Abstract

The current challenge with qualification of carbon fiber composites in the aerospace industry would be the low efficiency of testing hundreds of samples. The Trace Theory strives to streamline the qualification process by utilizing a material's Trace to predict properties of composites using Excel programs and basis data. To test this theory, predicted properties from the program, QuickLam, were compared to experimental data. Unidirectional 0° (T1), unidirectional 90° (T2), quasi-isotropic (T3), and hard quasi-isotropic (T4) laminates were made using HexTow® carbon fiber and TC250 resin provided by TenCate Advanced Composites. Tensile and compression tests were done according to ASTM D3039 and ASTM D695. Five samples were tensile tested for each orientation, with tensile strengths ranging from 39 MPa to 1806 MPa across T1-T4. Six samples were compression tested for each composite orientation with strengths varying from 236 MPa to 1198 MPa across all the laminates. The experimentally measured tensile and compressive strengths were then compared to the values predicted using T1 laminate results as a basis in QuickLam. The most accurate prediction from the program was for the hard quasi-isotropic layup, T4, which yielded a percent difference of 3.6% for tensile strength and 6.7% for compressive strength. It was concluded that although more testing would need to be done, the Trace Theory can be accurate in predicting properties for certain laminates.

Key Words

Materials Engineering, aerospace certification, Trace Theory, carbon fiber composite materials, tensile tests, compression tests, stiffness matrices, composite failure modes, delamination, strain gages, equivalency testing, laminate configurations

Acknowledgments

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1. Introduction

1.1 Problem Statement

The aerospace industry continues to expand and evolve with new technologies and revolutionary materials. Every innovation must undergo thorough qualification processes to meet set requirements. The current challenge with material qualification for the aerospace industry is the large amount of money and time required to test hundreds of samples. Some materials, such as metals, can be mechanically characterized to reduce the amount of samples needed for testing. Composites are difficult to mechanically characterize due to their anisotropy and complicated failure mechanisms. Therefore, composites are expensive and can take years to qualify for use in aerospace applications. Dr. Stephen Tsai, from Stanford University, and his colleagues have worked extensively to develop Trace Theory. This theory would cut down on the amount of testing for composite materials. It is based on the concept that by using invariants in a stiffness matrix and applying Hooke's law, the strength of that composite material can be predicted. Since the 1960s this theory has evolved to use scaling factors to predict various specific mechanical properties of unique orientations of composites. Different versions of programs have been developed to output these predicted mechanical properties using the Trace Theory.

To address the current problem of costly and time consuming composite qualification, tensile and compressive testing will be performed and compared to the Trace Theory predicted values for different orientations of a carbon fiber composite. The goal of this project is to prove that the Trace Theory can correctly predict the properties of carbon fiber composites. Testing methods and analysis techniques to accomplish the project goals include tensile and compressive tests, as well as statistical analysis to document the differences between the experimental data and the program's predicted values.

1.2 Material Qualification

Composites used in aircraft components carry a large liability. These materials must withstand harsh conditions and maintain specific standards. For example, a material used in an airplane wing must keep its strength for the lifetime of the airplane in order to ensure safety of all those on board. Thus, material qualification is an integral part of designing aircraft components. Material qualification requires extensive testing, large amounts of money, and years to accomplish. For example, a unidirectional ply of a composite has four stiffness properties as well as five strength properties. Furthermore, when composite laminates with several orientations are made from unidirectional plies, there are numerous combinations of these properties that all must be reported.

Before 1995, individual companies used customized qualification programs that were extremely expensive and differed between companies.[1] This led to companies solely relying on accepted materials instead of innovating and spending the time and money to qualify a new material. In 1995, a central material qualification database was developed to prevent this problem. By 2005, the Federal Aviation Administration (FAA) and the National Aeronautics and Space Administration (NASA) came together along with the National Institute for Aviation Research (NIAR), to form a recognized database called the National Center for Advanced Materials Performance (NCAMP).[1] This national database is still in use today, and follows regulations and procedures set by the FAA to ensure reliability and consistency across all materials.

Although qualifying materials, specifically composites, became faster and cheaper due to the formation of NCAMP, materials still must go through a rigorous procedure set by the FAA. This procedure specifies everything from the layout of each composite panel, to the form of testing and amount of samples needed to qualify the composite. In general, the amount of testing required is based on the purpose for the material system. The FAA explains two routes of testing: an A-basis and a B-basis. An A-basis would be used for a material system exposed to a single load path, whereas a B-basis would be used if multiple load paths exist in the design.[2] For an A-basis and a B-basis, a robust sampling method can be used shown in Figure 1. Only a B-basis can use a reduced sampling method (Figure 2). The robust sampling method, A-basis, would require five unique batches of material to produce 55 samples for each environmental condition and test method. For example, this would mean 55 samples would be administered to tensile test a material at a dry, elevated temperature condition. A reduced sampling method would have 18 samples per environmental condition and test method. There are four environmental conditions outlined by the FAA: cold temperature dry, room temperature dry, elevated temperature dry, and elevated temperature wet.[2] Even if the reduced method of testing can be applied to a material, this would still add up to hundreds of samples, a great expense, and many years to complete. Continuous research is being conducted to produce a way to generate the mechanical properties for composite materials in order to reduce this extensive testing process.

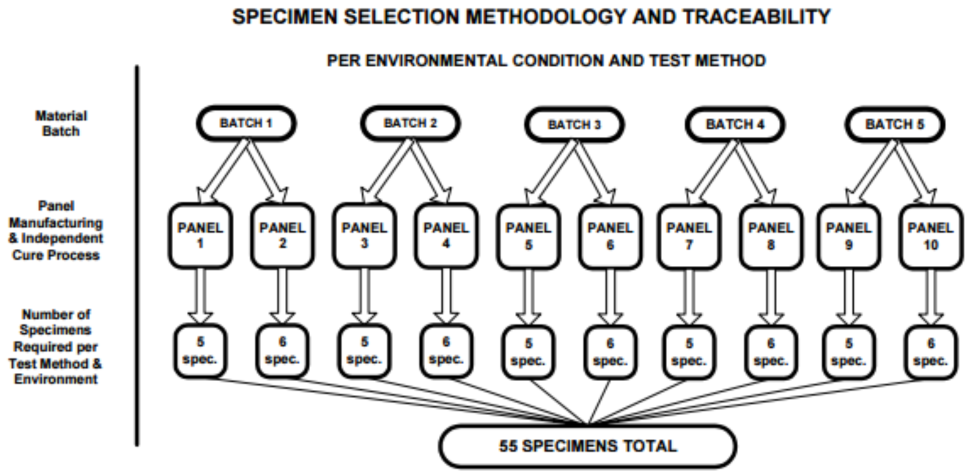

Figure 1. Robust sampling method for specimen testing for both A-basis and B-basis.[2]

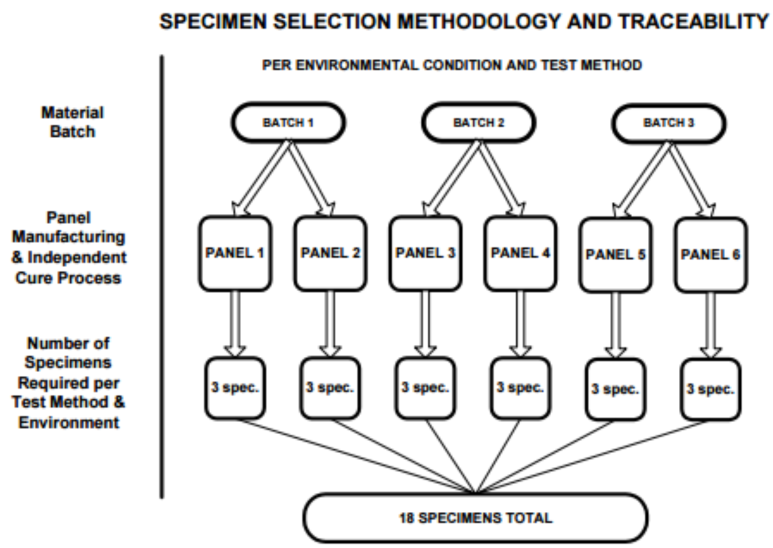

Figure 2. Reduced sampling method for material qualification for B-basis.[2]

1.3 Trace Theory

Carbon fiber reinforced composites have become an integral part of the aircraft industry due to their unique specific weight, specific modulus and high corrosion resistance. However, because composites are anisotropic and have complicated failure mechanisms, the mechanical characterization of the material is difficult[3]. Efforts have been made in an attempt to predict properties of composites. One theory, known as Trace Theory, strives to do so. For a given material, much like Young's modulus, Trace is a material property. The Trace is an invariant of a normalized stiffness matrix of that specific material. It is the sum of the diagonal components of the normalized extensional matrix (Equation 1)[3].

$$T = A^*_{11} + A^*_{22} + 2A^*_{66} \quad \text{(Equation 1)}$$

The matrix [A*] simply represents a normalized extensional stiffness matrix for a laminate. The stiffness of the laminate [A] can be normalized by thickness so that the material properties are useful for direct comparison with other materials. This also allows for the units of the [A*] matrix to be in GPa rather than N/m. Trace (T) is a linear combination of three terms: longitudinal modulus (A^*_{11}), transverse modulus (A^*_{22}), and shear modulus (A^*_{66}). In carbon fiber-reinforced polymers, longitudinal modulus makes up 88% of the Trace value, whereas transverse and shear modulus combine to make up the remaining 12%. This is due to several properties being dependent mostly on the fiber of the composite. An example of how Trace works can be seen in Figure 3. The two materials, AS4/MTM45 and IM7/997-3, are represented in two different stacking sequences, [π/4] and $[0_5/\pm45_2/90]$. The normalized stiffness matrix [A*] was determined through testing for each material and stacking sequence and normalizing by thickness. By applying Equation 1 to these values, the Trace was determined for each combination. The Trace value was calculated to be the same for each material regardless of the stacking sequence.

