ABSTRACT

Tensile Test Method for the Determining of the Structural Properties

of Individual Carbon Fibers

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With the advancement and continuing integration of composite materials and technology in today's modern industries, research in this field is becoming more and more significant. Predicting these composite materials' properties and how they will react under trauma and over time is one of the most critical aspects that researchers are striving to understand and expand knowledge in. Most research exercises plural composite plies to find ways to make these predictions. Others use entire fiber bundles or resin/epoxy blocks to relate their properties to the plies they are used to form. However, what are the properties of the fibers that the fibers in the fiber bundle are composed of, and can it be used to predict the properties accurately while consuming less of the material? This thesis conveys a test method designed to determine mechanical properties and characteristics of specifically carbon fibers, but could be used for other fibers, and for use to design composite materials.

APPROVED BY DIRECTOR OF HONORS THESIS

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DATE:_____

TENSILE TEST METHOD FOR THE DETERMINING OF THE STRUCTURAL PROPERTIES OF INDIVIDUAL CARBON FIBERS

A Thesis presented to the Faculty of

Baylor University

In Partial Fulfillment of the Requirements for the

Honors Program

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by

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Waco, Texas

May 2013

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ACKNOWLEDGMENTS

I am indebted to a lot of people, who directly or indirectly contributed in accomplishing this work and without who's support I likely would not have finished this work. First I thank my advisor Dr. David A. Jack, for his support and encouragement in every phase of this project. I am very fortunate to have had him as my advisor, as is anybody else to have him. I appreciate his patience in teaching me new concepts, improving my analytical skills, and reviewing my writing.

I give thanks to the many members of the SIC'EM group for their support and helpful insights. I'm sorry if I was a burden in any way to you awesome people. I would like to thank Rush Malen for having discussions expanded my comprehension of the theory behind my work and his help is procuring materials. Also, thanks to Sarah Stair for instructions of how to use the SEM and for kind help while trouble shooting problems. I am fortunate to know and work with such brilliant people as those in the SIC'EM research group, who always are willing to help and work around or with each other's schedules. I am really honored to know all of them.

My mother and father receive my deepest gratitude and love for pushing and supporting me to pursue honors program studies at Baylor University.

CHAPTER ONE

Introduction

The continued inquiry and research into the carbon fiber reinforced composites field is significant and becoming more so as "the demand for lightweight, durable composites is on the rise" [7] because of their superior strength to weight ratios. Carbon fiber and composite design and manufacturing technology is increasingly implemented in today's modern industries. Some popular products that have used carbon fiber based technologies and materials since the 1970's are fishing goods such as rods and reels, golfing products like club shafts, faces, and heads, and various sport rackets such as tennis, badminton, and squash rackets. Recently other industries have been starting to incorporate or transition to carbon fiber technologies for bigger products. In the marine industry canoes, masts, cruisers and yachts, and the hull of many ship types are starting to take advantage of properties of carbon fibers as well as the aerospace industries products for military, such as the F-22 Raptor, and commercial aviation with massive transport planes such as the Airbus Superjumbo A350 and the Boeing 787 Dreamliner. As the application of carbon fibers increases and is implemented in larger products the risk of harm to the public increases as well. Material failure may be present in any design, and the uncertainty in design can often be greatly alleviated for materials with well characterized and understood properties, such as the commonly employed steels and aluminums. In the present context, the focus is on developing accurate and repeatable

testing methods to determine fiber and laminate properties and characteristics, understand how this advanced material will behave relative to that of classical materials.

There is a wide range of fiber types available for reinforcement, each with different specific properties. There is often a design choice to be made based on the strength modulus ranging from low such as that found in natural fibers, standard such as that of glass fibers, intermediate as in Kevlar fibers, to high modulus fibers of often indicated by the IM7 carbon fiber. [20] Then there is the choice of tow size which is governed by the number of fibers that make up fiber bundle. The weight of the cloth per yard, which relates to how much material is in the sum of the fiber bundles, is also a design consideration in choosing a fiber type. Fiber bundles can be combined and woven into a wide variety of different cloth patterns which all modify the properties of the end product, a few examples are displayed in Figure 1.

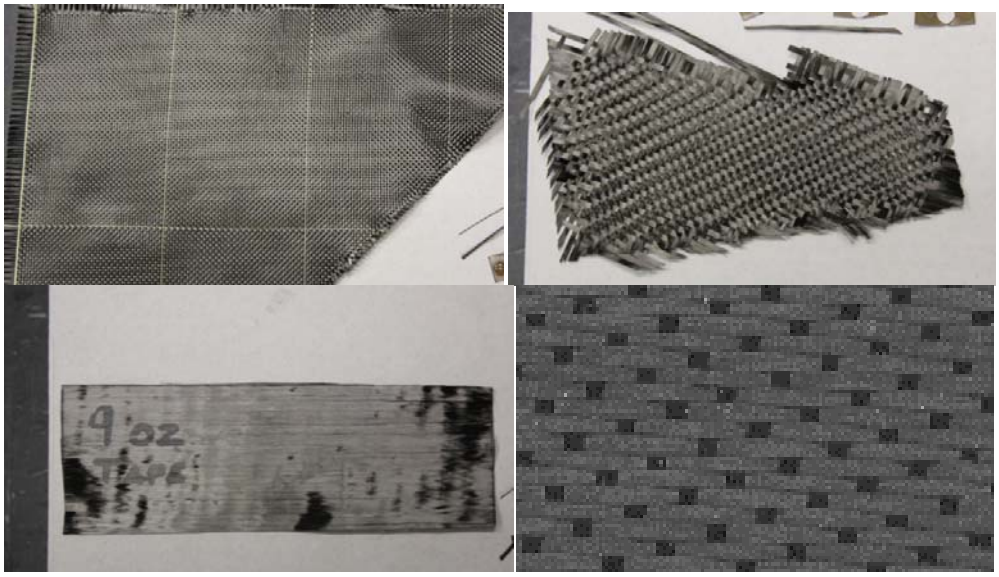


Figure 1. Different Carbon Fiber Weaves

These designations are often given or known by the producer and some, such as the weave and weight per yard, can be easily determined. Often the properties cannot be looked up or different fibers are not available from the manufacturer of the woven fabrics, or for certification purposes a company wants to confirm the properties given by the fiber producer.

Most tests concerning carbon fibers deal with neat bundles, bundles with no matrix, or the woven material after they are infused with a surrounding matrix typically composed of an epoxy or phenolic resin. The resin serves to allow inter-laminar shear, but also degrades the tensile moduli and thus it is difficult to obtain the properties of the neat fiber from the various properties of the overall product. There have even been tests conducted upon carbon composite laminate, and various other carbon fiber reinforced materials such as carbon-fiber overwrap pressure vessels. [7,13,19] However, tests upon individual fibers are not well known, and neither is information on individual fibers at such small scale. These tests would be similar in concept to tests performed on whole fiber bundles, but the gripping of the fiber ends will need to be significantly different. Fiber tests are potentially useful to conserve material and reduce costs for experiments to discover the properties of carbon fibers, but as there is little material in a single fiber bundle relative to a processed part this considerations minimal. The uniqueness of individual fiber testing is that such tests could reveal the response of unique individual fibers and not the behavior of the whole. Thus any fiber-to-fiber variation may become apparent. In addition, having the failure loads of individual fibers and not the failure of the bundle of fibers is critical for understanding the onset of failure and the failure mechanism.

For the work discussed in this thesis, several tests will be presented to determine the properties of individual fibers applicable to specifically carbon fibers, but may potentially be useful for a wide range of fibers such as organic fibers, glass, Kevlar, and many different synthetic fibers. The testing procedure is followed by experimental results for known carbon fibers to test the accuracy and precision, and to identify tribulations inherent in the test method and resolve them. Experimental results will be compared to values from available properties sheets from the fiber manufacturers and characteristics of carbon fibers as described by widely published sources (see [2] & [3]) and analyzed to validate the proposed testing method.

Tests will be performed on each carbon fiber type used in this experiment several times to observe fiber to fiber variations and also to observe if there are limitations in the proposed methodology for consistency. To this end the test method will be applied to different types of carbon fibers of known manufacturing and results will then be shown. also the results will be related to the bundle properties given by the manufacture.

CHAPTER TWO

Carbon Fibers

2.1 History.

"Multiphase materials have been known for millennia, recognition of this novel concept of combining together dissimilar materials during manufacture led to the identification of composites as a new class that was separate from the familiar metal, ceramics, and polymers." [3] Even from the early times, pre AD man made and used composite materials. They made and used straw-reinforced clay for bricks for homes and structures as well as pottery. Now today modern composites have evolved and adapted to use metal, ceramic, or polymer binders as matrices to reinforce a wide variety of fibers and particles of diverse materials from graphite to glass, from inorganic to organic. Just "over the last forty years, composite materials, plastics, and ceramics have been the dominate emerging materials" [2] field. Research into these materials is growing to advance modern technologies to capitalize on their particular properties to maximize efficiency and reduce costs. "For the last 30 years, the use of polymers and polymeric-based composites in industries such as aerospace, automotive, and petroleum has exceeded that of all metals." [7] "A composite is a multiphase material that is artificially made, and the constituent phases must be separated by a distinct interface." [3] So the materials can be similar chemically but have boundaries between the different materials. The primary image of a composite in today's media is the carbon fiber and epoxy, or

resin, matrix. The two materials have very distinguishable and disparate physical, as well as other, properties. Another example of a composite that may easily go unnoticed despite its regular use in many beginning construction projects is plywood. Since plywood is composed of pieces of assorted lumber it combines the differing properties of all the lumbers for its strength, and the different wood pieces can easily be differentiated, and the borders of each piece can be identified as well. The basis of the renewed interest in composite research is that "most physical, chemical, and processing-related properties can be enhanced by a suitable combination of materials." [2] The most commonly imagined composites are those made with strong fibers held together with a binder or matrix, though "particles or flakes are also used as reinforcements" [2] but they are not as effective as fibers.

2.2 Uses of Carbon Fibers

The volume and number of applications of composite materials has grown steadily, penetrating and conquering new markets relentlessly. "Aerospace and transportation industries, in particular, are finding composites useful for their weight reduction potential" [2] to allow their products to "become roomier, more luxurious, and more fuel efficient" [4] such as the products shown infigure2.

Figure 2. Boeing 787 DreamLiner (Left) and Lamborghini Aventador (Right) Use the Same "Advanced Carbon Fiber" (from http://www.imaginelifestyles.com/luxuryliving/2012/03/boeing-inspired-lamborghini-aventador-photo-gallery)

Fiberglass boats made from a polyester resin reinforced with glass fibers and graphite sporting goods number among the products that are taking advantage of these new technologies.

Figure 3. Sporting products employing Carbon Fiber Composite Techonlogy (from http://bangkok-companies.com/categories/thai_companies_p88.htm)

Some people and sources claim "the possible applications of composite materials are limited only by the imagination of the individual." [2] "Multiphase composites provides

exciting opportunities for designing an exceedingly large variety of materials with property combinations that cannot be met by any of the monolithic conventional metal alloy, ceramics, and polymeric materials." [3] The variety of material types provides ample potential for uses of composite materials, and this potential spawns interest in the development of modern composite technology with both modern materials as well as natural materials that are in abundance in the world. Uses for composite materials are constantly "expanding into the infrastructure construction and repair markets" [2] as those industries are keen to take advantage of the qualities of composites such as resistance to environmental degradation. Further the increased complexity of shapes that composites can be molded into increases the value of composite technology as the reduction in "part count and associated costs" [2] serve to compensate for the more expensive materials required for composites.

Figure 4. Complex Composite Structures, Single Part Performing in Place of Multiple Smaller Parts (from http://www.wolfdenproducts.com/)

Composite technology will find ever increasing uses as the need for a "host of high-technology applications such as those found in the aerospace, underwater, bio-

engineering, and transportation industries" [2] search for better materials with properties that will maximize efficiency.

2.3 Modern Tests

There are a great many tests procedures and methods to discover even more numerous and varied properties of materials. In attendance with these modern tests, there are abundant machines to perform them, both machines for specialized tests and machines for general and a wide range of tests. There are "machines to test everything from compression to tension to flexural, peel and cleavage" [12] Many of these tests and machines can be used on composite materials, and some are even designed principally for them. Some tests "expose materials to conditions from cold and damp to hot and dry or hot and wet...and even fixtures for testing the effects of long term exposure to load and elevated temperatures" [12] One test for composite testing is the

> *Hydromat panel test method (ASTM D6416). The Hydromat is a fixture that was invented to test flat panels for flexure. What makes it unique is the ability to deflect the specimen in two dimensions at the same time. Most of the equipment used with the MTS machines will allow multi-cycle fatigue testing as well as one-time static testing of both composite laminates and neat resin samples. [12]*

Another test used to determine properties of composite lamina and resin samples of the" heat distortion under load testing (DTUL) of neat resin samples as well as laminate. "[12] A couple specialized test machines are the "Differential Scanning Calorimeter (DSC) for thermal analysis of polymers and a Fourier Transform Infrared Spectrometer (FTIR) for evaluating polymer compounds and blends."[12]

Two modern, standardized tests from the American Society for Testing and Materials (ASTM) International are very similar to the method I designed. First is the

D3822-07 Standard Test Method for Tensile Properties of Single Textile Fibers and this test measures the " tensile properties of natural and man-made single textile fibers of sufficient length to permit mounting test specimens in a tensile testing machine." [16] While this test can be used to measure the "breaking force and elongation at break of single textile fibers" [16] it is for fibers used for yarn or tow. The second test is C1557-03(2008) Standard Test Method for Tensile Strength and Young's Modulus of Fibers and measures properties of the same kind that interested composite researchers. "This test method covers the preparation, mounting, and testing of single fibers (obtained either from a fiber bundle or a spool) for the determination of tensile strength and Young's modulus at ambient temperature." [17] However this test has a potential to be harmful to the user as it "may involve hazardous materials, operations, and equipment." [17]

