested. The dry samples are weighed to the nearest 0.01 gm using a balance, and their dimensions taken using a caliper to the nearest 0.001 in. These measurements are used to calculate the samples' apparent density.

A dry sample is placed in the sample chamber between the plug and the piston. The sample chamber is centered on the lower platen of the Instron load frame. The upper platen is lowered to a point where it simply touches the top of the piston of the sample chamber. The Instron test can then be carried out.

The purpose of using the sample chamber is to gather data that can yield the total energy absorbed by each foam sample. The total energy absorbed per unit volume is found by integrating the area under the Stress vs. Strain curve or plot. Integration was carried out using the Newton-Cotes closed integration formula using:

$$A = \frac{\sum_{1}^{i} x_i y_i}{100}$$

where A is the energy absorbed per unit volume, and

$$y_i = \frac{y_n + y_{n+1}}{2}$$

represent the arithmetic mean of stress at the *i*th interval as a function of the arithmetic mean strain.

$$x_i = x_{n+1} - x_n$$

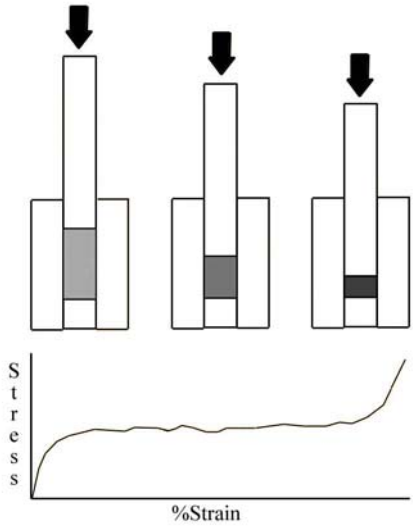
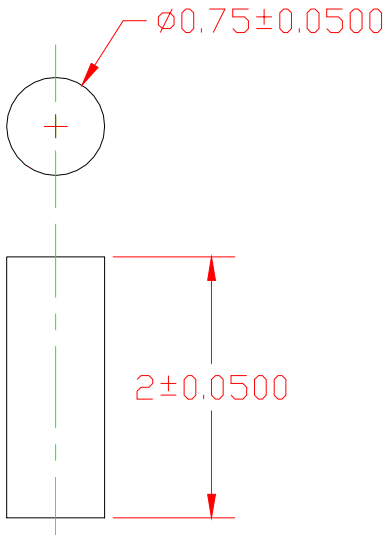


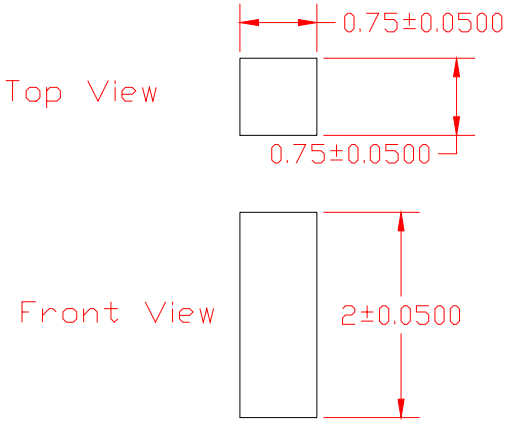

Figure 3.7.1. Schematic of Sample Chamber and Corresponding Stress vs. Strain Response

## 3.8 COMPRESSION MODULUS

Each test specimen was machined to the required tolerance, individually labeled and bagged, and sent to the Materials Test and Evaluation Team (MLSC) for analyses. Upon arrival the specimens were washed in distilled water and dried at 130°F under vacuum.




Modulus Rod Specimen
Figure 3.8.1




Modulus Block Specimen
Figure 3.8.2

Modulus specimen testing is merely supplementary to determine if a more accurate representation of the compression modulus is attainable through the same approach as the block compression specimens. This test is based upon American Society for Testing and Materials specification C365.[6] The dimensions of each specimen were measured and recorded. The modulus specimens were designed as shown in figures 3.8.1 and 3.8.2. Stress concentrations form at the corners due to the specimen geometry (i.e., rectangular block). Therefore, cylindrical specimens were made to test to determine if the compression modulus results will be different due to less stress concentrations.

The test setup for the modulus blocks is shown in figure 3.8.3 and for the modulus rods is shown in figure 3.8.4.

Each of the experiment was carried out using an MTS load frame controlled by a MTS 458 controller. The test speed was held constant at 0.05 inches per minute to ensure an accurate representation of the mechanical behavior of test specimen. The specimens generally were found to exhibit behavior similar to the block compression specimens. The major difference in behavior is the increase in the load that is necessary to fail each specimen when compared with the loads recorded in the block compression testing.