This shows how the Trace of the material is an invariant property and is independent of the stacking sequence. Before discovering Trace, each of the four stiffness matrices would be unique and independent. Now, there is a connection.

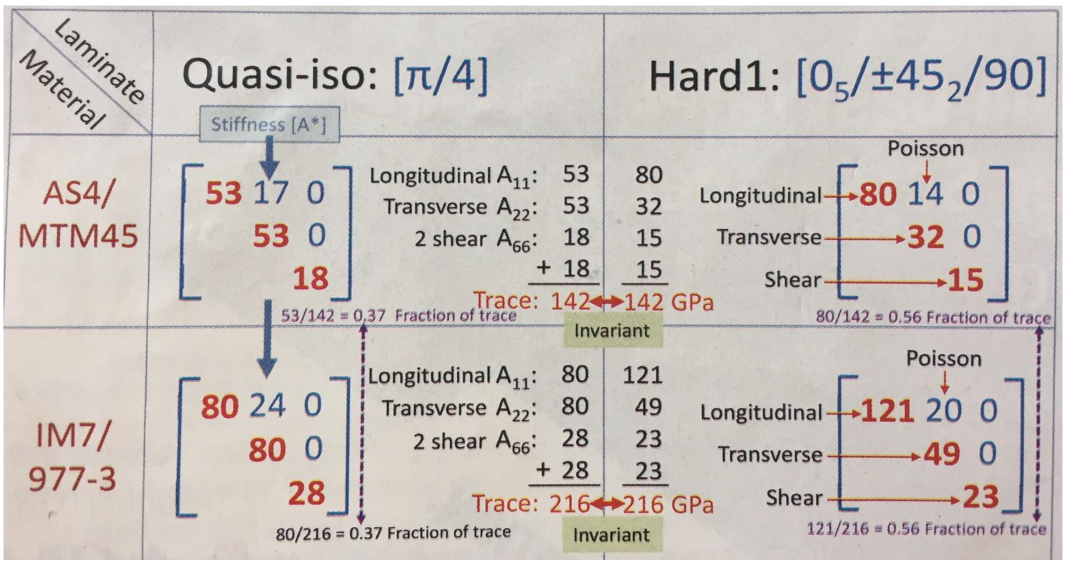

Figure 3. Two materials are shown with different laminate configurations. For both, the Trace was found from the normalized extensional stiffness matrix.[4]

Another connection can be made between stacking sequences. By taking the diagonal values of the stiffness matrix, and dividing them by the Trace value (e.g., 53/142 = 0.37), a fraction is determined (Figure 3). This fraction remains the same for each stacking sequence.[4] For example, for the quasi-isotropic stacking sequence, the longitudinal stiffness of AS4 divided by its Trace value, gives the fraction of 0.37; which is the same value as the longitudinal stiffness of IM7 divided by its Trace. For each stacking sequence, this fraction of Trace remains the same. This

mathematical connection is what allows the Trace Theory to have such an impact. These fractions have evolved to incorporate many other stacking sequences.[4]

These connections prove important for testing. A laminate's stiffness matrix only needs to be solved for once. If a new material is developed, it is not necessary to perform hundreds of tests. The predicted properties for the material can be derived from simple mathematical calculations of the material's Trace and various other fractions.[5] Each stacking sequence has a specific fraction that remains the same and can be applied to solve for all variations for the new material.[4] Other mathematical factors, known as scaling factors, that can be applied to a material's Trace include vibration and stress concentrations, buckling and natural frequencies, and deformation.[4] A scaling factor is a numerical value that represents a certain orientation or condition that is then multiplied by the material's Trace.[6]

All of these mathematical relationships are compiled and used to produce outputs through different programs developed by Dr. Tsai, et al. One program developed by Dr. Jose Melo, called QuickLam, utilizes these mathematical relationships to predict compressive and tensile strengths of various laminates. For this specific program, the material's longitudinal modulus, unidirectional tensile strength and compressive strength are used to provide the material's Trace as well as predicted properties. Therefore, values from tensile tests and compression tests of one unidirectional laminate could be used to produce predicted tensile and compressive properties for numerous combinations of orientations and environmental conditions. In theory, two tests (tensile and compression) can be performed for a room temperature condition of a unidirectional laminate, and predict properties for all other orientations and conditions. This would cut down on the amount of testing needed for these important properties.

The basic idea behind Trace Theory is to perform minimal tests on a composite laminate to produce the material's Trace and a basis value for the desired property (i.e., compressive strength). These values are then entered into a program and complex mathematical relationships calculate the predicted desired properties of different orientations and environmental conditions.

1.4 Testing

The performance of composites is characterized by conducting hundreds of standardized mechanical tests. However, Trace Theory focuses on minimizing the number of mechanical tests used to characterize composites. Mechanical properties determined by Trace Theory are predominantly based on the longitudinal modulus of a material (88%). Transverse and shear moduli also make up the Trace value; however, these properties, when combined only make up the minority of the Trace value (12%).[5] To obtain the longitudinal modulus, several tension tests following ASTM D3039 are performed on 0° unidirectional laminates.[7] In accordance with this standard, the tensile specimen is shaped as a rectangular coupon with a constant cross section

(Figure 4). To improve the quality of the tensile test, fiberglass–epoxy tabs are bonded to the grip ends (Figure 4).[8] The tabs are typically non-woven, [0/90] laminates that are beveled up to where the gage length of the coupon begins (ASTM D3039). This reduces stress concentrations which therefore lowers the likelihood of failure in the grip ends of the sample during testing.[8] Strain gages are applied on the gage length of the coupon in both the longitudinal and transverse directions to measure the strain that occurs as the sample deforms.[7] The use of the strain gages optimizes the accuracy of the test while the material deforms in its elastic loading behavior.

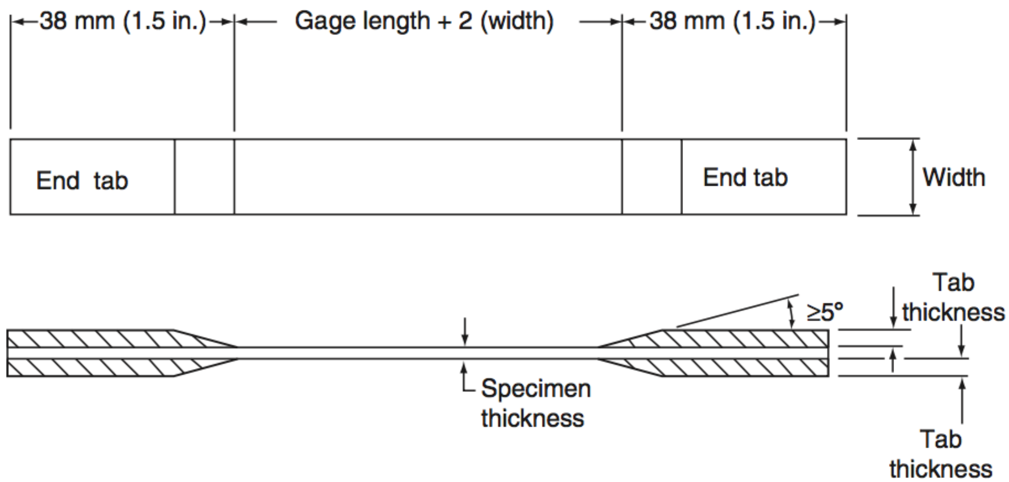

Figure 4. Schematic of tensile test specimen configuration based on parameters listed in ASTM D3039.[7]

Tensile strength, longitudinal tensile modulus and Poisson's ratio can be determined from the tension test of a unidirectional laminate that is run until failure. Tensile strength and modulus for unidirectional laminates are greatly dependent on the fiber orientation angle, θ.[8] As the angle of the fiber orientation increases, both tensile strength and modulus decrease.[8] The maximum tensile strength of a composite occurs in 0° specimens, while 90° specimens demonstrate the lowest values. The stress-strain curve of a tensile specimen has a linear elastic region, which begins to drop rapidly where the specimen fractures.

Compressive properties also play an integral part of basic design for composite materials. Although Trace itself does not require compressive properties, compression testing is necessary in understanding material properties of a composite. Compression tests following ASTM D695 can obtain the compressive strength and modulus of a composite.[9] It has been observed that unlike tensile stress-strain curves, the compressive stress-strain curves of 0° laminates may not be linear.[8] This is because compressive strength is dependent not just on fiber orientation, but fiber type, fiber volume fraction, matrix yield strength, and other properties of the matrix. Therefore, compressive properties can be difficult to determine through testing. Buckling of samples can occur, giving false strength and modulus properties. ASTM standards recommend using a test fixture (Figure 5) to secure the sample to prevent this.