2.4 Properties of Carbon Fibers

A composite material is formed by the combination of two or more distinct materials to form a new material with enhanced properties. "Hybrids are composites with more than one type of reinforcement material, an example is concrete further reinforced. Also different materials can be layered to form an enhanced products." [2] Further carbon fibers and other composite materials are classified into three major classifications based the fiber and ply composition of the material:

Classifications:

- *Reinforcement:*
    - *Continuous Long Fibers : Unidirectional, Bidirectional, and random orientation*

    - *Discontinuous Fibers: Random and Preferential orientation*
    - *Particles and Whiskers: Random and Preferential orientation*
- *Laminate Configuration:*
    - *Unidirectional Lamina*
    - *Laminate*
    - *Bulk Composites:*
- *Hybrid Structure:*
    - *Different material in various laminae*
    - *Different reinforcement in a lamina [2]*

One of the possible areas of application for which these composite have been "investigated is the area of electrical and thermal management. Aircraft and satellites often encounter a situation when they have to make a compromise on thermal or electrical management on account of weight or vice versa" [5] The main factors that drive the use of composites are weight reduction, corrosion resistance, and part-count reduction. other advantages that motivate some applications include electromagnetic transparency, wear resistance, enhanced fatigue life, thermal/acoustical insulation, low thermal expansion, low or high thermal conductivity, and many others." [2] Property combinations are designed by well thought-out arrangement of two more distinct materials, as properties compromises are weighed and balanced against each other. "Composites are stiffer and stronger than most convention materials on a per unit weight basis." [2] Carbon fibers and composites have higher strength and stiffness to weight ratios than most materials due to their usually low densities. During design the composite material's properties reduce from the fiber properties as the amount of matrix material used increases, this reduction proportional to the fraction amount of the matrix to the net material used since the properties of the matrix are generally lower than the properties of the fiber. [2]

CHAPTER THREE

Test Method

For this test method the objective is to determine mechanical properties of individual carbon fibers via a tensile force. Properties such as modulus of elasticity or ultimate strength, yield strength are useful to determine characteristics and materials. properties are calculated with a combination of dimensional and applied values. For example, in designing fiber/resin composites the modulus value of the fiber is crucial as it is used to calculate the modulus of a composite in the direction of the fibers in this equation found in Barbero's Introduction to Composite Materials Design.

$$E_f * V_f + E_m * V_m = E_c \quad [2]$$

This equation shows the Modulus of the composite, E_c, is calculated using the modulus of the fiber, E_f, and resin, E_m. See Barbero for more equations used to determine other properties.

Tensile tests apply forces directly the material sample, usually by using clamps to securely grip two opposite ends of the sample and then pulling the ends away from each other. As the force is slowly increased the stress on the material slowly increases and the sample elongates until it reaches its maximum strain and the material breaks. Similarly the DMA-Q800 also uses clamps in its tensile test assembly to secure and apply forces to samples. and this works well for samples with dimensions up millimeters for the size of its ends. Carbon fibers' diameters are on the scale of micrometers. Since a single fiber is so small it would be difficult to secure the clamps enough so the fiber will not slip; in

addition to other concerns such as stress concentration where the clamp pinches the fiber. Another concern due to the miniscule size of fibers is keeping the fiber fixed during various stages such as measuring its dimensions or when securing it with the clamps. Therefore, this method would require something to keep the fiber fixed before the test stage but would not affect the test stage. It was settled on to make a fixture to connect the carbon fiber to that would hold the fiber firm for measurements and allow it to be easily loaded into the DMA-Q800, but could be easily separated into two parts so only the fiber would be affected by the test.

Another concern was measuring the dimensions of the carbon fibers. The dimensions of the length of the fiber and its cross sectional area via its diameter are needed to compute values such as stress and strain. Since the fibers are so small and flexible they are very difficult to see and more so measure. So some mechanical assistance was required to find the dimensions of the sample.

Next, for the test itself the sample would need to loaded into the tensile test assembly straight and allowed to reach its full length before the test was performed. The sample would need to start unstressed and the stress slowly increased as the test continued until the sample reached ultimate strength and failed. The final set up of the fiber in the DMA-Q800 will appear like Figure 5.

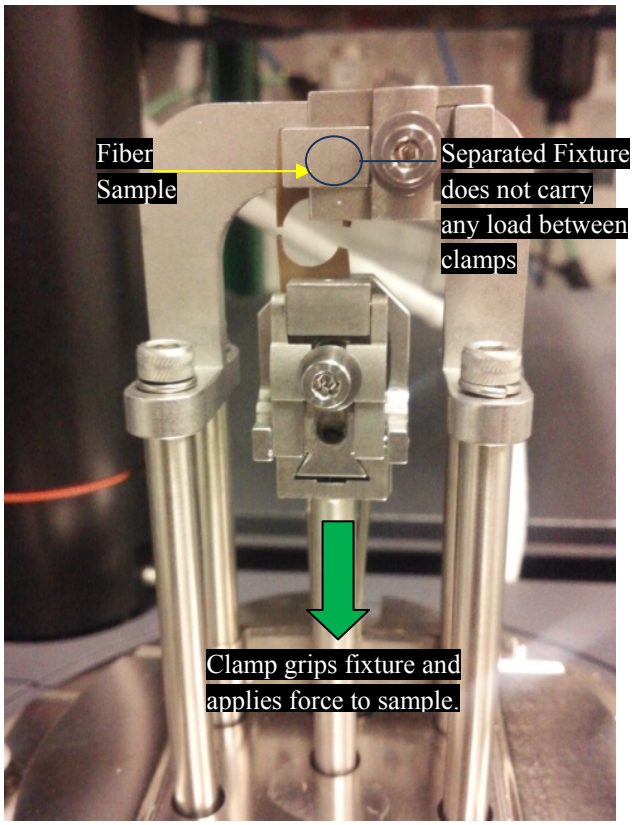


Figure 5. Test Overview

3.1 Sample Preparation

3.1.1 – Sample Components

Before any tests can be performed samples must be prepared. Samples are comprised of three key components: a fixture, an adhesive, and a carbon fiber, as illustrated in Figure 6.

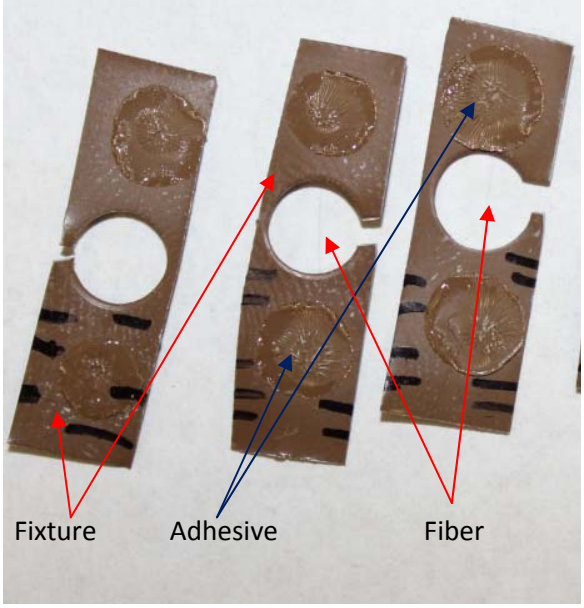


Figure 6. Completed Samples

The samples' critical component is t-he carbon fiber which can be arbitrarily chosen from known carbon fiber types or from unknown carbon fibers. The fiber which must be kept straight before and when the it is loaded into the DMA-Q800. The sample must be easy to move and load in the DMA-Q800. The sample must also prevent or reduce pretest stress or strain on its critical component to test improve accuracy by reducing indiscriminate effects which could affect the behavior of the carbon fiber.

3.1.2 – Initial Method for Creating Samples and Shortcomings

The first batch of samples used fixtures constructed from twelve-hundredth of an inch thick steel sheet metal. This material was very strong and tough, and those properties worked superbly to prevent pretest stress and strain on the carbon fiber while transporting the sample and loading the sample into the clamps of the DMA-Q800. However, once the sample was loaded in the DMA-Q800 and the tensile test attachment clamps secured onto

it, the spacer material started to provide a critical problem. When the sides of the sample spacer where separated using a tin snip, the sample experienced a shock stress and the fiber would snap. This shock stress occurred for every sample, regardless if the DMA-Q800 clamp position was locked or not, and ruined the entire batch of samples since no tests could be run. The next batch, Batch 4, of samples was made the same way but one side of the steel fixture was separated before the carbon fiber was attached. Concurrently Batch 5 was prepared in the same manner as Batch 4 except for one detail. For Batch 5's fixtures' material a tenth inch aluminum sheet metal was used. One side of the aluminum fixtures were separated before attaching the fiber like Batch 4. This adjustment to the sample preparation method reduced the shock stress of the samples when loaded into the DMA-Q800 and the second and final side was separated. While some carbon fibers of samples of Batch 4 remained intact the amount was still far too small. Batch 5 had better results, more samples' fibers remained intact after loading and the fixture was separated. Half the samples still were ruined when the tin snip was used. The number of ruined samples stilled need reduction. So for the next batch of samples the fixture construction was adjusted again. For Batch 6 and on when the fixtures were being hole punched the hole was punched as close to one side of the center of the fixture as possible while leaving the side intact. Then the other, thicker, side was trimmed using metal cutter snips while leaving the side intact. This adjustment resulted in a significant increase of tests being run for the reason that less samples were ruined when the fixture side was separated.

Figure 7. Fixtures of Samples Broken Before Testing

A superfluous note for Batch 6 was the presence of a phenomenon to the effect of samples' fibers disappearing while separating the sample fixture but before the fixture was fully separated.

For the second component of the samples, adhesive, various brands of superglue were used. There was no noticeable difference in sample performance or durability between the difference super glues. This is beneficial as it shows that the tests are relatively unaffected by the choice of superglue, only affecting the carbon fibers and that each adhesive equally performs the task of holding the fibers in place well.

Difficulties with the third component, carbon fiber, primarily dealt with isolating the single carbon fiber from a fiber bundle. Often a fiber would not fray further and appear as a single fiber, but would be actually be two when observed or measured later in the process.

3.1.3 – Current Sample Creation Method

Figure 8. A Batch of Completed Samples

The sample preparation begins with the fixture. First tenth inch thick aluminum sheet metal is used as the material for the fixture. Secondly, A shear press cutter is used to cut a strip about half an inch wide off one edge of the aluminum sheet metal plate. Then a metal cutting snip is used to divide the strip into parts of about 1 inch length; the shear cutter could also be used for this but is not as easy. For the next step a hole cutter was used to punch a hole in the middle of each sheet metal piece and as close to the edge as possible without completely separating the edge.

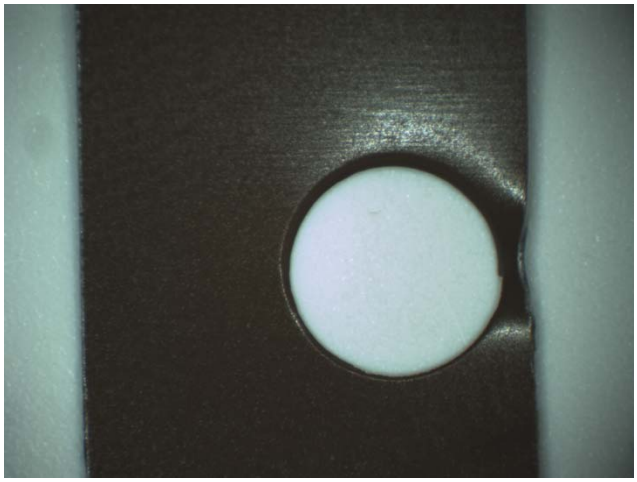


Figure 9. Sheet Metal Piece With Hole

Next the metal cutting snip is used to trim the sheet metal piece's thick edge as thin as possible without separating the spacer's edge. If one of the edges has been separated while making the fixture by this time, then care must taken to prevent the other edge from getting separated as well. If both edges are intact then a pair of tin snips are used to separate the thicker edge.

Figure 10. Fixture With One Edge Separated

The spacer is now ready for the attachment of the fibers

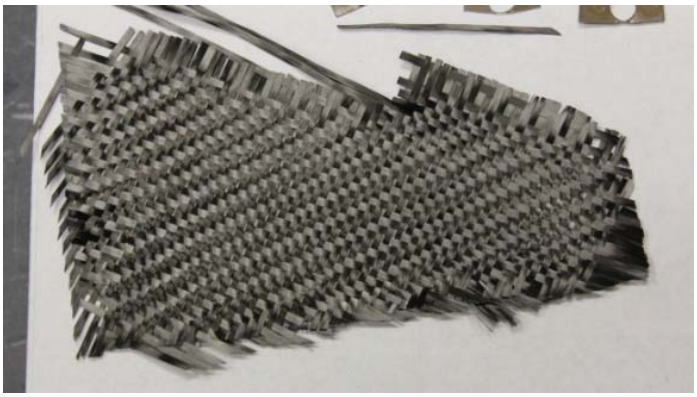


Figure 11. Carbon Fiber Weave

Select the fiber one wants to test and single out a fiber bundle from your supply of material. Next, separate the individual fibers from the fiber bundles by teasing the fiber bundle at one end until it frays, and a frayed strand of fiber can be gripped with tweezers and pulled away from the rest of the fiber bundle. Figure 12 illustrates how fraying of a bundle appears.

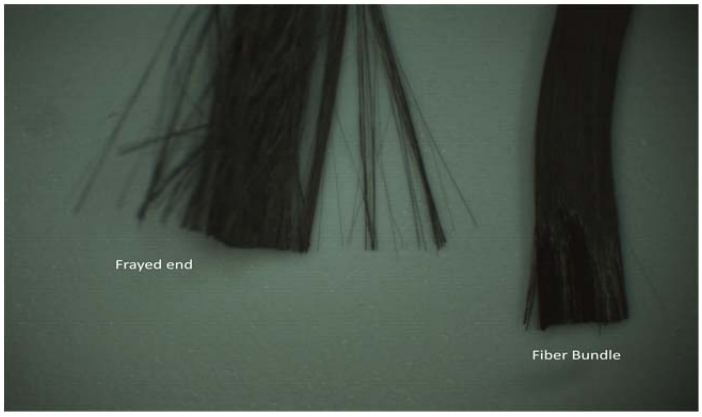


Figure 12. Fiber Bundle, Frayed and Whole

Repeat this process to separate fibers from the fiber and so on until no more fraying occurs and the fiber appears to be isolated. Then grip the fiber with tweezers or forceps

and carefully lay the fiber across the sample fixture so it traverses the hole in the center, parallel to the thin edges and extends both sides. When the fiber is in position, adhesive is applied over the portion of the fiber over the sample fixture to attach it to the fixture, and then the sample is left to dry. Figure 13 shows a batch of samples freshly completed and left to let the adhesive dry. Excess fiber that extends past the sides of the fixture are sniped with the tin snips.

