Fiber Reinforced Silicone

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Abstract:
This thesis revolves around reinforcement of silicone, fibers and rule of mixtures. Rule of mixture serves as a mean to anticipate properties of a composite made up of continuous or unidirectional fibers. However, it doesn't tell anything about short or particle fibers. The main objective of this thesis project is to investigate if the rule of mixtures is valid for short/particle fibers. Mold silicone 1540A and mold silicone 1540B mixed in the ratio of 1:1 and the mix were used as the matrix. Carbon fiber, glass fiber and cellulose fibers were used as reinforcements. Moulds for tensile test specimen and tear test specimen were designed using inkscape software according to standards ASTM D3039 and ASTM D624-C respectively. Five specimens of each fiber for tensile test and two specimens of each fiber for tear test were made of different fiber volume fractions of each silicone-fiber mix. Silicone-carbon specimens had fiber volume fraction ranging from 0%-17.8%, silicone-glass specimen had fiber volume fraction ranging from 0%-20.6% and silicone-cellulose specimen had fiber volume fraction ranging from 0%-27%. A general equation was derived from rule of mixtures combining upper and lower limit as $E_{experimental}^{p} = f\, E_{f}^{p} + (1 - f)\, E_{m}^{p}$ where 'p' is reinforcement factor and $-1 \leq p \leq 1\,, p \neq 0$. This equation was then used to find the p-value of each type of fiber. Carbon fiber was found to have the highest p-value of 0.3, glass fiber of 0.075 and cellulose fiber with the lowest of -0.5. Microscopic analysis of each fibers was done to examine what factors affected the p-value and it was observed carbon fiber had very less fibers in the range of 20-40 microns, most in the range of 100-120 microns and some in the range of 180 microns and above.
Glass fiber was observed to have most fibers in the range of 20-40 microns while some in 100 microns-above. Cellulose fiber whereas had most fibers in the range of 60-80 microns and very less fibers of higher length.

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FOREWORD

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29th March, 2020
Helsinki

Bhandari Amar.

1 INTRODUCTION

This thesis is about reinforcing silicone, a synthetic rubber, with addition of fiber to attain desired mechanical properties using rule of mixtures for mathematical modelling. Silicone based products has been used along the technological civilization. In the early 1940s, silicones were being researched upon by the US to help with the war effort. Then the first ever silicone-based product was produced which was in a gel form and was used to protect electrical sparking equipment's in airplanes. In the 50s silicone-based leather system was launched and during the 60s Neil Armstrong wore boots made up of silicone to leave footprints on the moon. During the 70s Liquid Silicone Rubber was developed. In the 80s silicone played a vital role in development of microprocessor-based technologies by protecting the products exposed to extreme overheating. [1]

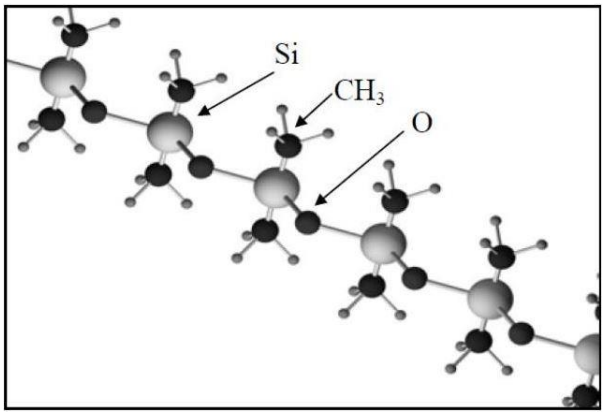

Figure 1: Molecular structure of silicone [2]

The above figure shows the structure of silicone polymer (polydimethylsiloxane). The polar main chain is shielded with the nonpolar methyl groups which can rotate freely around the silicon-oxygen chain. This permits the shear motion of the particles and gives it the elastomeric properties. [3]

Silicone rubber shows excellent resistance at both high and low temperatures, which allows it to be used on a large temperature range. However, it being in semi-liquid state leads to very poor mechanical properties compared to other synthetic rubber counterparts. [4] The shore hardness of silicone parts can range from 30 shore A durometer to 80 shore A durometer which allows silicone to be used for various areas with desired hardness. [5]

Although this elastomer shows promising mechanical properties without being mixed with any other components, the properties still need to be modified to some extent to use them for production. To obtain adequate tensile properties, elastomers are reinforced with other materials. Carbon black and fibers are some reinforcing materials used for reinforcement of elastomers. The reinforcement might both be physical and chemical. Difference in reinforcing material and their properties result in different reinforced rubber. The reinforced rubber might have change in tensile properties, modulus, hardness, abrasion resistance, tear strength, electrical conductivity, resilience and flex-fatigue life. [6]