Figure 5. Example of a compression test fixture for ASTM D695.[9]

1.5 Material Background

1.5.1 Fiber

This project investigates mechanical properties of carbon fiber reinforced composites. However, to understand the properties, the structures of the fiber and matrix must be known. The graphitic form of carbon has a high tensile modulus due to the way the carbon atoms are arranged in the crystallographic structure of the parallel planes.[8] Within a plane, the carbon atoms are covalently bonded, while the parallel planes are bonded together by secondary Van der Waals bonding (Figure 6). This specific atomic structure results in highly anisotropic physical and mechanical properties of the carbon fiber.[8]

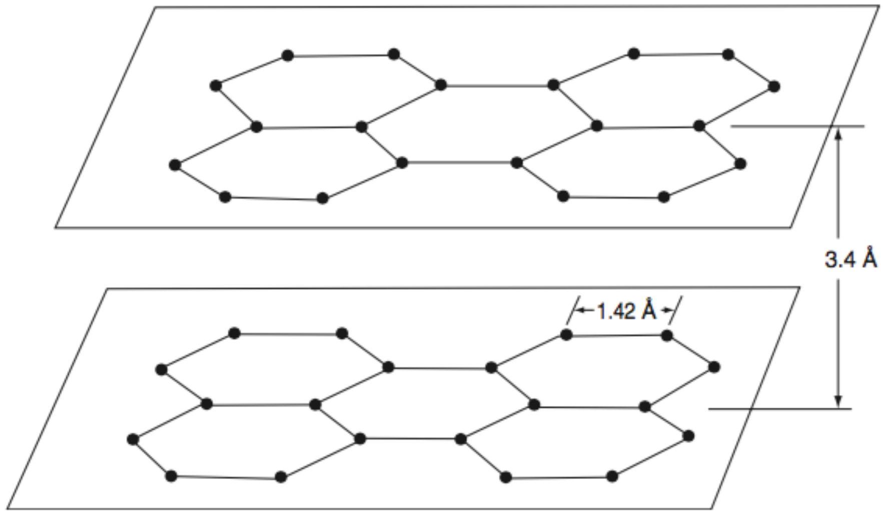

Figure 6. Covalently bonded carbon atoms in parallel planes in a graphite crystal.[8]

These fibers are produced in a continuous operation where the fiber precursor, polyacrylonitrile (PAN), undergoes three stages: stabilization, carbonization and graphitization. PAN is the most commonly used precursor for carbon fiber composites. The molecular structure of PAN contains highly polar CN groups that are randomly arranged following syndiotactic stereochemical configuration (Figure 7).

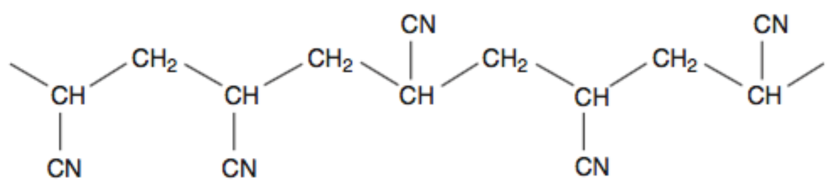

Figure 7. The random molecular arrangement of polyacrylonitrile.[8]

The PAN solution is spun into filaments, which are further elongated at an increased temperature to initiate alignment of the polymer chains. The filaments are stabilized through heat treatments at 200°C to 300°C to form a rigid ladder structure (Figure 8). Carbonization then takes place at the next stage, where the PAN filaments are heated to 1000°C to 2000°C in an inert atmosphere to eliminate oxygen and nitrogen atoms.[8] Tension on the filaments is held constant to prevent shrinkage and improve alignment in polymer chains. With the removal of oxygen and nitrogen atoms, the filaments now contain mostly carbon atoms arranged in an aromatic ring pattern in parallel planes. To improve properties further heat treatment at above 2000°C is done to assist in the alignment of the graphitic parallel planes.[8] The final carbon fiber structure is then produced, which gives the fiber its high strength and modulus properties.

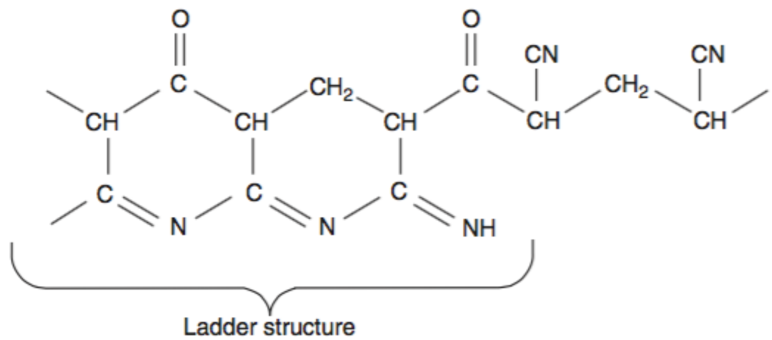

Figure 8. Rigid ladder structure formed after heat stabilization.[8]

Subsequent surface treatments and fiber sizing further enhance bonding ability to the polymer matrix as well as ensure the fibers do not fracture or deform during the manufacturing of the composite.

This project uses HexTow carbon fiber manufactured by Hexcel. Hexcel is a leading manufacturer of carbon fibers that are certified to meet specific aerospace and industrial specifications. The specific carbon fiber type used in this study is designated as AS4C-GP, 12k tow. Alone, this fiber has excellent tensile strength and elastic modulus, with values measuring 4482 MPa and 231 GPa, respectively.[10] The fiber would be best used as a weave or prepreg tape, while sizing levels range from 0.2-1.2. The AS4C-GP continuous fiber is compatible with epoxy, phenolics, vinyl ester, polyurethane, and cyanate ester.[9]

1.5.2 Matrix

The matrix material of fiber-reinforced composites is primarily used to transfer applied loads to the fibers, as well as protect the fibers and keep them in place. Although the matrix does not heavily influence the tensile properties of the composite, it does influence the compressive, interlaminar shear and inplane shear properties.[8] Epoxy is one of the most commonly used resin materials, especially in aerospace and aircraft applications. The molecules of the thermoset polymer are held together by a cross-linked network structure containing epoxide groups, shown in Figure 9. The presence of the functional group classifies this material as an epoxide; however, the base molecular structure to which it is attached is chemically compatible with most substrates, which which gives them good processing versatility.[11] Modifiers are easily incorporated to improve the properties, such as impact strength, of the cured epoxy matrix.

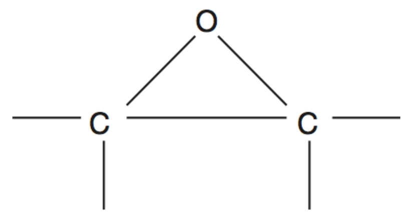

Figure 9. Epoxide functional group found in epoxy resin starting material.[8]

To transform the liquid epoxy resin to a solid, a curing reaction is initiated by adding a curative just before the fibers are incorporated into the liquid mix.[8] As the reaction proceeds, the curing agent forms cross-links and a three-dimensional structure is slowly formed.

Some manufacturing processes require a partially cured resin system. The curing reaction can be hindered by lowering the reaction temperature, so that not all the molecules of the resin are cured, and the resin would exist in a B-stage form and can be completed at another time.[8] The hardness, tackiness, and curative reactivity of the material depends on the degree of cure once the reaction has been delayed.

As the crosslinking density increases, the tensile modulus, glass transition temperature, and thermal stability are enhanced.[11] However, because of the cross-linked structure, the strain-to-failure and fracture toughness are considerably reduced. To compensate for these low properties, a liquid elastomer or thermoplastic can be blended in to toughen the epoxy, forming a second phase in the cured matrix that can impede microcracking.[8] This modified form of epoxy has higher toughness compared to unmodified epoxy.

The epoxy matrix used in this project is manufactured by TenCate Advanced Composites, designated TC250, which is typically used for aircraft structures and space structures. TC250 is a toughened epoxy resin system that provides good toughness, mechanical property translation, and hot/wet performance. The resin system is easily processed using vacuum bag/oven, autoclave, or press curing operations, at a 265°F cure.[12]

1.6 Industry Sponsor

TenCate Advanced Composites is an industry leader specializing in the production of advanced composite and prepreg materials.[12] TenCate emerged from the Dutch company, Royal TenCate, which focused on textiles for over 300 years.[12] Over time, TenCate has become a leading innovator in thermoset and thermoplastic composites in the aerospace and industrial sectors. Their thermoset materials have been used in aircraft components and radomes, as well as missile applications, while thermoplastics have been used as structural materials for commercial

aircrafts.[12] The company is also known for their unique resin systems that are integrated into their composite materials. TenCate has successfully dedicated the last 20 years to incorporating thermoset and thermoplastic technologies into European and North American markets.[12] They are currently working to develop new technologies and grow as a company. With the high demand for innovative materials, especially in the aerospace industry, TenCate would benefit greatly with a reduced testing method for material qualification. By implementing the Trace Theory, the amount of time and money TenCate spends on material qualification would significantly decrease.

2. Experimental Procedure

2.1 Preparing the Laminates

TenCate Advanced Composites provided 100 yards of prepreg material containing HexTow carbon fiber and TC250 prepreg. Four orientations were predetermined to be used for testing and named T1, T2, T3, and T4 laminates (Table I). To prepare these laminates, plies of the prepreg were cut into 12x12" pieces. Four plies were debulked for 15 minutes in a vacuum chamber to remove excess air and encourage a more even resin flow during curing. After all the plies were debulked and stacked together in the proper orientation, they were layed-up on a large steel plate and prepared according to LU-1 lay-up method. Dams were placed around the panels with non-porous FEP beneath and on top of the panel. A layer of porous Teflon and a caul plate was placed on top to ensure resin flow throughout the panel. A layer of breather covered the top of the panels and the entire steel plate was vacuum bagged. The laminates were cured in an autoclave up to 265°F (Figure 10). After curing, the laminates were C-scanned to assure the resin spread evenly during the curing process. All panels were approved, and then machined using a table top grinding saw into the proper sizes per ASTM D3039 and ASTM D695 for coupon testing. All coupons were tabbed using fiberglass and TC263 adhesive provided by TenCate. This was to provide better grip for tensile testing and is necessary for compression testing following ASTM D695. After the preparation of the samples was complete, the samples were brought to Cal Poly for testing.