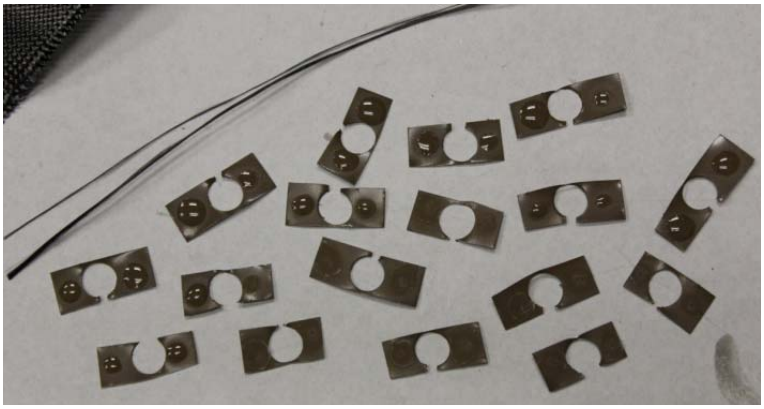


Figure 13. Batch of Samples Left to Dry

3.1.4 –Sample Adhesive Connections, Inspected

The connection between the carbon fiber and the fixture created by the adhesive should be given some consideration. This connection is crucial for reasons pertaining to loading the sample for testing and a good adhesive-fiber bond is essential during testing. A good connection requires the adhesive to bond flush with the fiber and as well as the fixture so the area the bonding strength applies to is a greater and any of stresses that apply across the bond are more evenly dispersed. A flush connection also helps to prohibit stress concentration anywhere along the adhesive-fiber bond. Another quality of

the adhesive connection also serves to prevent stress concentration is for the carbon fiber to lay straight within the connection. Keeping the fiber straight helps preserve the uniform distribution of stress from the test as well as reduce the stress on the bond to the adhesive.

Figure 14 and Figure 15 show the connection between the super glue adhesive used to attach the fiber to the fixture. Figure 14 shows images of a sample after being tested, acquired using the Summit OptixCam. The left image shows the fiber extends all the way through the super glue. The image on the right is zoomed on the inner edge of the super glue dot, and shows a little better the connection and shows that the fiber is broken. This is a significant display of the soundness of the connection made by the bond of the adhesive. It show that the super glue held the fiber and fixture securely, and further that the fiber-adhesive bond did not fail or let the fiber slip free the connection during the test.

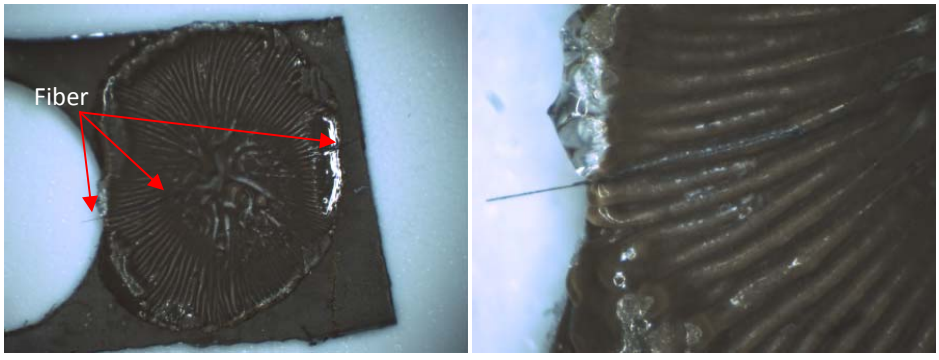


Figure 14. Fiber Seen Under Adhesive

Figure 15 shows images of two different samples obtained using the SEM. It shows the smooth transition into the super glue of the fiber and the adhesive's flush connection with the fiber and fixture.-

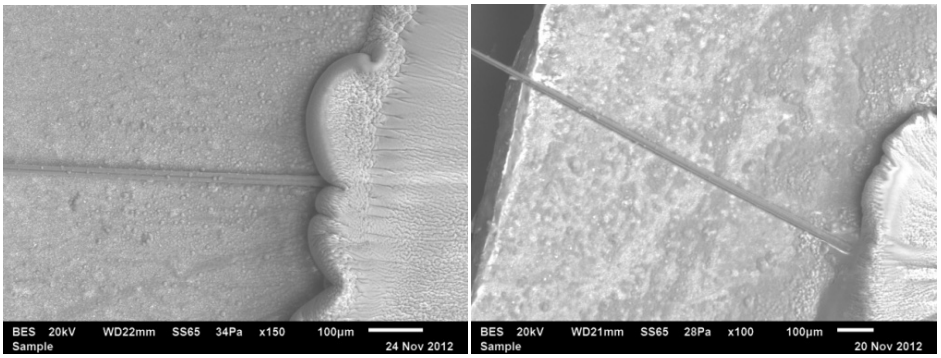


Figure 15. SEM Image of Fiber and Adhesive

3.2 Sample Measurements

3.2.1 – Objective of Measuring

The second stage of the process is to record the dimensions of diameter and effective length of each of the samples. The length of the sample's carbon fiber from one super glue dot to the other is utilized in calculating the strain percentage and it is the only part of the fiber that should experience elongation during the test. Any length under the glue dot will be supported by the glue in addition to being outside the inner part of the clamps which apply the force to the sample. This measurement is used to in the following equation from Callister.

$$\varepsilon = \frac{\Delta l}{l_0} \quad [3]$$

were ε is the value of strain of the fiber, Δl is the displacement of the fiber due to elongation caused by the applied stress, and l_o is the effective length or original, unstressed length of the fiber.

The second measurement that is required is the diameter of the sample's carbon fiber. The diameter is used to find the axial cross sectional area of the fiber which is then used to calculate, stress as given by Callister.

$$\sigma = \frac{F}{A} \text{ [3]}$$

Where σ is stress, F is the force applied to the sample, and A is the axial cross sectional area of the sample as calculated using the diameter, assuming the fiber is circular.

$$A = \frac{\pi}{4} * D^2$$

The sample's diameter can also be used to determine if there is there only a single carbon fiber in the sample, or if multiple fibers are present. From an analytical viewpoint it can be seen that error in the diameter measurement will propagate more than the length and have a greater influence the results. This is major reason why measuring fiber dimensions, especially the fiber diameter, as accurately as possible is critical.

3.2.2 – Optical Measurements

3.2.2.1 –Initial Optical Shortcomings in Measurements. This part of the test process had several stages of evolution. In general, for the diameter of the sample were placed under a microscope and had pictures taken of the fiber over the hole in the spacer at identical magnifications. Then the pixel diameter length of a straight line spanning the fiber was recorded. Then a picture was taken of a calibration slide under the same conditions and magnification was taken, and used to make a pixel to length unit converting factor. The initial stage used a Summit OptixCam attached to a 2X

ScienceScope M27-CP-20200M (OH3) scope with an additional 0.5X focus scope to take pictures of the samples and a calibration slide.

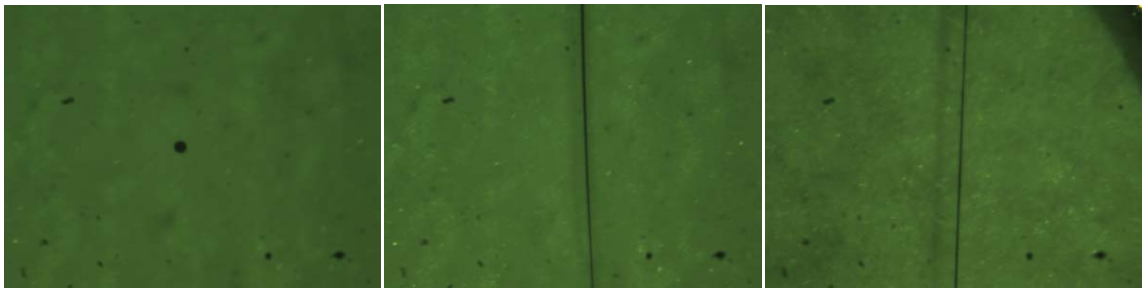


Figure 16. Images From the Summit OptixCam. .007mm Dot(left) and Two Fibers

As evident in figure 16 there was a coloration predicament which just added difficulty to determining fiber edges. Further at higher foci and zoom magnifications the fiber images would become very dark and edges would be blurry with shades. The coloration negatively contributed to this symptom as well. To try to clear of the images an additional ScienceScope IL-FOI-150 plus light source was used when taking images. While the additional light did reduce the magnitude of the darkness, the images of the fibers remained dark and blurry at higher focus and zoom. Further the coloration persevered in more or less evident forms with the adjustment of lighting, as seen below in Figure 17.

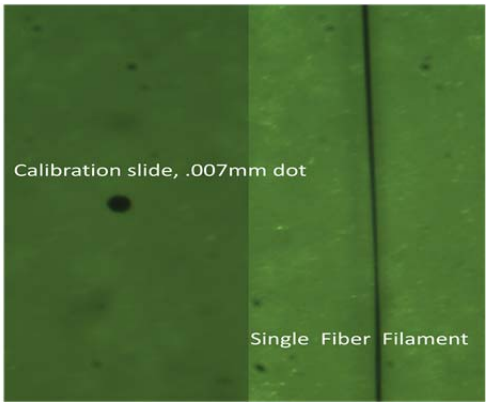


Figure 17. OptixCam Images with Addtional Light Sources

3.2.2.2 –Image Quality Criteria: Establishment and Measurement. The criteria used to determine the quality of a photo taken with the Summit OptixCam is very general and only easily determined during the measurement stage when determining the fibers' diameters.

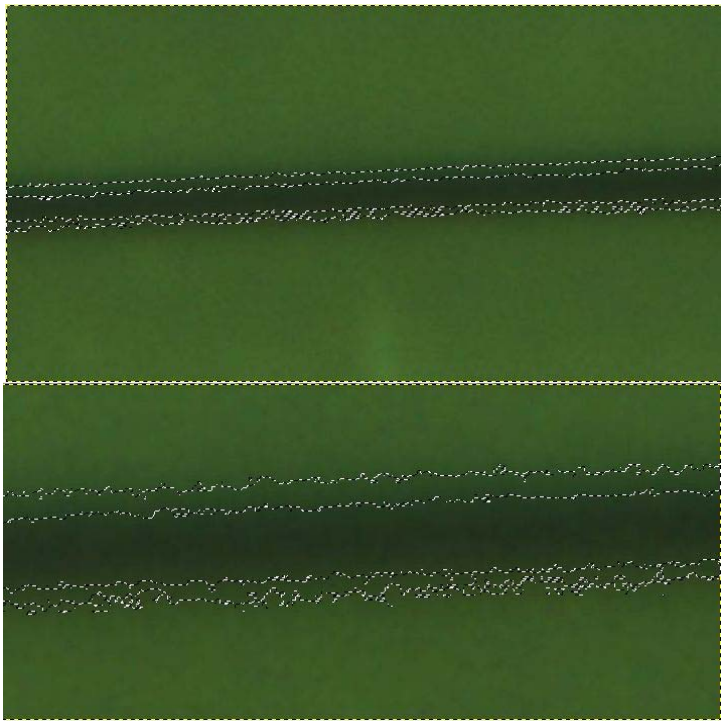


Figure 18. Unclear Fiber Edges

First a good picture has high clarity quality, which is a product of the camera resolution, how well the microscope is focused, and illumination. The above photos in Figure 18 are of Sample 3 of Batch 4 in GIMP, a software similar to Photoshop. Both images in Figure 18 are the same image but the top image is at 200% zoom and the bottom at 400%, and both photos are cropped to just the fiber. The presence of a fiber is evident in both but the images not qualify as the sort of good images that is needed. The edge of the fiber is unclear, there is a bright greenish area of the background, and a black area of the fiber, and transitioning between them is a region of shifting shades. Further the fiber itself is only identifiable as a black, linear area. The is no detail of what the fiber actually appears like, if it is twisted, if there are impurities such as fissures or lumps of extra material present, or if there are multiple fibers still stuck together. As this region is not clear nor is the transition the image does not have the high quality required for a good image.

3.2.2.3 – Issues With Quantifying the Edge of the Fiber. Due to the issues with that disqualified the images taken with the Summit OptixCam as good images, taking accurate measurements was very difficult. As shown in Figure 18 the borders are not linear. The images above have been modified in GIMP, to highlight a particular color or shade throughout the image, to show the edges of the different regions. This illustrates that the transition from the dark of the fiber to the brightness of the background is not smooth, and the displacement between the regions is nowhere near uniform. The highlighted areas form four more or less parallel paths which act as the rough borders of the indistinct region between what is clearly the fiber and what is clearly the background. This region is the primary obstacle with quantifying the diameter of the carbon fiber. It

acts akin to a fog, concealing an edge from being unmistakably identified. For diameter measurements taken from these images for the samples of the first batches the distance from the inner borders of the transition regions were used as the measurement of the fiber diameter. However the precision and accuracy for these measurements remained below desired standards.

3.2.3 – SEM Measurements

3.2.3.1 – General Operation of and Rational to use the Scanning Electron Microscope:

To improve diameter measurement accuracy and precision this section of the fiber test method was modified to utilize the JEOL JSM-6610LV Scanning Electron Microscope (SEM) to capture images of the carbon fibers on the samples and determine diameter measurements from those images, such as seen in Figure 19.