A variety of rubber are reinforced with reinforcing materials in industries to produce products with better mechanical properties. The reinforcement can be commonly seen in tires and hoses. Hoses are made of 3 layers; the inner, the reinforcement and the cover. The inner and outer layer are made of synthetic rubber and the reinforcement allows the hose to be resistant to abrasion, corrosion, oils, weather, ozone, aging, cut and sunlight. [7]

Test samples of reinforced material are produced by casting. A mold of desired test sample is designed and produced which is then used to produced test samples. The liquid mix is poured in the mold with the desired hollow shape and allowed to solidify. The cast is then taken out of the mold and then can be undertaken to various tests to analyze desired properties.

American Society for Testing and Materials (ASTM) specification of rubber standards specify the tests and the assessment of the physical, mechanical and chemical properties of a range of materials and products of rubber and its elastomeric derivatives. The standards set allows manufacturers and consumers to evaluate and examine their material to ensure quality and safe utilization.

The test methods are designated with alphabets and numbers. There are standards set for everything that rubber could be used for. The analytical test methods for rubber by ASTM are designated with ASTM F2466 – 10(2018) and ASTM F2524 – 06(2013). ASTM F2466 – 10(2018) is the standard practice for the determination of silicone volatiles in silicone rubber for transport applications while ASTM F2524 – 06(2013) is the standard practice for determination of volatile content for formed-in-place gaskets silicone adhesives and sealants for transport applications. [8]

Reinforced rubber can be tested as per other ASTM standards. Mixing can be done according to ASTM designation D15-70. Tensile testing of reinforced rubber by ASTM are

designated by D 412-51 T, using dumb bell specimens. ASTM designation D 395-61A is a standard test for the determination of tensile properties, elongation at break, tear and compression tests. Hardness can be measured according to the ASTM standard D 676-52T. [9, pp. 338-340]

1.1 Objectives (hypothesis)

- ***To modify silicone mechanical properties using carbon fiber, glass fiber and silicone fiber with the purpose to increase E, sigma, …***
- ***To establish if the rules of mixtures is valid for short or particle fibers***
- ***To test to change in compressive and tensile modulus due to reinforcement***
- ***To study the change in strength due to reinforcement (types: short or long fibers)***

1.2 Methods

Composites are formed by comprising two distinct components: fibers and matrix (often polymer resin). These components when mixed remain discrete but interact to form a completely new material which offer completely different properties than its raw components. For example, thin glass fibers exhibit high tensile strength but are likely to be damaged in contrast of which most of the resins are quite tough in nature but provide low tensile strength. These two fiber and matrix components when combined result in a material far more useful than either of the components with improved mechanical properties. [10] This thesis is focusing on reinforcing silicone (matrix) using carbon fiber (fiber) to see the changes in compressive and tensile properties of silicone before and after reinforcement.

1.2.1 Mechanical Tests

Mechanical tests are done to find the tensile, compressive, and various mechanical properties of the composite. These tests are done using Universal Testing Machine (UTM). The graphs produced by these machines under testing are used to find necessary properties of the specimen.

1.2.2 Compression set of Elastomeric Materials

This test is performed to measure the ability of an elastomer to return to its original thickness under certain temperature and deflection for prolonged amount of time. Under a compressive stress over long period of time a rubber material tends to lose its ability to return to its original thickness. This resiliency loss might affect the performance of elastomer as seal or cushioning pad used for longer period. Compression set results are expressed in percentage. Lower percentage in compression set means higher resistance to permanent deformation under given temperature range and deformation. An elastomer with less than 20% compression set is considered excellent and has reasonable compression results. [11]

1.2.3 Abrasion Test

Abrasion testers are used to test wear resistance of rubber in everyday wear. DIN Abrasion Tester H015 is used to perform these tests. It uses sandpaper to induce wear environment. The sample is loaded on the machine and using precise parameters the sample is moved under friction with the sandpaper. The samples are then taken off and weighted to calculate the loss of material due to wear. The wear resistance is the determined by comparing the test results alongside the data of comparison compound. [12]

1.3 Sustainability- earlier studies- industrial relevance

Silicone seems to be more beneficial to environment compared to plastics. It has a wide variety of applications and lasts much longer than plastics products do. One of the best areas of silicone uses is production of kitchenware because it is comparatively more inert than plastic which prohibits the chances of leaching chemical into the food during food

storage. [13] Furthermore, its wide variety of properties enables it to be used in various industries. It is widely being used in Aviation and Aerospace, as a composite reinforced with additives, for assembly and maintenance; in construction as sealants, adhesives and coatings; in electronics to protect components from extreme heat and moisture; in healthcare as drug delivery systems or pacemakers; household coatings etc. [14]