Table I. Laminate Configurations

Sample	Orientation	Layup Sequence
T1	Unidirectional 0°	[0°]8
T2	Unidirectional 90°	[90°]16
T3	Quasi-isotropic	[+45/90/-45/0°]2s
T4	Hard quasi-isotropic	[0/45/0/-45/0/45/0/-45/0/90]s

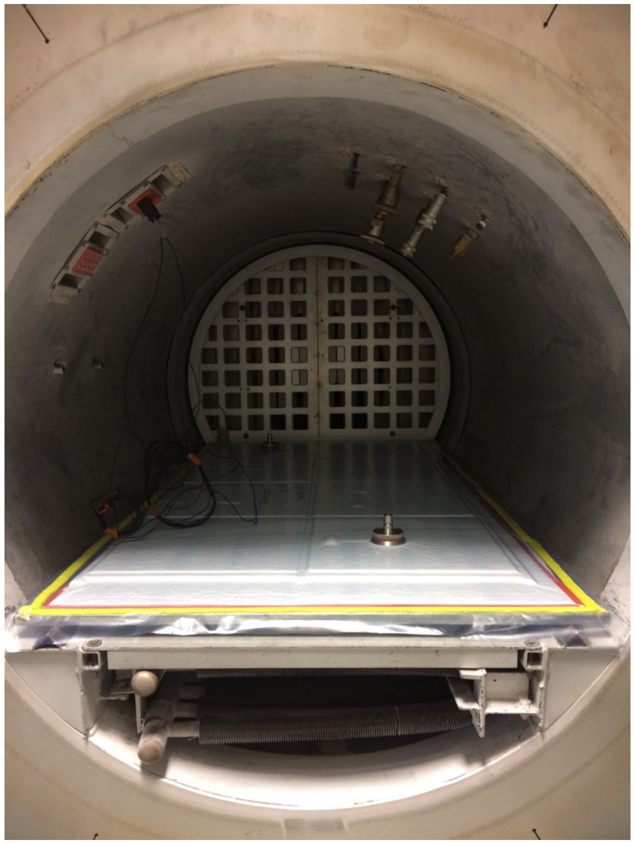

Figure 10. Composite panels on a steel plate under a vacuum bag following the proper lay-up method. The steel plate is loaded into an autoclave for the curing process.

2.2 Tensile Testing

For tensile testing, ASTM D3039 specified sample dimensions based on the orientation of the laminate. As stated, all samples had fiberglass tabs attached using adhesive. For all four laminates, five test coupons were tested in compliance with ASTM D3039. For the T1 laminates, the test coupons were cut to 254 mm long and 12.7 mm wide, with a gage length of 127 mm. The remaining laminates measured to be 254 mm long, 25.4 mm wide, with a gage length of 127 mm. To prepare the T1 test coupons for tensile testing, uniaxial strain gages were applied to the surface to obtain a more precise elastic modulus value. The elastic modulus data for the T1 samples must be more exact because this modulus will be used in the Quicklam program to determine the material's Trace value and predict properties of other laminates.

Strain gage application was done by roughening the gage area with emery paper and cleaning the area using isopropyl alcohol. Next, the gage length was measured and marked at the exact halfway point. A conditioning acid was applied to the area using gauze and followed with neutralizing base to ensure no remaining acid was left on the sample. The strain gage was aligned in the proper area using cellophane tape. A catalyst was then applied to the back of the strain gage and left to dry for several minutes. Once dry, the adhesive was applied and the strain

gage was pressed down on the sample with light pressure. Wires were soldered onto the leads of the strain gages to connect with the strain measurement software (Figure 11).

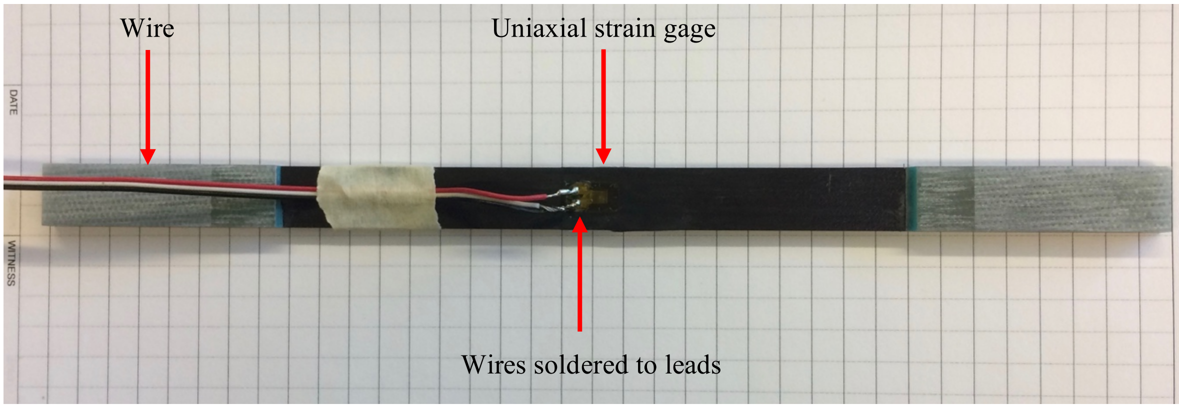

Figure 11. Uniaxial strain gage attached to the T1 sample. Wires are soldered on the leads of the strain gage and taped down to avoid being broken off.

Once the strain gages were applied, the test method was produced on the Shimadzu Trapezium software following ASTM D3039, setting the crosshead speed to 2 mm/min until fracture. The test reported the maximum load, which calculated the maximum stress based on the dimensions of the specimen. The specimen geometry was measured using a caliper. The width, thickness, and length of the sample was measured and recorded. Before the specimen was placed in the tensile grips, the machine was calibrated and the alignment was inspected using a steel plate. The specimen was then secured into the machine, making sure the it was aligned within the grips. A HEPA filter vacuum was attached to a 3D-printed chamber that enclosed the sample during testing to ensure that no broken fiber particles circulated in the air after testing (Figure 12). A polycarbonate shield was placed in front of the fixture for added protection. For each sample, the wiring from the strain gage was connected to the StrainSmart strain software, which measures the strain every ten milliseconds. Once connected, the software was calibrated with the strain gage to secure the connection and to verify the strain gage was functioning properly. During calibration, if the meter in the software read above 1 µstrain and below 35,000 µstrain, the test was ready to run. However, because the StrainSmart software was not connected with the Trapezium software, there was a time delay between recording the strain data and recording the strength data from the tensile test. In order to solve for the precise modulus after testing was completed, the strain and strength data were directly compared. Therefore, the time between starting the StrainSmart software and beginning the tensile test was recorded to account for the delay in the data.

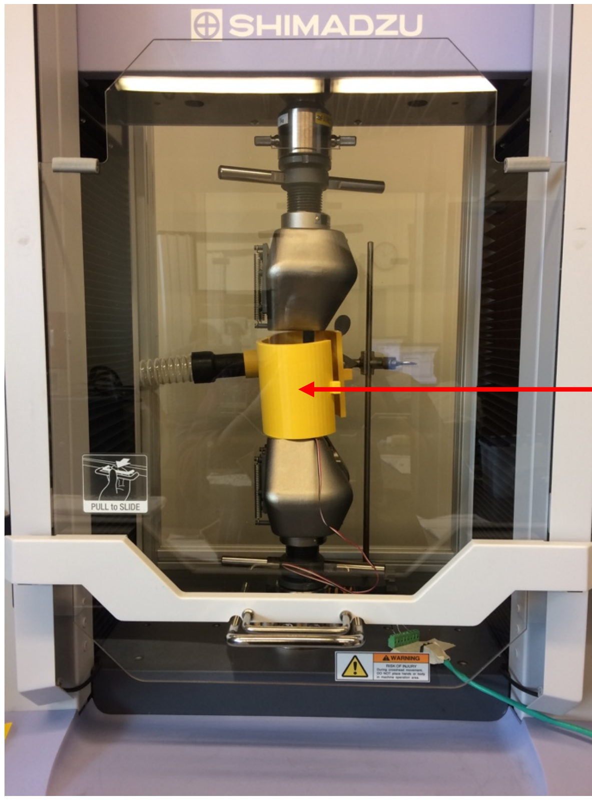

Figure 12. Set up for tensile testing on the Shimadzu test frame. A vacuum was attached to a chamber that surrounded the test samples.

To calculate the elastic modulus of the T1 samples, raw strain and stress data were extracted and compiled into an Excel spreadsheet. Both the Strainsmart software and Trapezium software records data points every 10 milliseconds, so therefore the data points were able to match. The strain data measured from the Strainsmart software was recorded in microstrain, therefore this was accounted for by dividing by 10^6. Then all the data points were plotted against each other in a stress vs strain graph. Four graphs were produced for four test coupons. Unfortunately, one test coupon did not produce strain data and therefore five test coupons did not produce graphs due to an error in the StrainSmart software. A best fit line was attached and the equation of the line was displayed. This is using Hooke's Law (Equation 2) to solve for the elastic modulus from the stress (σ) and strain (ε) data. The average of the four moduli was taken to be the average elastic modulus for T1.

$$E=\sigma/\varepsilon \qquad \text{(Equation 2)}$$

The remaining T2, T3, and T4 laminates were tensile tested on the Instron test frame. For these tests, the elastic modulus values did not have to be as precise, so an extensometer was used. The Instron test frame is equipped with an extensometer, whereas the Shimadzu is not. A test method was produced in the BlueHill software following ASTM D3039. The crosshead speed was set to 2.00mm/min. The dimensions of each test coupon for the three laminates were measured three times, averaged, and recorded. The test frame and extensometer were calibrated before loading

the specimen into the tensile grips. In order to prevent any harmful exposure to composite splinters, the vacuum chamber apparatus and polycarbonate shield were used for all tensile tests performed on the Instron. After dimensioning and set up was complete, the coupons were loaded into the test frame. For T2, the extensometer was set to be removed at 1% extension. However, the samples started to break before the 1% extension, so the extension was reduced to 0.5% for the T3 and T4 samples. The load and extension were recorded by the Instron software and the modulus and strength were calculated based on the specimen geometry.

During all tensile tests and handling of the test coupons, nitrile gloves and safety glasses were worn to protect from harmful carbon fiber splinters.

2.3 Compression Testing

The test coupons were prepared per ASTM D695. To comply with this standard, the coupons were cut using a precision machine to 79mm long and 2.75mm wide. Each had 37mm tabs leaving a gage length of 4.8mm. The ASTM D695 calls for five samples to be tested, so for this project six were tested for more representation. All the laminates (T1-T4) were compression tested using a fixture (Figure 13). The use of this fixture is to provide stability and to encourage a failure in the gage length of the sample. Before testing, all six samples for each of the four laminates were measured using a caliper. The width and thickness was taken in three places, averaged, and recorded. The thickness of the samples was measured in the gage length of the samples.