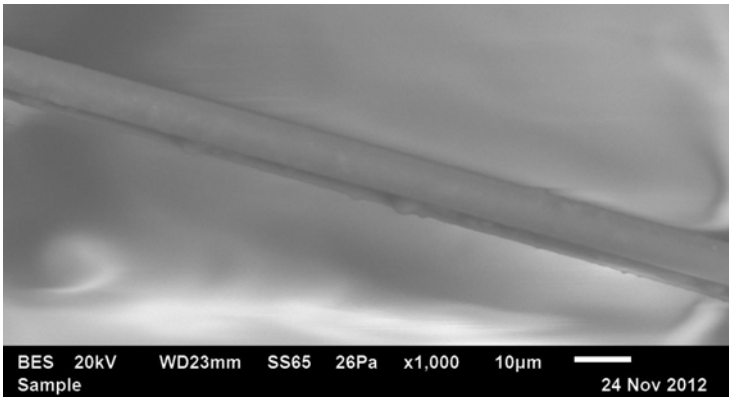


Figure 19. SEM Image of Sample Fiber

The SEM was chosen because the SEM has a much greater magnification power potential than the microscope, over 100,000x. Also the SEM functions by using electrons and a sensor rather than by light so additional illumination would not be required like with the

OptixCam. The SEM had several different resolution settings, four in all, all but the one of which surpassed the OptixCam's resolution. In supplementary fashion, the SEM also has better positioning control for the sample and image shifting.

SEMs in general have a much greater magnification power than microscopes to apply to objects being studied. Usually SEMs have magnification powers upwards of 300,000x. Further

> *SEMs also have tremendous depth of field compared to traditional microscopes, providing an almost 3-D image for researchers to analyze, as compared to the flatter image an optical microscope produces. These advanced microscopes can look past the surface of an object, telling researchers information about its composition... SEMs have their share of drawbacks as well, like cost. Even the cheapest among them cost tens of thousands of dollars. They're also bulky and complex instruments, requiring considerable expertise to operate. As a result, their use is typically limited to research and industrial applications. [8]*

The SEM is made up of two critical systems, the detector main body and the amplifier. The detector body has three main parts including the incident electron beam, the detector elements of the SEM and fixation metals. Likewise, the amplifier includes the cable and flange, preamplifier, circuit board, and display section. In general the SEM used utilizes two detectors when performing the backscatter electron scan; one on the SEM chamber wall and the other on the SEM objective lens. The two elements are semiconductors and their signals from detected electron are combined and refined into the image displayed by the SEM, even though the detectors detect the backscattered electrons independently of each other. [9]

The SEM constructs images by scanning surface of the sample or object of interest with an incident electron beam it emits from its objective lens. Then specimen

emits backscattered electrons "carry topographic, physical, and chemical properties of the specimen" [9] which are then detected the SEM's semiconductor detectors. From the semiconductor detectors the varying input is converted into an electric signal which is sent to the preamplifier and from there is enhanced and sent to the operational amplifier unit. From there, "the operational amplifier unit further amplifies the signals and performs arithmetic computation" [9] Finally the resulting signal of the computations becomes the signal from which the SEM image is structured from and the image is displayed.

3.2.3.2 – Measuring Diameter Using the SEM. When using the SEM to measure the fiber diameter dimension, the test method altered slightly. To determine the diameter of the sample's fiber in the SEM more steps must be taken, as well as more care not to damage the SEM. The procedure starts with selecting a 32mm diameter SEM specimen holder and cutting a strip of double sided adhesive copper tape and placing it on the SEM specimen holder. Then the sample is positioned near the center of the SEM specimen holder on the copper tape. Next the SEM is vented to room atmosphere before opening the SEM chamber. Then use the SEM specimen exchange tool to grip the 32mm diameter SEM specimen holder and securely place the SEM specimen holder on the SEM platform inside the SEM's chamber. Once this holder is securely placed, close the SEM and evacuate the chamber to low vacuum, about 40psi. When the chamber is under vacuum the IR camera set into the SEM is turned on and used to observe inside the SEM as the SEM platform and specimen holder are slowly raised to 20-25mm from the SEM objective lens to prevent the SEM specimen holder or sample from contacting the SEM

objective lens and detector. When the SEM platform is in position the IR camera is turned off and then the SEM incident electron beam settings are adjusted and turned on. Then the coarsest resolution setting is selected on the SEM, and auto focus and auto contrast are selected. When the SEM is focused and contrasted then the SEM specimen holder is moved via adjusting the position of the platform until the sample is found. Then a location of the fiber in the center of the sample is centered in the SEM display screen. Next the scan setting is increased by one stage and the SEM is refocused using manual controls. The SEM picture is re-centered if necessary. This process is continued until the finest resolution setting is selected and focused. Then an image is captured of the fiber. From this image the diameter of the fiber is found by using the scale included in the image. After the SEM picture is taken the SEM detector, objective lens and electron beam are turned off and the SEM platform is returned to its original position and a distance of 80mm from the SEM objective lens. The SEM is then vented to room atmosphere, opened, and the SEM specimen holder removed with the specimen exchange tool. Tweezers are used to remove the sample from the copper tape and specimen holder.

3.2.3.3 – Early Issues With Electron Buildup. While using the SEM to measure diameters a strange occurrence was encountered multiple times, concerning electron build up. Before this occurrences could be noticed the proper procedure for operating the SEM as well as the procedure to get samples' fiber diameters. This is a quick walk through of these steps taken to investigate this occurrence and the conclusion established from it. First, the standard procedure to obtain sample fiber measurements was followed to the stage where the sample was centered in the sensor image, as follows. Copper tape

was placed on the 32mm specimen holder, then a sample was positioned on the tape using the forceps. Then the specimen exchange tool was used to insert the specimen holder with sample into SEM and secure it to the SEM platform inside. Following this, the chamber was evacuated to low vacuum, and the platform raised to 20mm below the SEM objective lens.

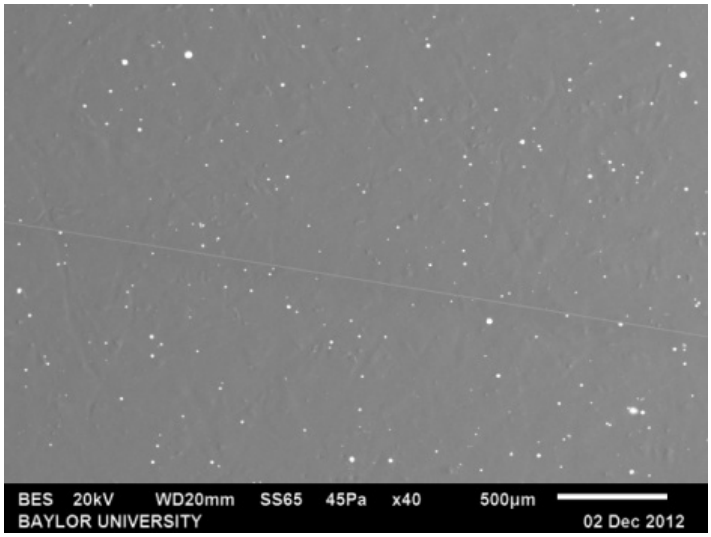


Figure 20. Image of Sample at Low Resolution

Magnification was increased in by small increments, refocusing on the fiber at each step. After 150X magnification the autofocus software in the SEM would not properly focus the SEM's produced image. At this step the image is manually focused via the control panel. Some stage between 300-450X the SEM was unable to be focused to an acceptable standard.

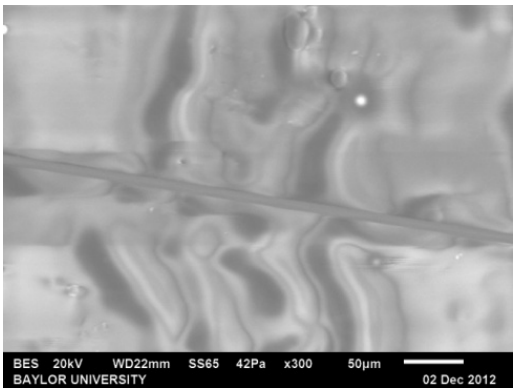


Figure 21. Unfocusable Image of Sample at 300X

Then magnification was reduced and a impression was noticeable that was not there when beginning.

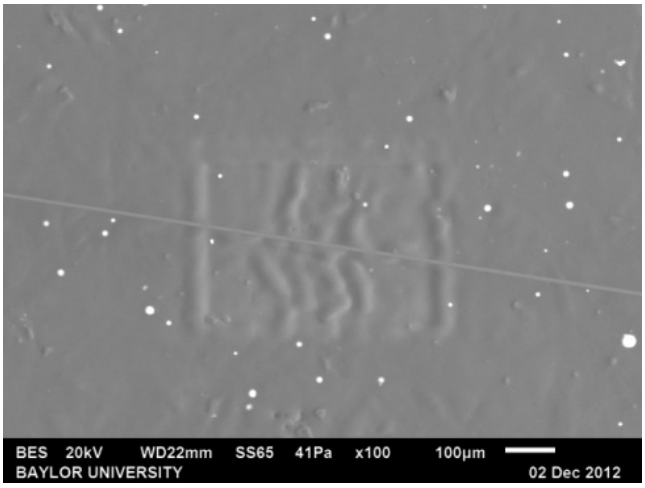


Figure 22. Mysterious Mark

Initially thoughts were that the tip of the SEM objective lens had contacted the sample. If the objective lens had contacted anything then it would potentially have caused major damage to the SEM, since the SEM objective lens is very delicate. However the platform was set at a height of 20mm from the SEM tip, and given a sample height of

3mm so the SEM safety protocols would not allow the specimen holder or sample to make contact with the SEM lens or detector.

Figure 23. Specimen Holder and Objective Lens Positions

To check the SEM platform positioning the SEM detector, objective lens and electron beam were turned off, and the Infrared (IR) camera installed in the SEM turned on. The IR camera was used to check the vertical position of the SEM platform and specimen holder and its relation to the SEM objective lens. As the image above, in Figure 23, shows the SEM specimen holder was not in physical contact with the SEM objective lens, in addition to confirming the height the SEM platform was set to was a safe distance away from the SEM objective lens. Then the IR camera was shut off and the SEM detector, objective lens and incident electron beam turned back on, and the magnification decreased to continue the investigation into this occurrence at another location of the sample. When the magnification was decreased another mark was noticed on the copper tape and the sample's fiber in addition to the first mark, shown below in Figure 24.

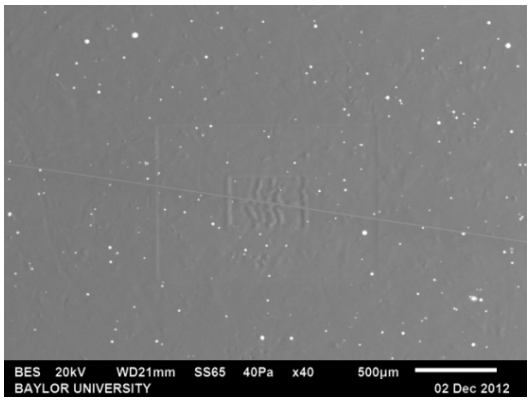


Figure 24. Another Bigger Mark

For the next step of the investigation the procedure was repeated at a second location closer to the inside edge of the spacer but still with only fiber and copper tape in the area of focus. This area was chosen to check the reproducibility of the marking occurrence. Again, magnification was increased in small increments, focusing on the fiber at each step. Eventually the SEM was un able to be focused at 500x magnification. For a second time, magnification was reduced to 150x in two separate stages and after each occasion a new impression was noticed that was not present before. Figure 25, Figure 26, and Figure 27 show the stages.

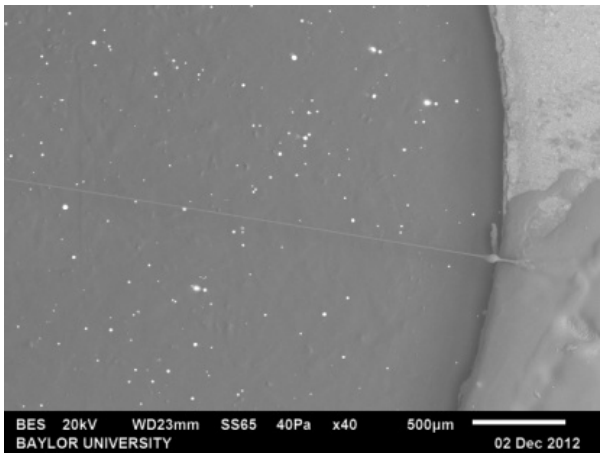


Figure 25. Unmarked Location, Fiber/Tape

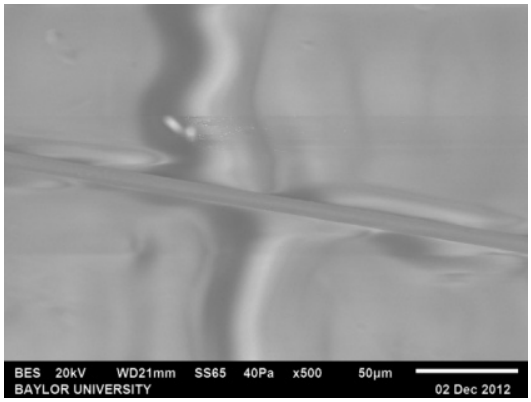


Figure 26. 500X Unfocused, Fiber/Tape

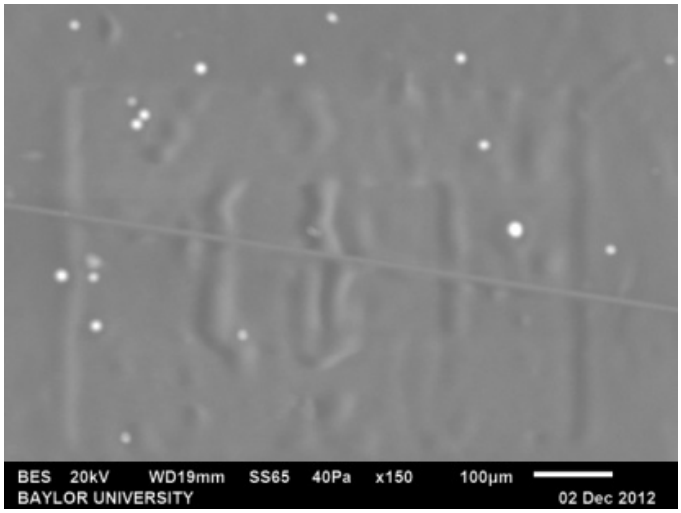


Figure 27. Marks on Location, Fiber/Tape

Next the same procedure was repeated on a clean section of the sample fixture. An area of the fixture where there was is no fiber or adhesive was present was selected. This would serve to isolate any occurrences or affects to just the material of the fixture, without additional effects or influence from the factors or other materials such as the fiber. The steps were repeated as before: Increased magnification by small increments, focusing the SEM at each step, this time on the aluminum fixture. The SEM magnification was brought to a halt at 500x magnification to remain as consistent with

the procedure used at the previous location as possible. Next magnification was reduced in two separate stages, to 150x power magnification. At each stage there was no identifiable impression present, as Figure 28,Figure 29, and Figure 30 provides evidence of.