Figure 3.8.3
Modulus Block Specimen
Experimental Setup

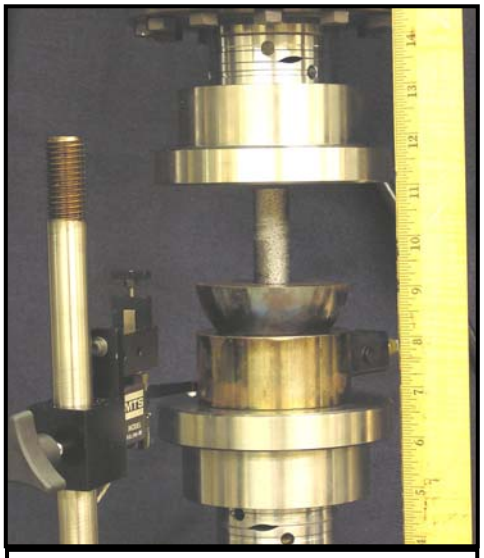


Figure 3.8.4
Modulus Rod Specimen
Experimental Setup

Extension and load values were digitally recorded using LabView, and the recorded data file was saved as an excel sheet to be reduced at a later date. The samples were compressed at a constant rate controlled by the load frame. The compression platens are flat and should ensure uniform loading on each compression sample. Extension was recorded using a deflectometer as described previously in the block compression section of this report.

## 4.0 CONCLUSIONS

Each mechanical test was used to evaluate certain behavioral characteristics of the various carbon foams. Evaluation of the bulk properties of this porous cellular solid will result in a better understanding of the structural behavior of relatively large samples of carbon foam. Understanding that each type of mechanical test may or may not be suitable for all materials, newer forms of some conventional ASTM testing methods must be instituted to properly evaluate carbon foam. The benefits and drawbacks to each form of testing are also important for any future work (i.e., identify trouble areas associated with each test method).

The block compression testing is generally an easy test to prepare; there is minimal machining and specimen preparation when compared to other types of foam testing. Measuring the samples is simple as well the cross-section of the foam is required for stress calculations, very easy for a cube. Modulus is easily evaluated as the linear portion of the stress strain curve. However, a few drawbacks are associated with this type of testing. The first is actually foam dependant. Any cellular structure has a network of ligament structures that comprise the entire material. The foam is an open-celled cellular structure, which is not ideal for compression testing on a flat surface. The open cells slowly collapse upon themselves creating small and erratic drops in load, and because the cells collapse on other cells the load continues to increase until the next level of cells collapse. This behavior is easily identifiable and it is called surface crushing. The small loads and surface crushing of the foam make this test relatively difficult to perform on synthetic pitch based foams, because of decreased density and strength.

The dogbone compression specimens are difficult to machine because of the reduced section of foam that is required at the gage length. This test is intended to be a much more accurate representation of the compressive properties of carbon foam. The reduced section of foam allows for one to also reinforce the surfaces of the carbon foam that are exposed to the compression platens. The surfaces are reinforced with aluminum stiffeners that are bonded with epoxy. This makes the results much more reliable and if everything goes well, repeatable. One source of error that could be attributed to this test is the assumption that each aluminum stiffener is uniform and flat. This can result in the platens becoming non-parallel. Generally this test is intended to be overall better than the block compression test. However, it is not always as easy to perform. The aluminum stiffeners and the epoxy are the weak points of this test. If they are bonded incorrectly or aligned incorrectly it is hard to anticipate the affect enacted upon the samples. Assuming ideal conditions, this test is excellent for all types of foam.

The tensile and torsion specimens were not changed since the previous round robin initiative. Therefore all the results and conclusions of that portion are identical[3,5].

The Plate Shear testing was performed according the previous Round Robin effort. The testing was performed as repeatable as possible, however the amount of epoxy that was forced into the foam varied with each specimen. The amount of epoxy that was inside the foam greatly influenced the accuracy of this type of test. Most groups of specimens had large variance in max stress and strain and shear modulus values. This inconsistency

has led us to believe that this test may not be accurate enough to correctly characterize carbon foam unless the epoxy bond line can be controlled.

The crushing and thermal conductivity testing that was performed utilize standardized test procedures. Thermal conductivity test are based off of 1961 technology, whereas the crushing done with a constrained piston is straight forward.

Modulus testing was a unique test exclusive to this Round Robin effort. The ideas came from a meeting of MLSC and MLBC personnel involved in the initiative. The group decided that a larger and longer foam sample may be able to represent a compressive modulus of the foam that is easy to test and easier to have confidence in the validity of the results. This test was added as a supplementary test to compare with the block compression and dogbone compression results. Most of the stronger denser foams were easier to test, while the more porous synthetic foams were still failing at the specimen ends. However with each type of modulus test, round or rectangular, we were able to determine that the failures of the round specimens were more directed to the center of the foam, while the rectangular ones were more directed to the larges defect at each corner. However this test would probably work best with an aluminum stiffeners added to the ends of the samples, similar to the dogbone compression samples.