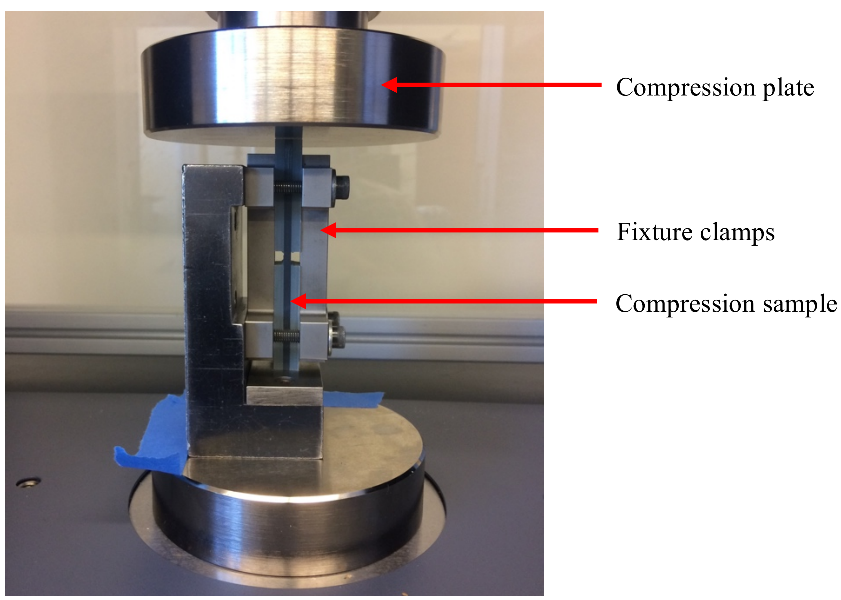

Figure 13. A tabbed compression sample is clamped into the ASTM D695 fixture using a torque wrench. The sample remains flush with the bottom of the fixture to ensure the load is applied evenly across the top of the sample.

The compression tests were performed using the Shimadzu test frame. A test method was produced in the Trapezium software following ASTM D695. For this compression test, each sample was loaded in the fixture and carefully tightened using a torque wrench so that the bottom

of the sample laid flat with the bottom of the jig. The torque wrench was set to 15 in-lbs. This was determined after preliminary testing proved that the 10 in-lbs. stated by the ASTM D695 was not enough torque to hold the samples in place and produce a failure in the gage area. Preliminary tests were performed on extra samples that were also cut to the standard size. These tests were run to determine the proper torque as well as to set a crosshead speed that was in range of the ASTM standard. The crosshead speed was set to 1.27mm/min. After preliminary tests were completed, and the sample information for all the test coupons were recorded the test coupons for each of the laminates were loaded into the fixture and tested in the Shimadzu load frame. The maximum load of the compression samples was recorded as well as the displacement. The compressive strength was calculated and recorded based on the sample dimensions.

At all times while handling the test coupons, nitrile gloves and safety glasses were worn. This is to prevent any composite splinters from causing any harm.

2.4 Equivalence Testing

After collecting the data from the tensile and compression tests, an equivalency test was performed to verify if the data was comparable to values TenCate Advanced Composites had recorded. This was done using a program called HYTEQ (Hypothesis Testing of Equivalence) that was produced by Wichita State University in 2007 for polymer matrix composite material systems. HYTEQ is used for a comparison of an acceptance or equivalence data set to data generated by the qualification data set as material basis values. TenCate provided the basis data for 0° tensile strengths, elastic modulus, and compressive strength data for the same Hextow® carbon fiber and TC250 resin system from a previous qualification project. The HYTEQ program compares the qualified and equivalence data using the t-test of the mean for modulus properties and a t-test of mean and minimum values for strength properties. The mean, standard deviation, coefficient of variance, minimum, maximum values, and number of specimen are entered into the program. The tests are performed for equivalence at α=.05 and acceptance at α=.01. Therefore, for strength values, the data set has two equivalence tests and two acceptance tests. For modulus values, there is one equivalence and one acceptance test run on the data sets. TenCate as well as other companies use this HYTEQ tool to compare a small data set to a data set of the qualified material to ensure that the small data set can be considered accepted or equivalent. T1 tensile and compressive strengths, as well as elastic modulus were compared to the data provided by TenCate to either prove or disprove equivalence or acceptance.

2.5 QuickLam Program

To validate the Trace Theory for this project, the recorded data must be compared to the QuickLam program predicted data. Quicklam is an Excel-macro that was produced by Dr. Daniel Melo in August of 2015. This program applies the complex mathematical relationships between a materials Trace and other properties. QuickLam produces predicted compressive, tensile strengths, and elastic modulus. In order to do so, the modulus, compressive strength, and tensile strength of a 0° unidirectional laminate must be input into the program in section 1 (Figure 14). This provides the elastic modulus to determine the material's Trace, as well as provides

reference data for tensile and compressive strength. Then, by adjusting the number of plies in different orientations in section 2 (Figure 15), the compressive strength (X') and tensile strength (X) are calculated through the program and displayed in section 3 (Figure 16). The elastic modulus is calculated using the reduced stiffness matrix (Q) and applying the fraction of plies in each orientation. This then produces a predicted elastic modulus based on the orientation. To check the calibration of the program, after the 0° unidirectional T1 data was input into section 1, 8 plies of 0° was entered into section 2. The output in section 3 should equal the input in section 1, if the program is calibrated correctly. After performing this check, the program indeed produced an output that matched the input for the T1 laminate.

The other orientations were entered into section 2 and the outputs were recorded. After T2, T3, and T4, were all documented, these values were compared to the actual data produced for those laminates during tensile and compression testing. A percent difference between the QuickLam predicted values and the experimental values was calculated by taking the experimental data as the basis data and the QuickLam predicted values as objective data.

Property	
Ex (GPa)	137.80
X (MPa)	2076.00
X'(MPa)	1211.90

Figure 14. Section 1 of the QuickLam program. Ex represents the longitudinal elastic modulus. X represents the tensile strength and X' represents the compressive strength.

Laminate for the omni envelopes in stress space							
Laminate	theta 1	theta 2	theta 3	theta 4	theta 5	theta 6	# plies sublaminate
Ply angle	0	45	-45	90	-60	-60	
# plies	8	0	0	0	0	0	8

Figure 15. Section 2 of the QuickLam program. The ply angle and number of plies can be adjusted.

Laminate Strengths	
X (MPa)	2068
X' (MPa)	-1211

Figure 16. Section 3 of the QuickLam program that reports the predicted values for tensile strength (X) and compressive strength (X').

3. Results

3.1 Tensile Tests

The five tensile samples from laminate T1 were plotted on a graph of stress versus strain (Figure 17). The Shimadzu's Trapezium software offsets the curves to be more visible, so that the samples appear to not start at zero strain. The strain for the T1 samples were measured using the Strainsmart software. An example of the raw strength data was extracted from the Trapezium

software and plotted against the raw Strainsmart data to produce a stress versus strain graph where the elastic modulus could be found from the slope (Figure 18). The modulus data for the four T1 samples are shown in Table I. The T1 test coupons fractured violently, however the failure occurred within the gage length (Figure 19). This confirmed that the tensile tests followed the correct failure mechanism as stated by ASTM D3039.

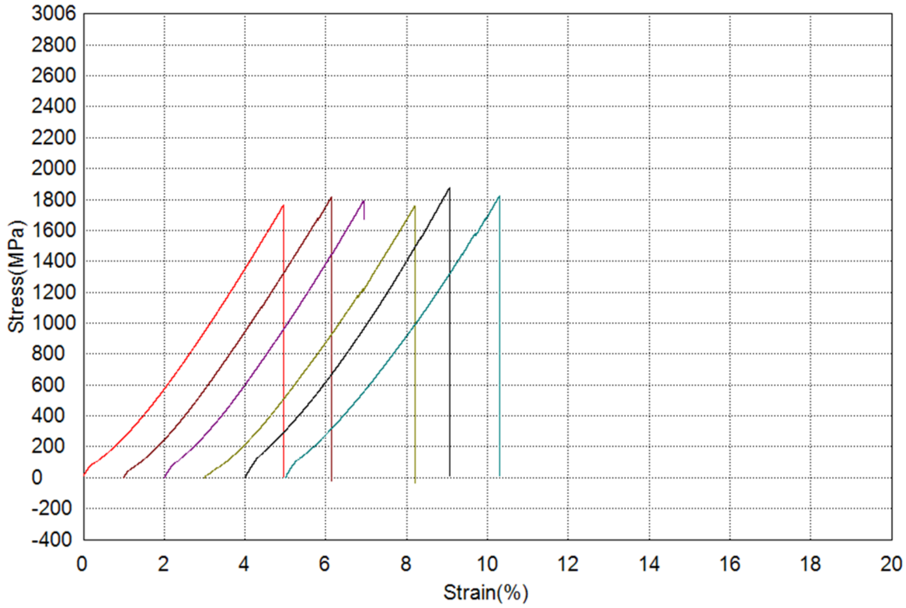

Figure 17. Stress versus strain graph for the unidirectional 0° laminate T1 plotted by the Trapezium software.