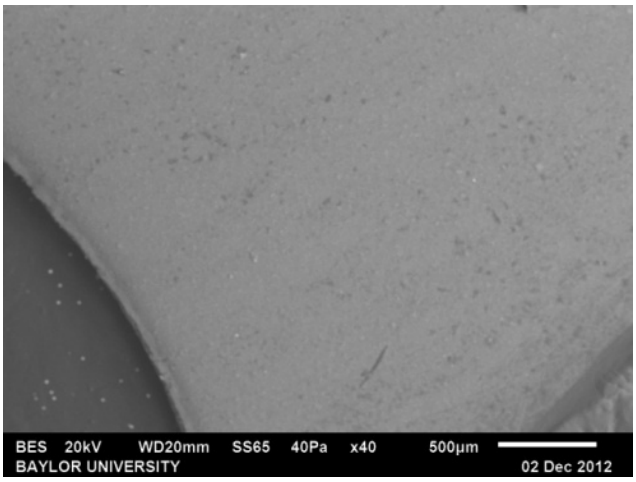


Figure 28. Unmarked Location, Fixture

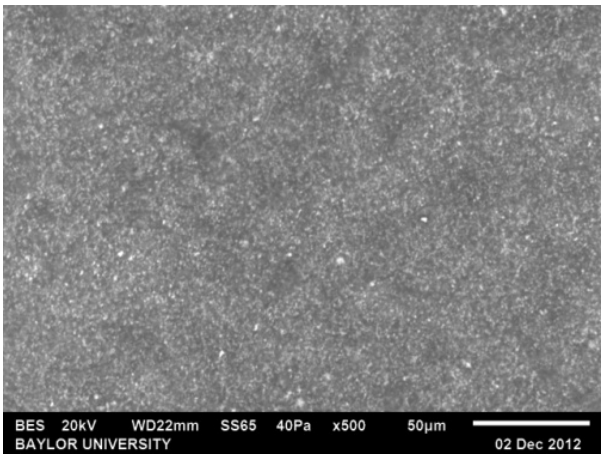


Figure 29. 500X, Fixture

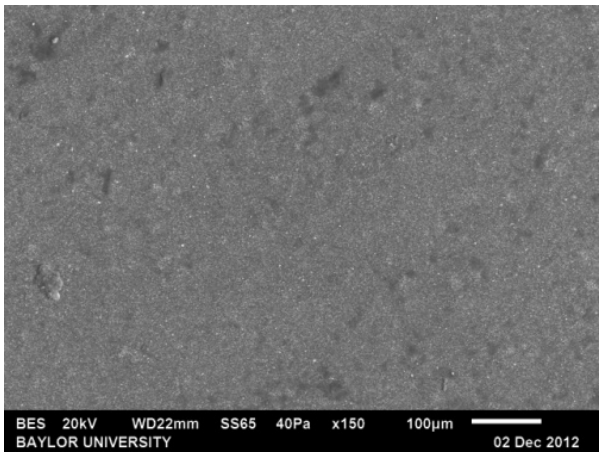


Figure 30. No Marks on Location, Fixture

For the next step of the investigation an area of the superglue adhesive was chosen to see if the SEM would leave a mark. The area of the sample fixture completely was completely covered by the adhesive, but not directly in the center of the super glue dot. As the area would be completely covered it could be considered to solely adhesive with no effect from the fixture. Further the area chosen was off center to avoid any potential influences from fiber the adhesive was securing to the fixture. After the area was centered in the SEM detector, the steps as before were repeated: increased SEM magnification on the adhesive area by small increments, focusing the SEM at each step. Stopped increasing the SEM magnification at 500x magnification for consistency of the process used. Again, reduced the magnification to 150x power magnification in two stages, leaving the SEM focused the intermediate focus stage for an appropriate amount of time. Again, at each stage there was no identifiable impression present, evidenced by Figure 31 and Figure 32

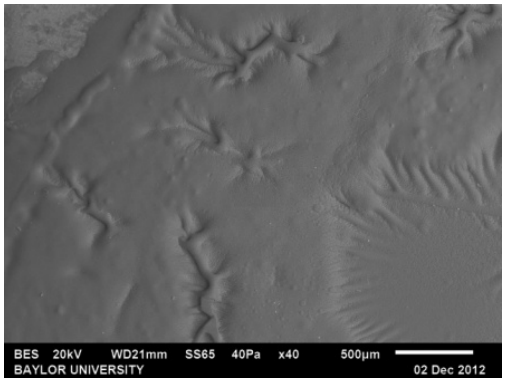


Figure 31. Unmarked Location, Adhesive

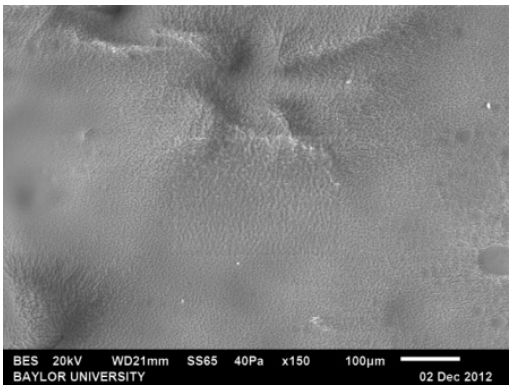


Figure 32. No Marks on Location, Adhesive

Next the process used on the previous locations was applied on a section of only the copper tape used to hold the sample in position. A plain, clean area of the copper tape where there was is no fiber or spacer nearby was chosen to focus on to continue the investigation. This area would serve to isolate the copper tape and effects it would experience from any potential influences from the carbon fiber or the aluminum fixture. Repeat the steps as before: increased magnification, refocus the SEM at each step to the power of 500x magnification, etc. consistent with process used at the previous locations. At 500x magnification the SEM detector image was unable to be focused. Remaining

consistent, the magnification was reduced to 150x power. At each stage there was an identifiable impression or mark present, as Figure 34 provides evidence of.

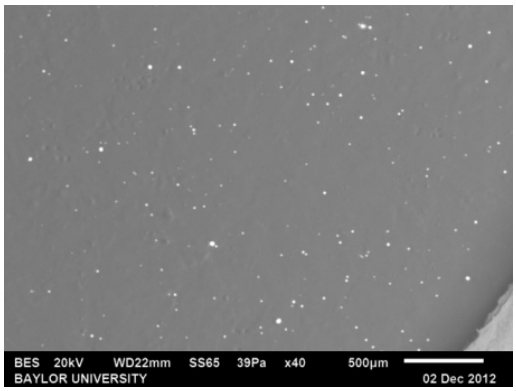


Figure 33. Unmarked Location, Tape

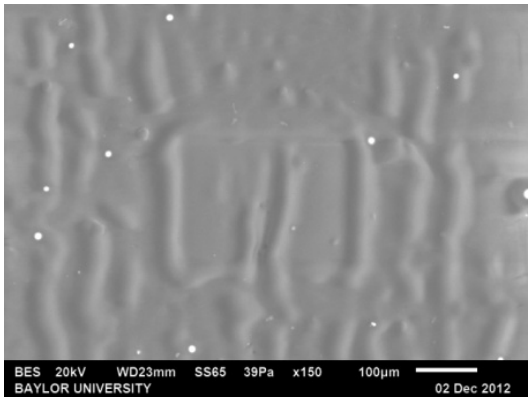


Figure 34. Marks on Location, Tape

For the final stage of the investigation into this occurrence the process was repeated at the area where the sample's carbon fiber traverses the fixture-copper tape boundary. This location was chosen to combine the different elements that were isolated in the previous locations and observe what would occur. The procedure was performed as before. However since there were multiple elements in this area it was decided to focus the SEM on the fiber since it is the most critical component for this test method. At this

stage, shown in Figure 36, the fiber, aluminum fixture, and edge of the adhesive dot are in focus, but the copper tape is not. Then magnification was reduced in a consistent manner as before to 150x power magnification. Shown in Figure 37 there are distinct impressions in the area of focus but the impressions are only manifested in the area of the copper tape.

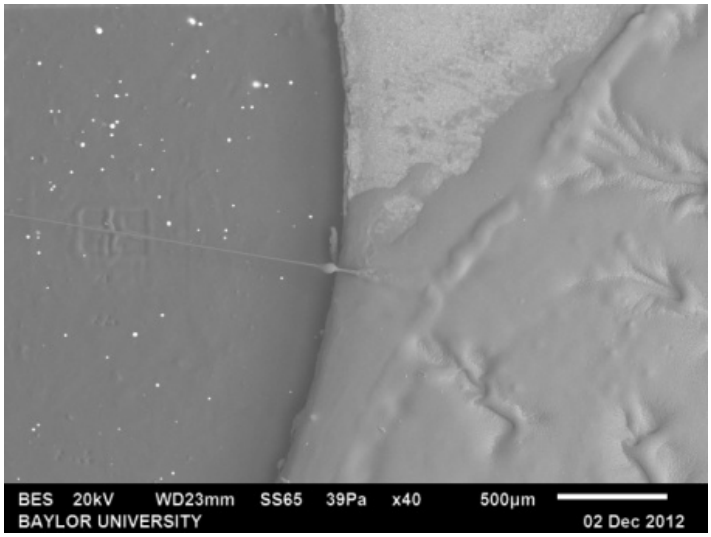


Figure 35. Unmarked Location, Combination

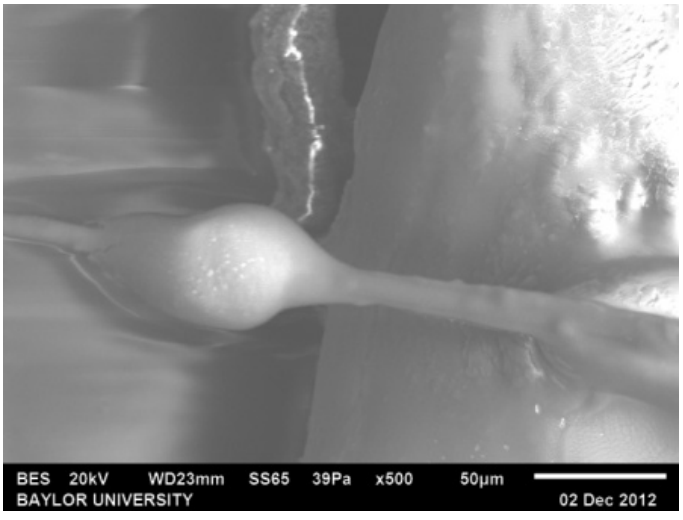


Figure 36. 500X, Combination

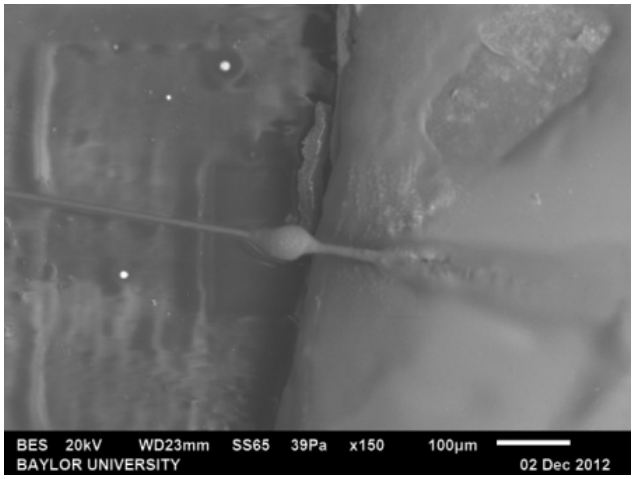


Figure 37. Marks on Location, Combination

From this investigation it was determined that the tip of the SEM objective lens does not come into contact with the sample or the 32mm specimen holder. This is significant as it determines that the SEM safety protocols are functioning and the SEM detector and objective lens are not in danger of being damaged during this carbon fiber test method. Further evidence from the images reveals that the strange marks only occur on the copper tape. This should be due to ductility of the copper tape and the concentration of the electron beam due to the magnification and intensity of the beam emitted by the SEM objective lens. If left long enough the electrons will depress the copper tape leaving an permanent impression in a shape similar to the SEM emitter. Since the sample is unaffected by the electron beam the test method is unimpaired and the primary concern of this investigation in regard to the test method is alleviated.

3.2.3.4 – Presence of Multiple Fibers Determined. Through use of the SEM and the superior quality of the images of the samples obtained from it revealed the presence of multiple fibers on some the samples as the following images show. The presence of

two fibers was the most common occurrence when there were multiple fibers, but the presence of up to four fibers were seen. Further sometimes the fibers would twist around one another, which would require a new location to be found where the fibers could be distinctly identified and measured.