In conclusion carbon foam is an extremely tailorable cellular solid with seemingly unlimited potential. Characterization of this material in all aspects of its physical behavior is vital to all applications of this material. The tests that have been afore mentioned are the best attempt of adaptation of currently accepted testing methods and standards. With more continued effort in the pioneering of carbon foam characterization, many industries and military applications will benefit from the various applications this unique form of carbon can potentially offer.

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# A. APPENDIX

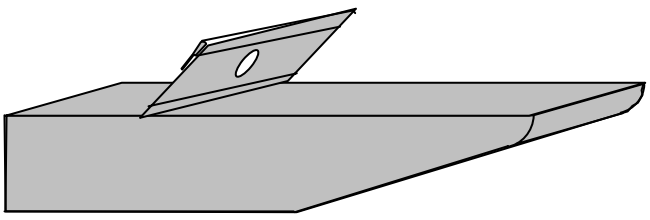
## A.1 ALUMINUM SURFACE PREPARATION

Aluminum is used for various aircraft applications and also in applications dealing with testing of adhesives. Using aerospace epoxy creates an extremely strong bond for the interface between the foam and the aluminum when used as a reinforcing material. Unfortunately the thickness of the bond-line is difficult to control due to the basic shape of the porous foam. Epoxy slowly enters in to the open cells, bonding to the foam ligaments and reinforcing the foam. The introduction of the epoxy into the cellular system of carbon foam creates more sources of error that may affect the validity of the data. As the testing occurs it is difficult to determine if mechanical failure of the specimens is directly related to the material or indirectly related to the epoxy.

Aluminum quickly develops a very thin layer of aluminum oxide on the surface, interacting with the adhesive properties of the Loctite 9394 and 9396 adhesives. The aluminum oxide layer must be removed to ensure complete bonding onto the aluminum. To remove the aluminum oxide layer surface preparation must be performed. Step one in preparing the aluminum for adhesive is to remove any excess adhesive or oils with a mild detergent and scotch bright abrasive pads. The detergent that was used in this case was Alconox detergent. Scrub the surface thoroughly making it appear shiny as opposed to the dull grey of the aluminum oxide layer. Use plenty of water. When complete with cleaning the aluminum, rinse it thoroughly and dry in an oven until the metal is dry. Step two is to meticulously grit-blast each surface that will have adhesive applied onto it. The grit used in this case was 50-micron aluminum oxide particles. The grit blasting process is to make the surface gain some texture to increase the ability of the aluminum to accept the adhesive. Bonding to the aluminum surface must be done within a few hours; otherwise the corrosive layer of aluminum oxide will reappear. The adhesive under load will pull the corrosive layer off the metal and cause the adhesive properties to drop.

## A.2 ADHESIVE APPLICATION ON CARBON FOAMS

Instructions for adhesive mixing may be found on the container. Use a small amount of adhesive to coat the aluminum plates with approximately one tenth of an inch. Mask off areas of foam where adhesive is not intended to enter with flash breaker tape. Be sure to apply a small amount of pressure to help the adhesive squeeze into each material, thus making a secure bond line.



**Shear Plate with Razor Adhesive Application**
**Figure A.2.1**

## A.3 EQUATIONS

### A.3.1 DEFINITIONS

ε → *Experimental Strain* – determined after the data has been recorded and is directly in relation to the extension of the experiment while observing the initial gage length of the specimen prior to testing.

σ → *Stress* – the functional relationship between forces recorded on the specimen and the initial cross sectional area of the specimen.

e → *Extension* – actual displacement of the specimen due to movement of the load frame. Determined experimentally by means of an extensometer being applied to the surface of the lower compression platen.

g.l. → *Gage Length* – Length measurement of the specimen in either the gage length or the entire length of each given specimen.

P → *Force* – Recorded measurement of force from a calibrated load transducer.

A → *Cross-Sectional Area* – the area of the specimen, which directly relates to the cross section in the middle of the gage length.

$E_c$ → *Compressive Modulus* – Young's modulus that is determined only in the linear elastic portion of a stress vs. strain curve, for a sample that is being compressed.

### A.3.2 EQUATIONS

Experimental Strain $\varepsilon = \frac{e}{g.l.}$ (strain)

Stress $\sigma = \frac{P}{A}$ (Psi)

Compressive Modulus $E_c = \frac{\Delta\sigma}{\Delta\varepsilon}$ (Psi)

Shear Modulus $G = \frac{\Delta\sigma}{\Delta\varepsilon}$ (Psi)