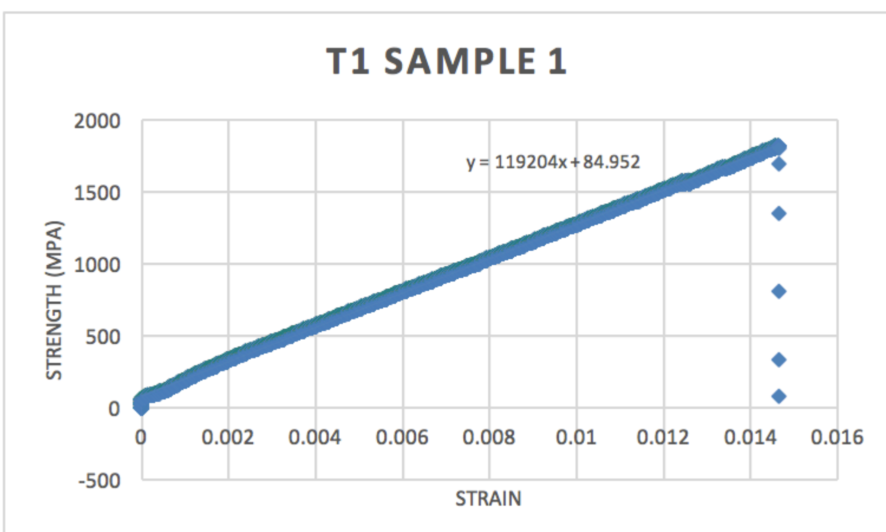

Figure 18. Raw strengh values were plotted against Strainsmart values to produce a stress versus strain plot. The slope of the line was found and used as the elastic modulus. This is a plot for sample 1 of the T1 laminate.

Table II. Modulus Data for T1 Samples

Sample Number	Modulus (GPa)
1	119.20
2	127.70
3	118.70
4	135.30
Average (GPa)	125.20
Std. Dev.	6.82
Coeff. of Var. (%)	5.45

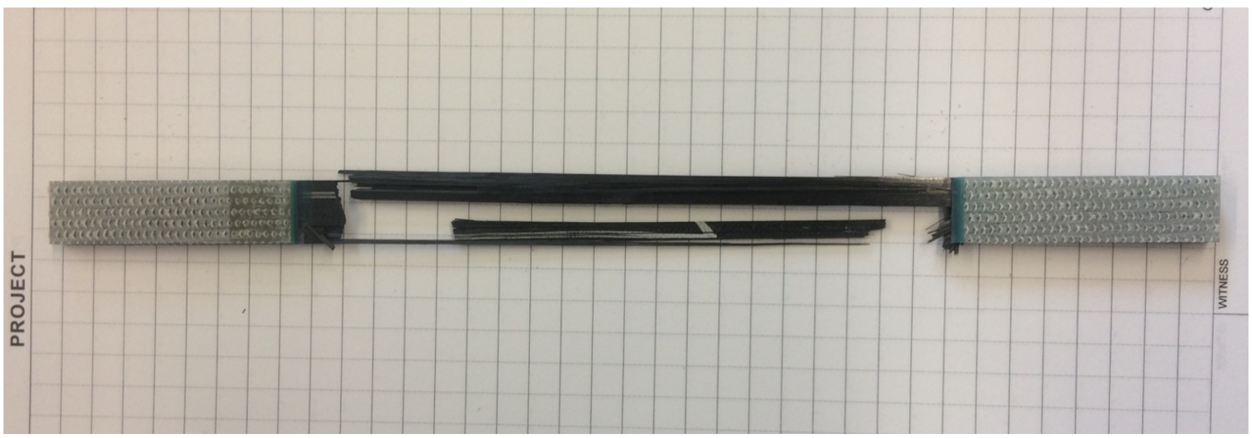

Figure 19. T1 tensile sample fractured within the gage length explosively during testing.

The Bluehill software from the Instron test frame produced stress versus strain graphs of T2, T3, and T4 (Figures 20, 21, 22). The extensometer produced the strain values of the elastic modulus for each sample. Each of the five samples from T2, T3, and T4 failed within the gage length of the coupon.

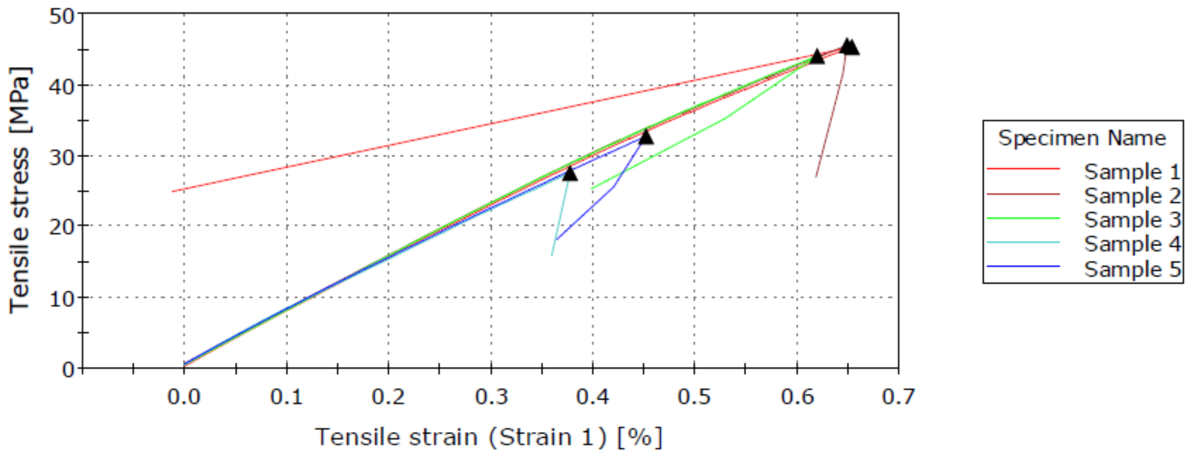

Figure 20. Stress versus strain plot for the laminate T2 reported by the Bluehill software.

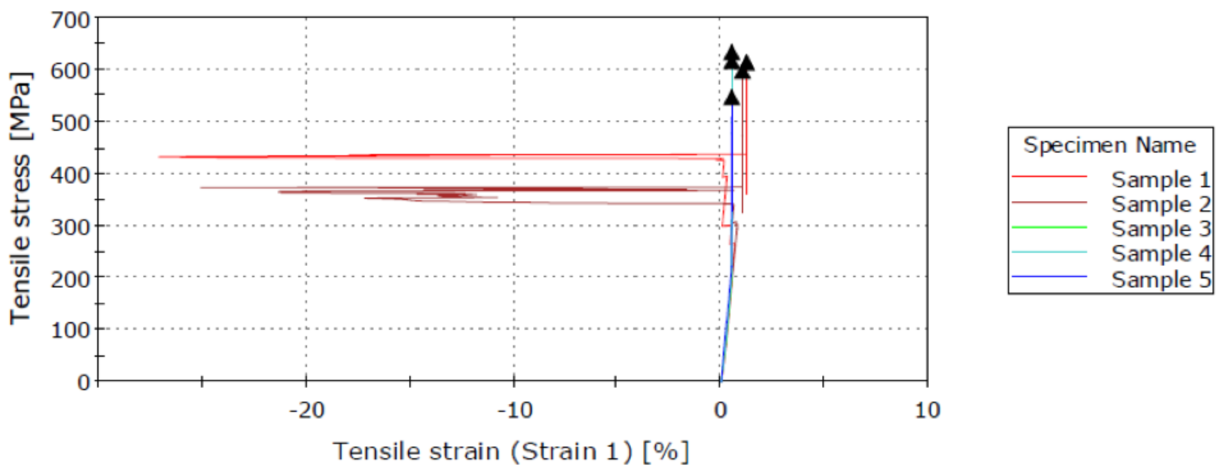

Figure 21. Stress versus strain plot for T3 laminate reported by the Bluehill program. The negative values for strain is a result of the extensometer being removed during the test.

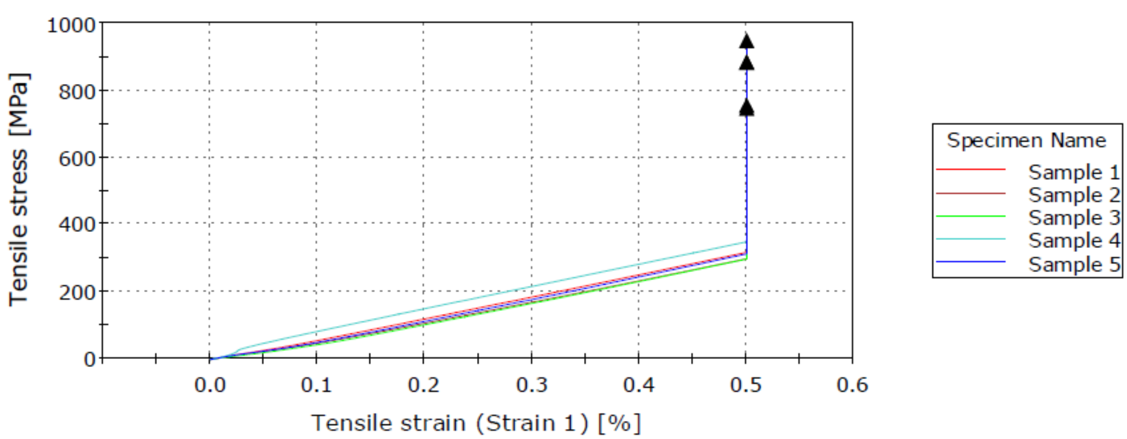

Figure 22. Stress versus strain plot for T4 laminate reported by the Bluehill software. The points where the lines move vertically up indicate that the test reached the 0.5% strain rate and the extensometer was removed.

The average load, tensile strength, elastic modulus, and standard deviation of the tensile strength values were determined and recorded in Table II. The coefficients of variance were also calculated for the strength data. Both the standard deviation and coefficient of variance were used to show that the amount of variability around the mean.