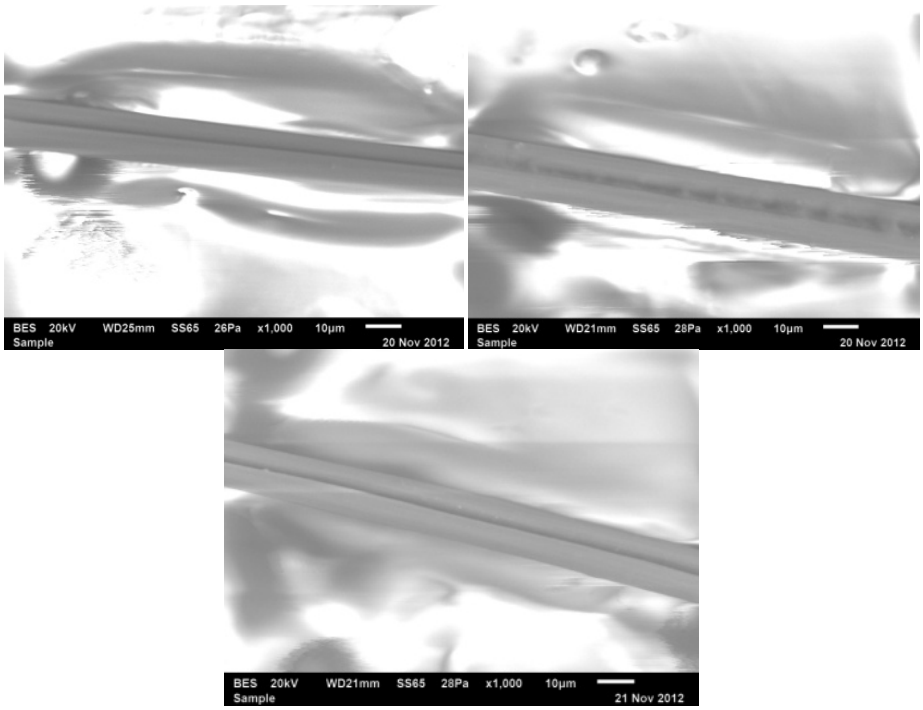


Figure 38. Two Fibers Samples

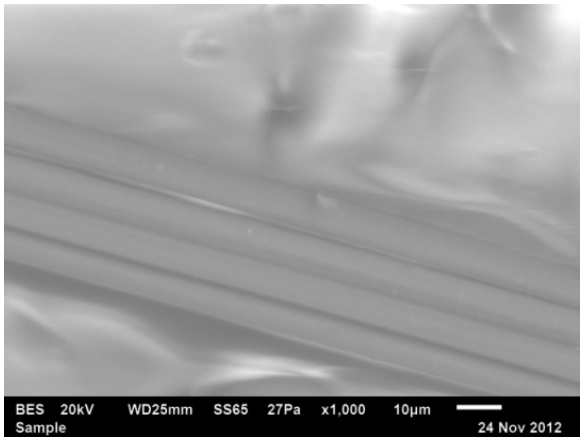


Figure 39. Four Fiber Sample

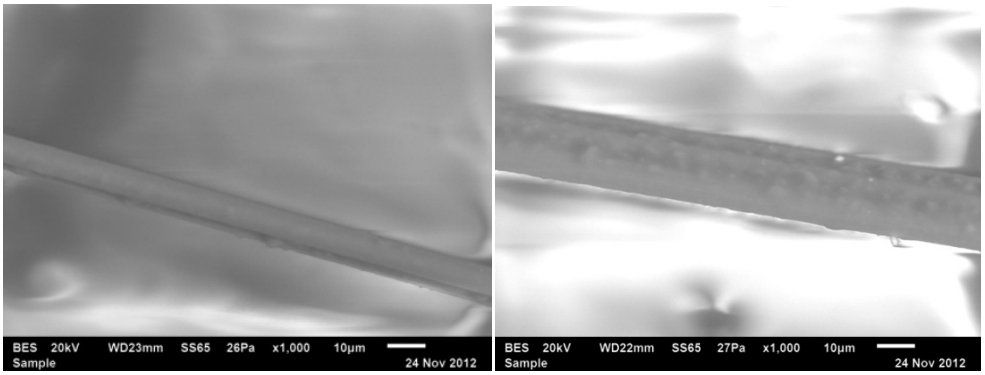


Figure 40. Twisted and Undistinguishable Samples

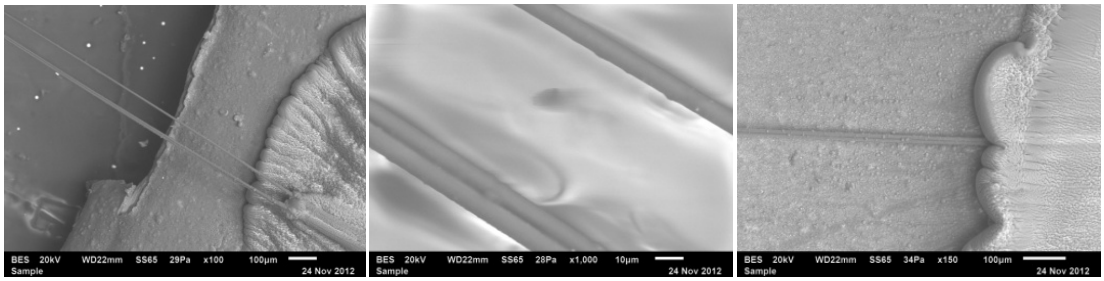


Figure 41. Three Fiber Sample That Twists

To conserve supplies, the samples with multiple fibers where tested the same as the rest of the samples, but diameters for each fiber are measured and the cross sectional calculated for each. Then the sum of the areas are added together to find a net area and this net area used to determine stress. Though, the stress has to be computed in a secondary program; I employed Matlab.

3.2.4 Physical Measurements

3.2.4.1 - Caliper Measurement Technique. For the length of the sample's carbon fiber a dial caliper was used to measure the length of the fibers from where they exited

the inside edges of the super glue dots on the sample. While this did not give the actual length of the fiber in the sample, it did give the effective length that would be subject to strain. Any length under the super glue dot would be prevented from elongation. Since the adhesive bonds to the fixture and the fiber, they would all extend the same length. It is possible the glue's bond could fail, but if it did the bond between the super glue and the fiber would fail first due to the smaller surface area between them. This would affect the results, but would also show when the data is analyzed.

This technique was very unreliable and uncertain. Calipers are adept at measuring physical dimensions such as the thickness of a plate, the inner diameter of a circular hole, or the diameter of a drill bit. All these allow the user to place the teeth of the caliper's grips on the object being measured and apply some force to guarantee accurate measurements. However no such practices could be applied to find the fiber length. There was not any edge present for the grips to press together on in the inside of the glue dots. Further, risk to the fiber was present for any practice that involved the caliper's grips in proximity to the fiber. Secondly, while the inner diameter of the sample spacers were sturdy enough to be accurately measured with the caliper, it would not give the effective fiber length. For the reason that the super glue dots were not always on the edge of the hole of the spacer, as shown in Figure 42 the carbon fiber effective length is greater than the hole in the spacer since the super glue dots are displaced away from the hole.

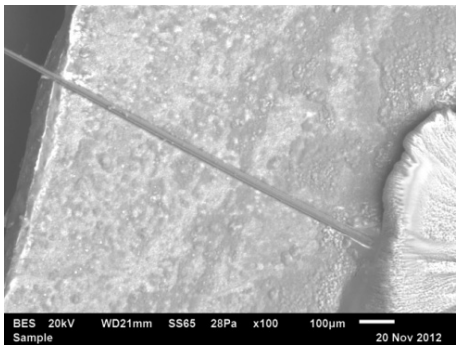


Figure 42. Additional Effective Length of Fiber

3.2.4.2 DMA-Q800 Measurement Technique. To increase the accuracy of the effective length of the fiber I adjusted the test method to integrate the DMA control software to employ it in measuring the length. Technically this stage occurs simultaneously with loading the sample into the DMA-Q800 in the third stage of the test method, but it is included here as it relates to the measurements of the sample dimensions. First the mobile DMA-Q800 arm with the tensile clamp is raised until it comes into flush contact with the immobile tensile clamp attachment, as shown below in Figure 43. Then the arm position is locked. Next the position measurement reading from the DMA control software is recorded as the local origin or first position. Once this is recorded, the DMA-Q800 mobile arm's position is unlocked and the sample is loaded according to the test method as described below. When the sample is loaded and secured in the DMA-Q800 the arm is position locked again. Then the sample's spacer's side is separated with the tin snips and the DMA-Q800 arm is allowed to float so it extends the fiber to its full effective length and then the position is relocked, as also shown in Figure 43. Afterwards the new position measurement is recorded as the second position. This new position measurement is used to find the effective length by computing its displacement from the first position measurement. This displacement, computed as the

absolute value of the difference of the position measurements, is a more accurate effective length measure of the sample's fiber since the sample is secured at the inner edges of the super glue dots. This is done for each sample, so each sample has its own effective length measurement.

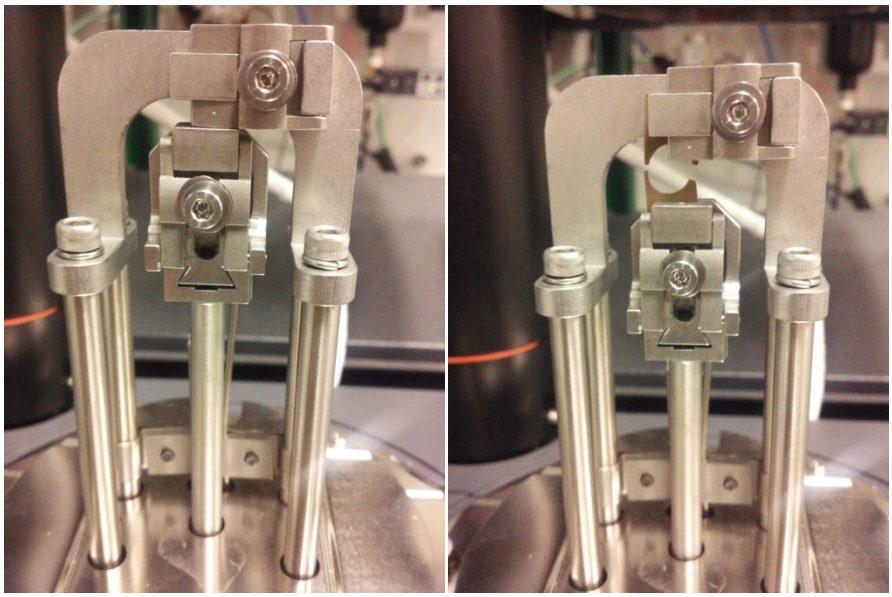


Figure 43. Change in Position of Clamp as Effective Length. Position 1 (left) and Position 2 (right)

3.3 Test Setup and Implementation

For the third stage and final stage of the test method for the fiber specimen requires the items of the DMA-Q800 tensile test clamp assembly to be installed on the DMA-Q800. Start with opening the DMA-Q800 control software followed by installing the tensile test assembly. The later step of the procedure is completed simply by uninstalling any assembly or attachments currently on the DMA-Q800 and replacing it with the tensile test clamp assembly. For more specific details or instructions, see the

DMA-Q800 instruction manual and supplements for the pertinent items. After the DMA-Q800 tensile test clamp assembly is installed it must be calibrated. Calibration must be performed anytime the tensile test clamp assembly is reinstalled on the DMA-Q800. Calibration comprises of three stages which are arranged by and performed easily due to the DMA-Q800 control software, and use some materials in the tensile test clamp assembly box specific for calibration. When the DMA-Q800 is calibrated for the tensile test clamp assembly and the calibration materials are returned to the tensile test assembly box the test method may proceed to the next step.

The sample is loaded into the DMA-Q800 and the process in 3.2.3.2 DMA-Q800 Measurement Technique section above is followed while loading the sample to acquire its length measurement. First the empty mobile tensile clamp is positioned in contact with the immobile clamp and locked in position using the DMA control software, and the position measurement recorded from the control software's display. Next the mobile clamp arm's position is unlocked and moved to load the sample. To load the sample into the DMA-Q800 tensile test clamp assembly the clamps must be loosed using the appropriately sized Allan wrench supplied in the tensile test assembly box. Next the sample is carefully held vertically between one of the clamps and the clamp is tighten so its inside edge secures the sample on the inside edge of the sample's super glue dot and its fiber. Whether the immobile or the mobile arm clamp is secured first does not matter or affect the test method, so either approach is acceptable. Then the DMA-Q800's mobile arm clamp is repositioned while the second clamp is tightened using the Allan wrench so it secures the sample at the inside edge of the other dot of super glue and the fiber. The DMA-Q800's mobile arm is position locked again. Then the sample's fixture's side is

slowly and carefully separated horizontally with the tin snips to reduce shock stress that transpires when the spacer is separated. For the next step, the DMA-Q800 arm position is unlocked and slowly allowed to float down until it stops. This straightens the sample's fiber so it extends to its full effective length and doubles as a check to tell if the fiber broke when the sample fixture's edge was separated. When the sample is fully extended the DMA-Q800 arm is position locked again.

Now the DMA-Q800 control software is used to set up and run the tensile test. First, DMA controlled force is selected for the mode of operation, then for clamp set choose fiber tension. This informs the DMA-Q800 control software that the specimen will have cylindrical dimensions which match the physical characteristics of carbon fibers. This also displays two input boxes labeled length and diameter with units. The sample's measurements found in 3.2 Sample Measurements stage for fiber effective length and diameter are used here for their appropriate input boxes. Next for the procedure pick force-ramp, and this decides on and sets up the steps for a standard procedure for the experiment, and still allows the parameters of the experiment to be adjusted. The operations tab is selected to adjust the parameters of the experiment procedure. The operation parameters for the experiment procedure for this test method are set as follows: temperature for the test is set at room temperature which was estimated to be 25 degrees Celsius, then when that temperature is reached it is set to rest at that temperature for a span of 5 minutes to allow the specimen and clamp temperatures equilibrate to the set temperature as well as let the fiber sit at its effective length and show if its failing before force is applied, then initial applied force is set to 0 Newton and the force ramp is set to increase the force at a constant rate of 0.01 Newton per minute up

to 1 Newton or until the sample's fiber sample fails and breaks. Now select the comment tab and name the sample and leave a comment to describe the set up and anything diverging form the test method with respect to the sample. Return to the first tab and input a file name and a location for the DMA-Q800 control software to make and name a file to save the data streams from the test in. Next click the apply button near the bottom of the DMA-Q800 software window to apply the inputs and parameters to the experiment run. Double check all the inputs and parameters on all the tabs in the DMA-Q800 display to make sure they are correct. If any inputs are not correct, then fix them and click the apply button and repeat until everything is correct and accurate. When everything is right and ready, close the DMA-Q800 furnace chamber. After it is closed click the go button to start the experiment run. Repeat this third stage for all samples to be tested with this test method.

CHAPTER FOUR

Data Analysis

Expect for sample 1 of batch 5, for which the force ramp of 0.001 Newton per minute was unintentionally used force was applied at the same rate to each sample. There are only two completed tests with samples using steel as the fixture material, and both are in batch 6, which was the last batch of samples to use the steel sheet metal. This shows that the changes to sample fixture were positive, but also that the fixture material was a primary issue, but also limits any analysis to see if fixture material affects the carbon fibers during testing.

4.1 First Complete Tests

The first complete tests occurred during Run 3 of testing, with four samples of batch 5. These samples used the aluminum fixtures and the Summit OptixCam for diameter and the DMA-Q800 software for length of the sample. Figure 44 shows the raw data force against displacement.

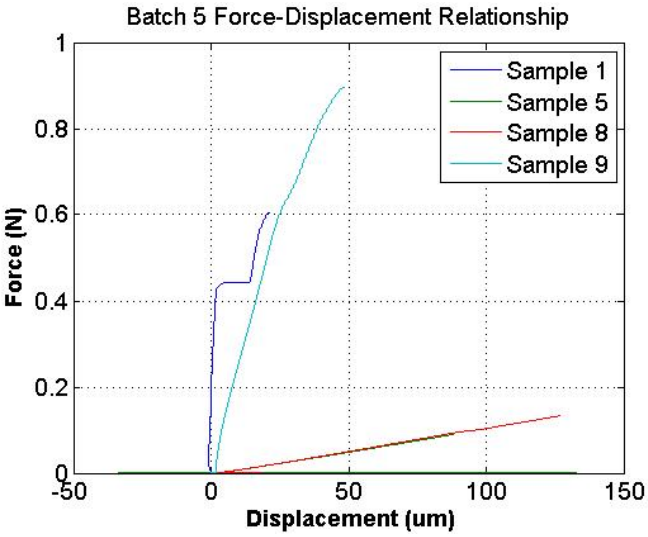


Figure 44. Raw Data of First Complete Tests

This data is then used to calculate stress and strain for the sample which are then used to find the modulus of elasticity. Sample 1 is noteworthy in that it experienced a sudden increase in displacement at a little over four-tenths of a Newton. It is unlikely that the fiber sample rapidly stretched, but probably that the bond between the fiber and the adhesive connecting it to the fixture failed letting the fiber slide. The fiber did not slide completely free since it resumes a slower increase in displacement and most likely was experiencing friction between the fiber and adhesive as it was pulled out. Since the adhesive failed for that sample and it can be assertively conjectured that the adhesive connection was not secure and the cause for the character of its displacement. Sample 9 experienced a displacement with similar character to sample 1, and can be claimed to also experience a failure of the adhesive with the same assuredness as done with sample 1. Samples 5 and 8 display data very similar to each other and which display properties similar to what are expected for carbon fibers, such as linear strains.

From the raw data stress and strain are evaluated using equations $\sigma = \frac{F}{A}$, where σ is stress, F is the force applied, and A is the cross sectional area of the sample the force is applied to, and $\varepsilon = \frac{\Delta l}{l_0}$, where ε is strain Δl is the elongation or displacement of the fiber's end, and l_0 is the original, effective length of the sample. [3]

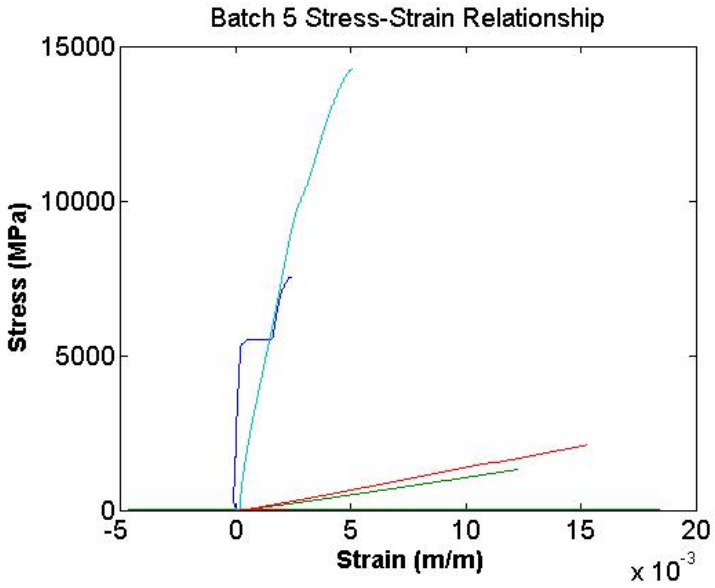


Figure 45. Stress-Strain Graph of Batch 5

Figure 45 displays the relationship between the stress and strain of Batch 5. This value of the slope of this graphs is value of the modulus of elasticity of the material, as the equation for the modulus is stated in Callister's is $E = \frac{\sigma}{\varepsilon}$ Where E is the modulus of elasticity, σ is stress, and ε is strain. [3]

Sample	1	5	8	9
Max Stress (MPa)	7.53E3	1.31E3	2.10E3	1.42E4
Max Strain (m/m)	0.0024	0.0122	0.0153	.0051
Elastic Modulus (MPa)	3.16E6	1.07E5	1.38E5	2.82E6

This table show the max stress, max strain and modulus properties of the fiber samples. It can be seen that the properties of samples 5 and 8 are much more similar to each other than those of samples 1 and 9. More data would be required to determine what the modulus is but it can be estimated that it is in the area of 110 GPa.

4.2 Test Results

Following these first few successful samples of batch 5, later sample batches continued to display improvement in the form of more samples making it to the testing stage and providing results. I will omit data from batch 6, since only two samples were still intact when the test was started. Batch 6 used steel fixtures and only serves to demonstrate the improvement to the test method by changing the fixture material to aluminum. It is worth to note that the diameters of the samples from batch 7 where found using the Summit OptixCam to take images, with such problems as discussed above in section *3.2.2 Optical Measurements*. The SEM was employed to replace the Summit starting after batch 7; and it was used to measure diameters of samples from batch 8 and 9.

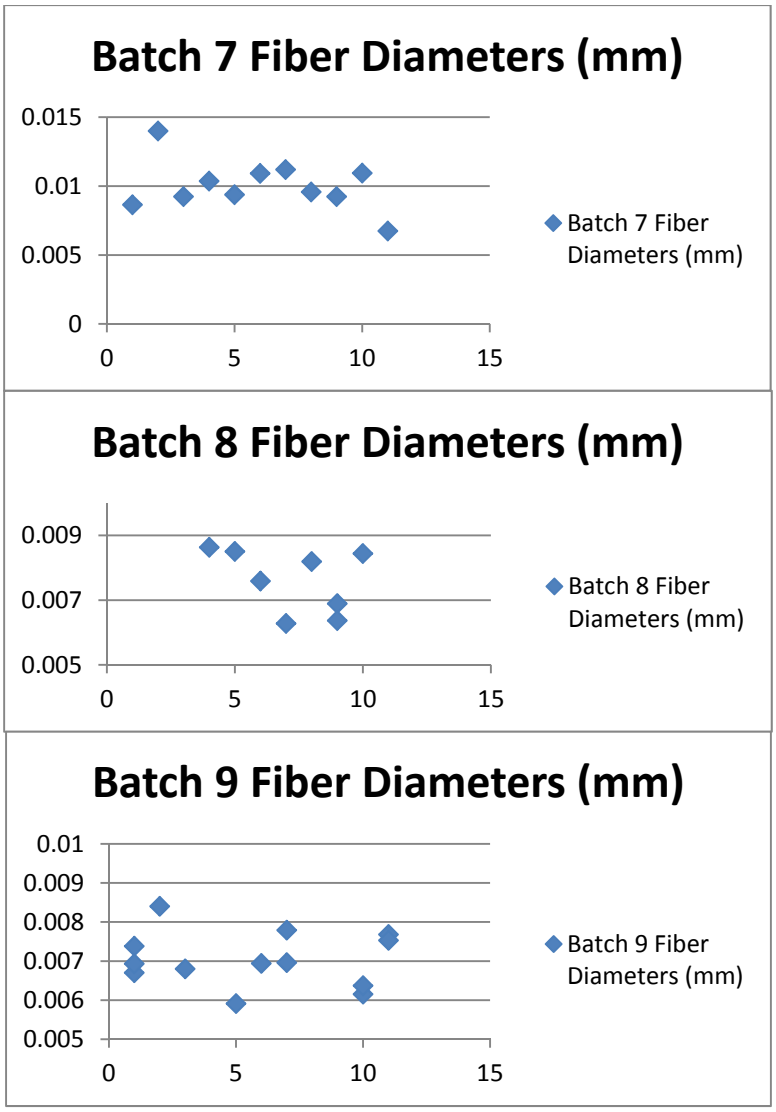


Figure 46. Fiber Diameters

Figure 46, above, shows the measured diameters for each successful sample against the sample number of the batch. These graphs show the improvement in accuracy given by the SEM over the Summit OptixCam. It can be seen that the diameters of from batch 7 are larger on average than those of the other two batches. It has values close to ten micrometers where batch 8's values are closer to seven and a half micrometers and batch 9 around seven micrometers. Further there are samples in the later batches with

plural diameter values because it could be seen with the SEM that there were more than a single fiber present. There is only one value per sample in batch 7 which could be due to the presence of only one fiber or more likely the low quality of the images produced by the OptixCam. The diameters of batches 8 and 9 are very close to the diameters given by the manufactures for their fibers, and there values fall in the middle of the range of values seen.

Visual analysis of raw data from batches 7, 8, and 9, displayed below in Figure 47, based on information from batch 5 reveal the presence of a number of tests where the super glue adhesive failed again.

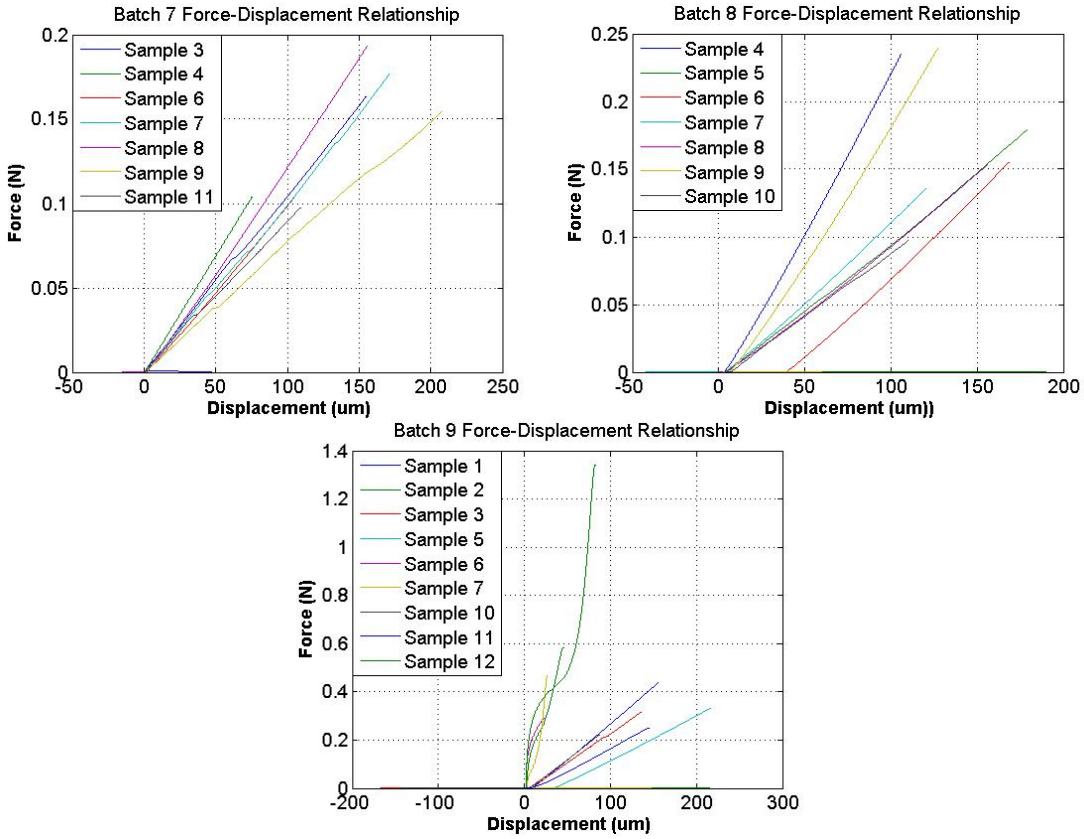


Figure 47. Batch 7-9 Raw Data

Sample 8 of batch 8 display starts with an initial displacement. This probably means that the step after loading the sample into the DMA-Q800, when the lower clamp is allowed to float to let the fiber straighten, was skipped. This did not adversely affect the test since it exhibits characteristics similar to other samples. The raw data also shows that the fibers who adhesive did not fail, generally failed with a range of two tenths of Newton.