Table III. Average Values for Load, Modulus, and Strength for Four Laminates Tested in Tension

Tensile Samples					
Laminate	Avg. Max Load (kN)	Avg. Modulus (GPa)	Avg. Strength (MPa)	Std. Dev. (MPa)	Coeff. of Var. (%)
T1	28.40	125.50	1806.79	48.88	2.70
T2	2.60	7.83	39.00	7.00	17.90
T3	39.70	51.60	602.00	30.00	4.98
T4	68.30	63.71	842.84	88.36	10.48

3.2 Compression Tests

Preliminary testing was done on several laminates to ensure proper failure mechanisms during the test. A proper failure mechanism typically fractures across the gage length of the sample. During preliminary testing, however, delamination occurred under the tabs (Figure 23). After increasing the torque of the compression fixture, the coupons fractured within the gage area (Figure 23).

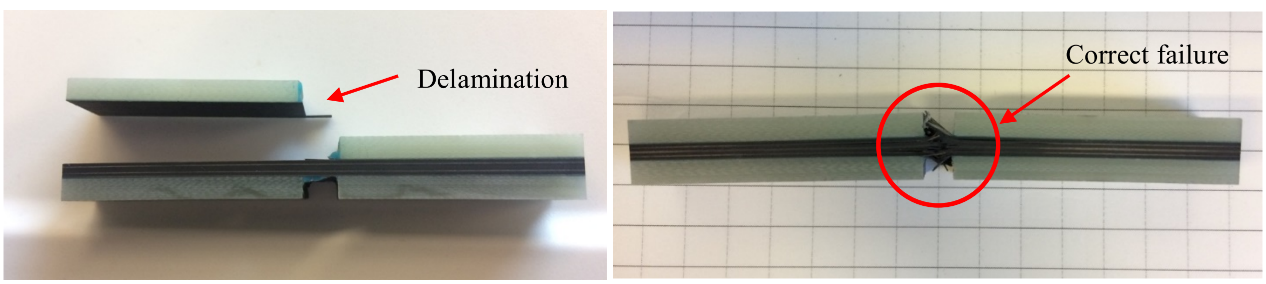

Figure 23. (Left) Delamination occurred under the tabs of the compression sample. (Right) Correct failure mode within the gage length of a compression sample.

Six compression samples were tested for each laminate orientation using standard compression testing procedures. The results were extracted from the Shimadzu's Trapezium software that produced stress versus displacement curves, along with reported maximum stress values based on the geometry of the specimen. The average maximum stresses have been compiled into Table III, along with the standard deviation and coefficient of variance of the data. The corresponding stress-displacement graphs are shown in Figures 24, 25, 26, 27.

Table IV. Average Load and Strength Values for Four Laminates Tested in Compression

Compression Samples				
Laminate	Avg. Max Load (kN)	Avg. Strength (MPa)	Std. Dev. (MPa)	Coeff. of Var. (%)
T1	22.90	1198.00	186.00	15.52
T2	8.70	236.00	30.00	12.65
T3	25.20	755.00	89.00	11.70
T4	32.00	675.00	48.00	7.11

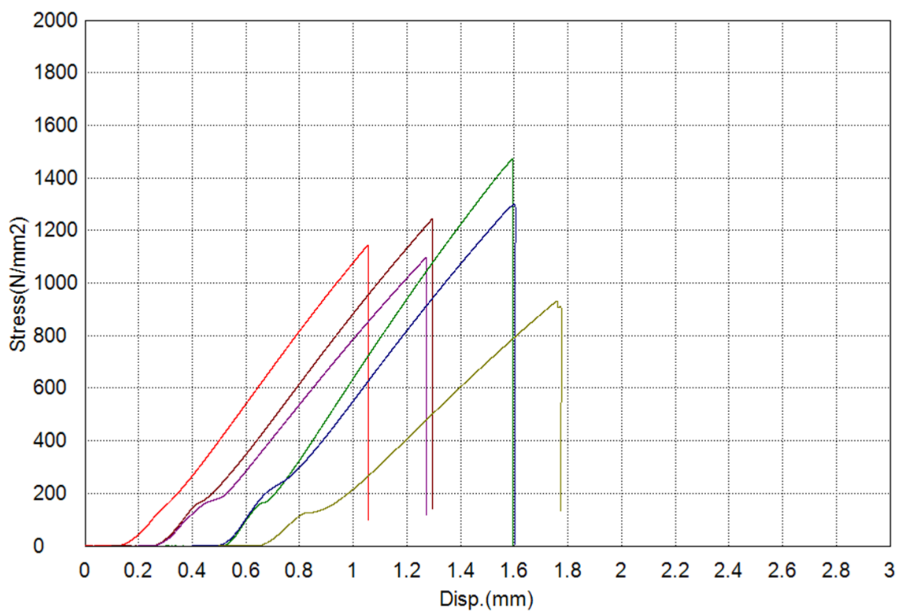

Figure 24. Stress versus displacement of the laminate T1.

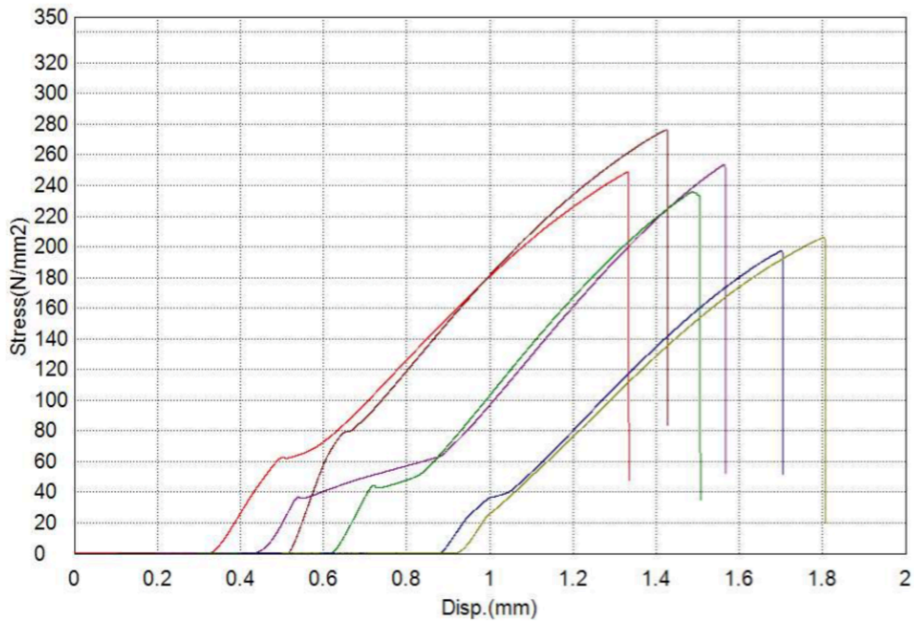

Figure 25. Stress versus displacement of the T2 laminate samples.

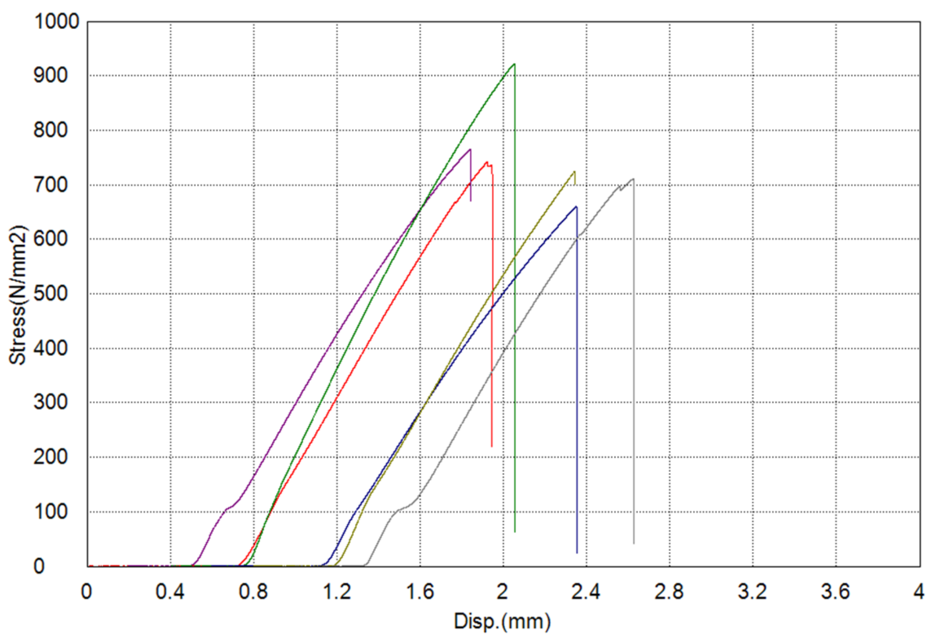

Figure 26. Stress versus displacement of the laminate T3.

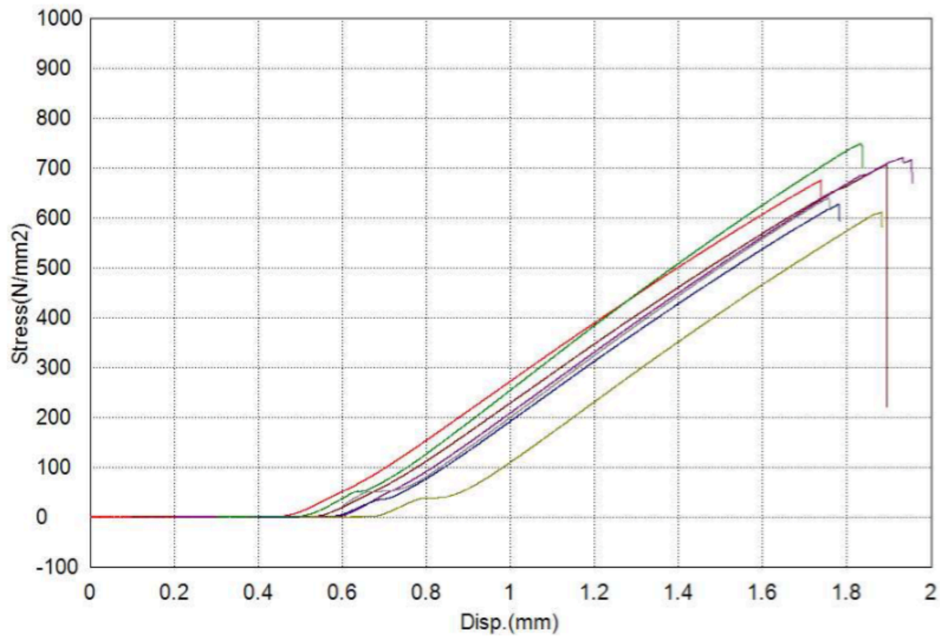

Figure 27. Stress versus displacement for T4 laminate samples.