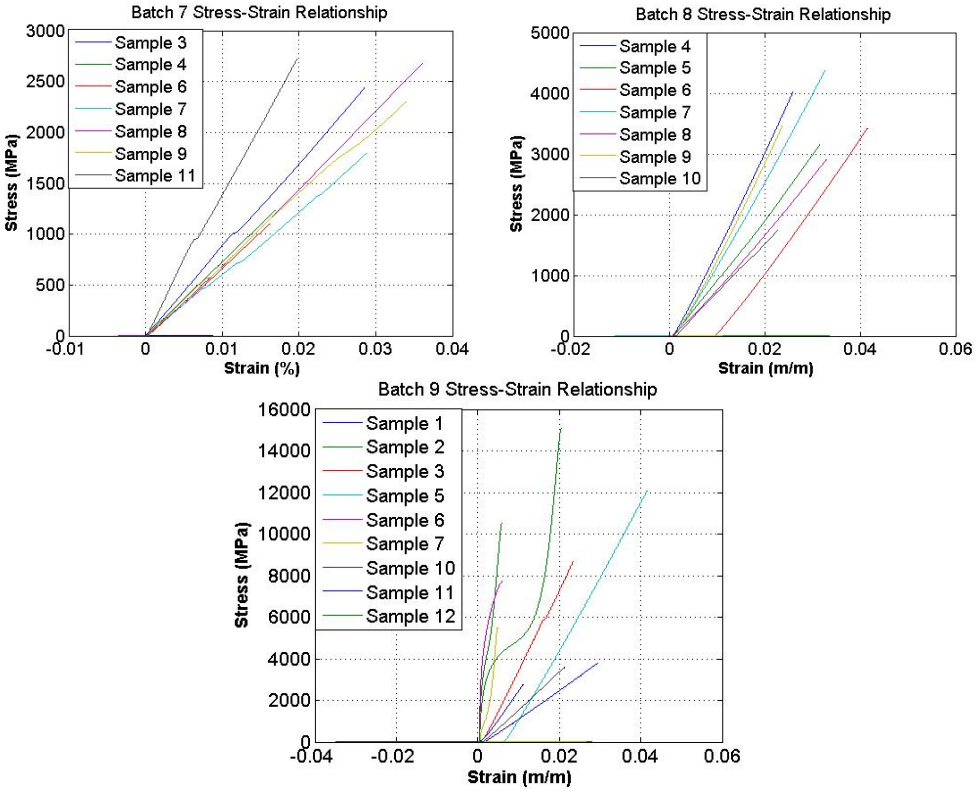


Figure 48. Calculated Data

Figure 48 presents the data from Figure 47 after it has computed into stress and strain values. Ignoring samples 2,6,7, and 12 from batch 9 which were designated above as having a major error, these figures show that the majority of samples reached max strain and failed before four percent strain. This informs that fiber samples contain a

characteristic of brittleness. The table below shows the moduli of elasticity, in MPa, for each successful test, and corrected for any initial displacements, in numerical order.

Batch 7	85399	72804	68006	62215	74240	67753	138040
Batch 8	155400	100370	100150	129330	88150	149400	81143
Batch 9	131790	369930	318400	170260	247190		

For batch 7 the average is 81.2GPa with a standard deviation of 26GPa, but if the outlier, sample 11, is removed these values change to 71.7GPa and 7.9GPa respectively. Batch 8's mean modulus is 114.8GPa with standard deviation of 29.7GPa. Batch 9 has 247.5GPa as its mean with a standard deviation of 99.2GPa. These deviations are not insignificant but possibly will decrease as more data becomes available from more tests on more samples.

CHAPTER FIVE

Conclusions

5.1 Conclusions

Based on the data from the samples whom experience successful test this tensile test method is valid for the determining structural properties of individual carbon fiber. However, it could use more modification to improve its precision and to eliminate the fault that resulted in the fiber slipping due to adhesive failure. The test method possesses good quality for the amount of carbon fiber material and the cost of other materials consumed during the test method. It does show the linear relation of stress to strain via Figure 48. This shows the modulus of elasticity is constant until fiber failure, and that the carbon fiber is very brittle. The Modulus of Elasticity is similar to known materials, such as steel, that possess an elastic region that also demonstrate a similar relation of stress to strain in it. In addition materials with brittle properties, such as ceramics, have similar moduli that are linear until failure. The samples' failure strengths are very close to the strengths listed by manufactures from their expensive, high quality tests.

5.1.1 Fiber-Bundle Relation

It can be said with adequate confidence that the fibers of carbon fibers possess the majority of the strength exhibited by bundles of fibers. However individual fibers' strengths are only modestly lower, most likely due to the lack of interaction with other

fibers to gain secondary, physical strengths. Property wise fibers demonstrate characteristics similar to those of entire bundles. Their stress increases linearly to failure, along with strain.

5.2 Progression

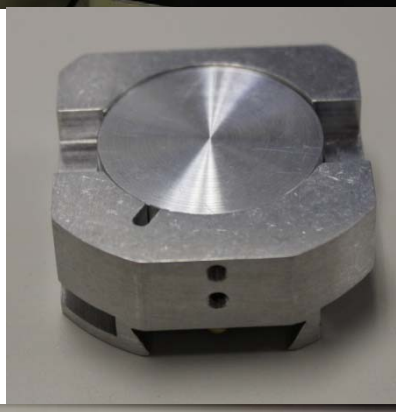
In addition to the improvement to the test method, The fixture material may be changed to a thin plastic which could be melted with after being loaded into the DMA-Q800 to eliminate the shock stress from separating the fixtures' sides. An natural next step would be to use the test method to determine properties of unusual fiber materials who's qualities are unknown, such as banana fibers. This is would be a natural progression for the test method as it relates to fibers. Another possible path would be increase the implementation of the SEM in the test method. Baylor University's SEM is one of five in the nation with a load cell that can be installed in it. The load cell would operate fundamentally the same way that the DMA-Q800 does for this method, but it would reduce the machines used and combine the optical and physical measurements of the sample to the same machine. However the load cell operates on strain rate instead of force. In addition this permit the potential to video record fiber failure. A third avenue this test method could progress down is to start testing samples under environmental stresses and conditions, such as temperatures other than room temperature. For this method the adhesive would have to be checked to see if it would still function at the desired temperature, and possible replaced if it fails to.

APPENDIX

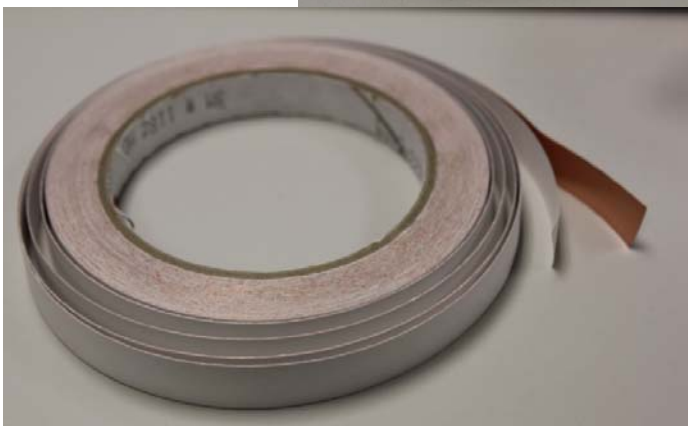
MATERIALS LIST

JEOL JSM-6610LV Scanning Electron Microscope(SEM): - imaging and determining physical dimensions of sample carbon fibers.

32mm Specimen Holder: supports and hold sample in position in SEM.



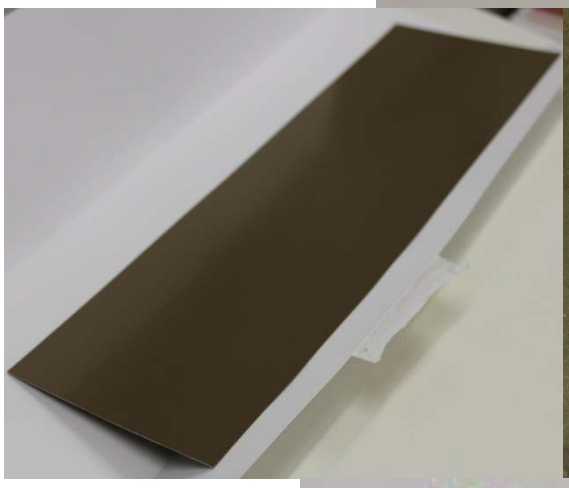
Copper Tape: connects sample to 32mm specimen holder.

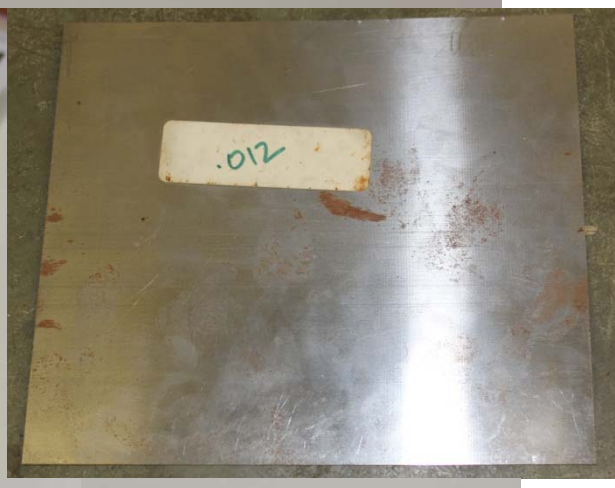


Specimen Exchange Tool: Used to insert and remove 32mm specimen holder to and from the SEM.

Forceps: grip sample while positioning on copper tape, and when removing sample from tape.

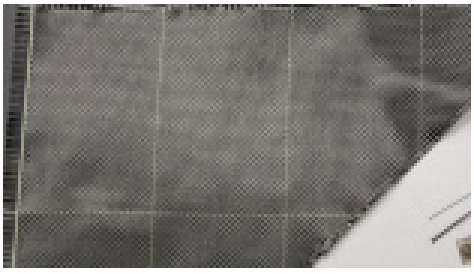
Sheet Metal: Material used to construct sample spacers.

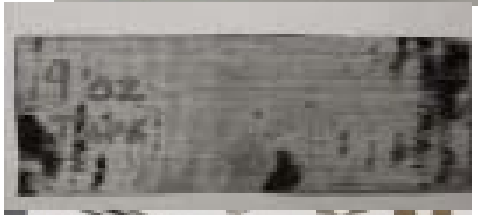


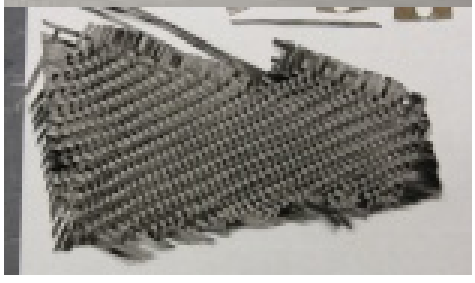


Superglue: Secures fiber sample to sample spacer.

Carbon Fibers: Material used to isolate fiber from for samples.







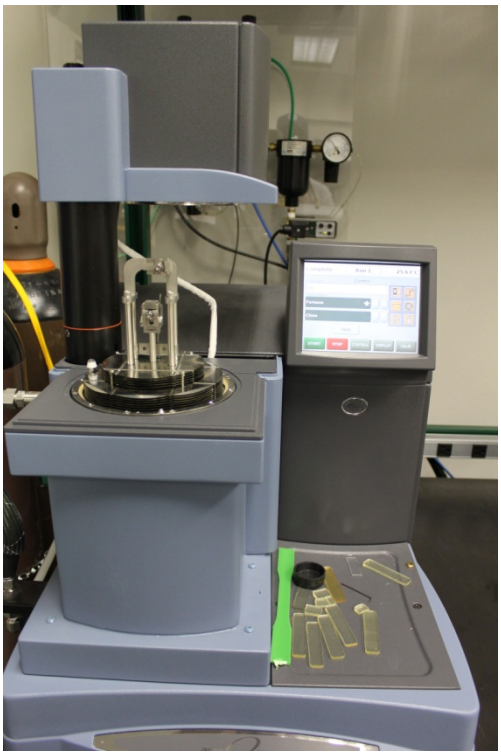
Shear Press Cutter: Cuts sheet metal into strips for first part of spacer constructi on.

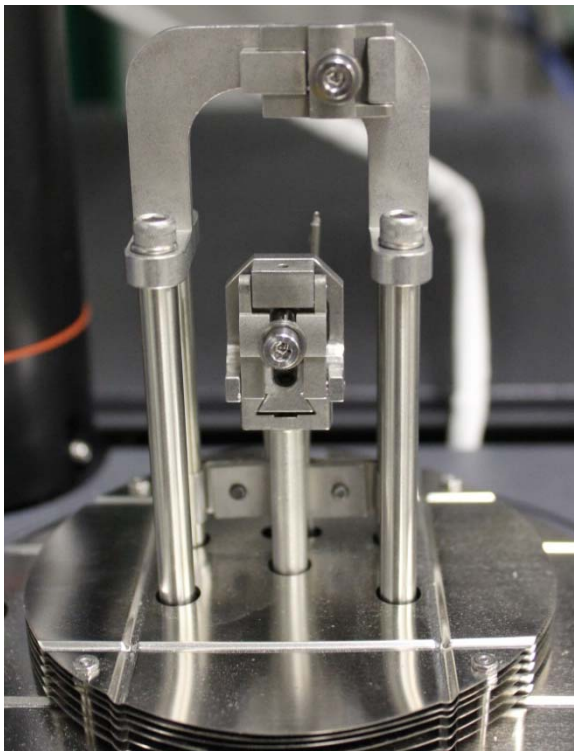
Metal Cutting Snip: Cuts sheet metal strips into spacers, and trims spacers in construction process.

Tin Snips: Separates spacer sides, and trims excess fiber length from samples.

Hole Puncher: Punches hole in sample spacers during spacer construction.

Dynamic Mechanical Analyzer-Q series(DMA-Q800): The Q800 DMA is a precision instrument designed to measure viscoelastic properties , such as modulus and damping of rigid and soft solid materials. [1]See image on the left, below.





Tensile Test Clamp assembly: Installed on the DMA-Q800 to perform tensile tests on sample's fibers. See image of the right, above.

Summit OptixCam with 2X ScienceScope M27-CP-20200M (OH3) scope with 0.5X focus: used for diameter measurements, during initial demonstrations before adaptation of test method.{no image}

Calipers: used for length measurements, during initial demonstrations before adaptation of test method.

ScienceScope IL-FOI-150 plus light source: Used to provide additional illumination when using the Summit OptixCam.

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