3.3 Equivalence Testing

After the results of the tensile and compression testing for T1 were collected, it was compared to 0° tensile strength, elastic modulus, and compressive strength data provided by TenCate using the HYTEQ program. The average, standard deviation, coefficient of variance, minimum value, and maximum values of the five T1 tensile strength values were compared to a data set of 47. The T1 tensile strength values passed all four equivalence tests. Therefore, the T1 tensile strength values can be considered equivalent to data that had passed qualification according to NIAR standards. This process was also done for T1 elastic modulus data, comparing the four values calculated through Strainsmart to a data set of 47 provided by TenCate. The modulus data failed the equivalence test at α=.05 and failed the acceptance test at α=.01. The 6 values of the T1 compressive strength data were compared to a data set of 51 provided by TenCate. The data set failed both equivalency and acceptance for the mean and minimum of the data sets. After looking at the T1 data, there was one data point that fell well below the rest of the data. After consulting the ASTM D695, only 5 values are necessary for a set, so the lowest value was considered an outlier and was dropped. The new data set of 5 was compared with the qualified values, and the T1 results passed equivalency and acceptance for the mean values of the set, but failed for the minimum values of the set.

3.4 QuickLam Predicted Values

Once all the data was obtained from testing, the average elastic modulus, tensile strength, and compressive strength of the T1 samples were entered into the QuickLam program. The resulting predicted values for samples T2, T3, and T4 are shown in Table IV, along with the percent

differences. The general trend of the data set shows that the QuickLam predicted values were lower than the experimental values. Another trend within the data would be that the tensile strength values have a lower percent difference than the compressive strength data. The sample T2 shows the largest percent difference in both tensile strength and compressive strength, while the T4 (hard quasi-isotropic) laminate has the lowest difference in both values.

The Trace of the material was also determined based on the elastic modulus of the T1 sample. As stated previously, Trace is composed of three values of the stiffness matrix but can be determined from the longitudinal modulus. The Trace of this Hextow® carbon fiber TC250 laminate is 142.27 GPa.

Table V. QuickLam Predicted Values Compared to Average Experimental Values for Laminates T2, T3, and T4

Tensile Strength			
Sample	Avg. Exper. Strength (MPa)	QuickLam Strength (MPa)	Percent Difference (%)
T2	39.0	16.0	59.0
T3	602.0	546.0	9.3
T4	842.0	813.0	3.4
Compressive Strength			
Sample	Avg. Exper. Strength (MPa)	QuickLam Strength (MPa)	Percent Difference (%)
T2	236.0	11.0	95.3
T3	755.0	395.0	47.7
T4	675.0	633.0	6.2
Elastic Modulus			
Sample	Avg. Exper. Modulus (GPa)	QuickLam Modulus (GPa)	Percent Difference (%)
T2	7.8	7.4	5.1
T3	51.6	47.9	7.2
T4	73.0	73.6	0.8

4. Discussion

4.1 Equivalency Testing

The equivalency testing was performed to compare the 0° laminate data that was collected through experiments done at Cal Poly and previously qualified materials tested at TenCate Advanced Composites. The comparison was conducted to be exposed to equivalence testing used

in the aerospace and composite industry. The test was also performed to see if the means and minimums of the data were within range in order to be called "equivalent". Although the data set did not pass equivalency for compressive strength and elastic modulus, it does not discredit the data used in this project. There are many factors that could lead to not passing equivalency. It is difficult to compare a set of four samples to a data set containing 50 samples. Definitive outliers could be exposed if more tests were performed, which could be appropriately eliminated during statistical analysis. This method can also apply to the compression test data. The mean of the data set passed equivalency, however the minimum value did not. This could be due to outliers, or the use of different standard methods while running the compression tests. Another reason for the differences would be that the material used for this project had been stored in a freezer for two years before being used. Prepreg materials are stored in a freezer to prevent curing at room temperature for thermoset resin systems. The freezer life is based on the resin system and can be unique to each resin. For TC250, the freezer life is less than 12 months at <0°F.[13] By storing the material in the freezer longer than its conservative freezer life, the mechanical properties of the material may be affected.

Despite the compressive strength data and the modulus data not passing equivalency, the Trace Theory results should not be affected. The Trace of the material that predicts properties using QuickLam is dependent on base data which was part of the same batch of material. Regardless of the amount of time in the freezer, the same batch of material is used as a basis to predict values of that material. Therefore, although the data set did not pass equivalence compared to TenCate's data, the Trace Theory should still hold.

4.2 Prediction Comparisons and Trace Theory

After comparing the experimental data with the QuickLam data, certain trends were discovered. The first of these trends showed that the laminates with the most 0° plies (T4) had the most accurate predicted strength values. The more 45° and 90° plies that are in a laminate, the more influence the matrix has on the mechanical properties. While tensile strength is a fiber dominated property, compressive strength is influenced by both fiber and resin properties. Therefore, predicted values from QuickLam will be most accurate for the laminates including more 0° plies. The laminate that had the largest percent difference is T2 which has all 90° plies. This further confirms that QuickLam predictions will be the least accurate for orientations that are almost completely influenced by resin. Tensile tests and compression tests on all 90° plies, are almost completely testing the matrix rather than the carbon fiber. Therefore, it is more difficult to predict these properties using a program based on the properties of the carbon fiber in 0° laminate.

The elastic modulus had low percent differences between the QuickLam predicted values and the experimental values. Predicting tensile and compressive strength at failure is difficult for composites due to their complicated failure mechanisms. However, the elastic modulus is determined before failure in the elastic portion of the stress-strain graphs. Therefore, the modulus can be more accurately predicted based on fiber properties because the factors that contribute to complicated failure mechanisms usually occur when the laminate first begins to fail. Therefore, QuickLam can accurately predict the elastic modulus of carbon fiber composites. Elastic

modulus data for composites is more consistent than strength data in general. In industry it is common to design components based on modulus data versus critical failure data. As a result, limiting the design using elastic modulus proves to be more reliable and consistent.

Tensile strength proved to be more accurately predicted than compressive strength. Compression testing for composites is known to have more scatter and can be more unpredictable based on the testing methods used.[14] Compression testing poses many complications such as general buckling, microbuckling, and slippage within fixtures. Also, compressive strength is affected by both the fiber and matrix properties as well as the interfacial strength.[3] It is therefore more difficult to predict values for compressive strength.

It was also discovered that the predicted values were all lower values than the experimental results. QuickLam provides conservative values for tensile and compressive strengths. As stated previously, composites have complicated failure mechanisms. Composite materials experience brittle failures with little margin of safety through ductility. Therefore, the propagation of brittle failure mechanisms must be understood. However, currently there are numerous theories of understanding failure mechanisms of composites because a composite laminate can develop local failures such as matrix cracks, fiber breakage, and fiber matrix delamination under normal loading conditions. All of which are difficult to predict and therefore predicting properties based on failure mechanisms is challenging. Therefore, it is understandable that QuickLam is conservative in the predictions for strength. Even with a larger percent difference, if a project is designed to what the Trace predicted properties are, there would be no compromise in the design.

4.3 Compression Testing

Determining the compressive strength of a thin material specimen has been a long-standing challenge because the specimen tends to buckle prior to compressive failure.[15] Preliminary tests were done for compression testing to ensure correct failure mechanisms during the tests. Following the recommendation from ASTM D695, it was advised to use a torque wrench at 10 in-lb to clamp the fixture, which resulted in delamination under the tabs. After discussing this incorrect failure with industry contacts, a 15 in-lb. torque clamping the fixture obtained the correct failure within the gage area. Although increasing the torque seems to be intuitive, a low clamping force was recommended by the standard. Because the thickness varied with the different laminate orientations, it is reasonable to believe that the results of compression testing may vary. If a thicker specimen is used, the gage length should be increased. However, end-crushing and tab delamination would present additional problems. Instead, tightening of the fixture accommodated for the thicker specimens, which prevented the tabs from slipping during the test.[15] Additionally, the standardized geometry and tabbed ends forced the samples to have a limited gage length, which made it more difficult to obtain a fracture within the gage length of the samples.

As previously stated the compressive strengths of the laminates were considerably off from the predicted QuickLam values. Discrepancies between the two sets are likely due to the use of the ASTM D695. The standard poses many complications that could potentially compromise the results.

Although the greater torque applied was successful in obtaining the correct failure mechanism in the experiment, it did not follow the procedure set out by the standard. This may have jeopardized the accuracy of the results, especially with the compressive strength from the unidirectional 90° samples (T2). The popularity of ASTM D695 has decreased considerably since the introduction of ASTM D6641, the Combined Loading Compression method.[16] This new standard has shown to report more reliable results from testing with fewer complications. ASTM D695 is slowly being removed from testing procedures in industry because of the inconsistencies of the reported results.[16] Compression testing for composites has proven to be complicated, and many methods are becoming outdated as the industry evolves and advances.

5. Conclusions

1. Trace provides conservative values for strength, and can therefore provide a more efficient way of coupon testing for material qualification for the aerospace industry without compromising the design.
2. The Trace Theory is most accurate for predicting properties for laminates that are more dependent on fiber properties and less dependent on matrix properties.
3. Experimental data for compressive strengths showed more scatter than for tensile strengths. Compression testing produced a larger standard deviation and less reliable results. This could be due to the complications from ASTM D695 and difficulties in reproducing compression failure modes.

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