While recycling there is nothing prohibiting chemically from silicone being recycled but recycling programs rarely accept it and it can be hard to get silicone post-consumer products to silicone-recycler. This problem occurs because most consumer confuse polyurethane with silicone. [13] The specialized silicone recycling companies downcycle the silicone waste to lubricant used for machine in the industries. [15]

One of the key objectives of this thesis is to improve mechanical properties of the silicone which will help it last longer while developed into a product. With a long-lasting product and material, it would need less replacement which will eventually reduce the use of silicone itself.

2 LITERATURE REVIEW

2.1 Silicone

Silicones are made up of long repeating chain of monomers called siloxane which is a functional group in organosilicon chemistry with Si-O-Si linkage. Depending on the organic groups attached to the silicon by C-Si bonds, different types of silicones are formed. The backbone structure of silicone is governed by -Si-O- bond unlike Carbon-Carbon and Carbon-Hydrogen bond in plastics. Despite silicon and carbon being in the same group in the modern periodic table, they have very distinct properties.

Siloxanes are formed by the presence of oxygen atoms in the silicon chain along with an alkane. Because it has an inorganic polymer backbone and an organic secondary structure, their chemical and mechanical properties are halfway between the inorganic and organic compounds, producing an elastomeric material with promising mechanical properties. [16]

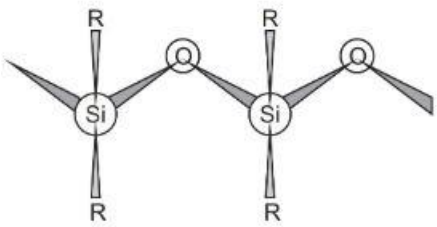

Figure 2: Structure of Silicone [17]

With different organic group connected to -Si-O-Si- in the siloxane chain different types of silicones can be formed. A siloxane chain has phenyl group as organic compound, the resulting silicone will be called poly(diphenylsiloxane).

While a various organic group can be added to make different kinds of silicone the most widely used silicones are the ones with methyl group along the backbone, which can also be called as poly(dimethylsiloxane). Properties of the silicone can be altered by substituting methyl groups by other organic compounds. [17]

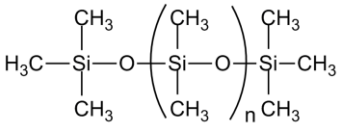

Figure 3: Poly(dimethylsiloxane) [17]

The chemical structure of silicone provides it a greater flexibility with the forms in which it exists. It can exist in the form of fluids, gels, rubber and resin. The physical form in which the silicone exists is influenced not only by the structure of the polymer but also depends upon whether it is long or short chained molecule, as a cross linked species or a three dimensional network and also the organic group attached to the Si-O framework.

- **Silicone fluids**

  They basically are repeating structure of straight chains of poly(dimethylsiloxane) with usually having trimethylsilyl groups, $Si(CH_3)_3$ at the end of each chain.

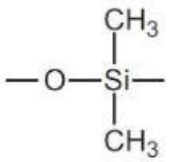

Figure 4: Repeating structure of poly(dimethylsiloxane) in silicone fluids [17]

  The silicone fluids are highly resistant to temperature and thermal oxidation. They show excellent resistance to temperatures ranging from -60 ^{o}C to +300 ^{o}C. They even so excellent stability towards electromagnetic and particle radiation (alpha, beta, gamma and UV radiation). Low volatility, excellent shear resistance, low surface tension and water repellency are other characteristics that silicone fluids have. They are transparent, tasteless and odorless and possess no harmful effects on human health. [18]

- **Silicone gels**

  These are also based on poly(dimethylsiloxane) chains but also with a few cross-links between the chains and as an open three-dimensional network. Silicone fluid is poured in a mould together with a reactive group under the influence of heat or catalyst to induce crosslinking between the polymer chains to produce silicone

gels. They have similar properties to that to silicone fluids and have a wide range of applications. They are mostly used in cosmetics as a topical formulation of scar tissues forming a barrier to keep the skin hydrated. They have been widely used as implants in human bodies as they do not possess any harm chemically. [19]

- **Silicone elastomers (rubbers)**

  Silicone elastomers are the result of introducing even more cross-linking into the linear chain polymers. This can be done in two main ways: adding curing agent at room temperature (RT vulcanizing) or adding a peroxide at an increased temperature (HT vulcanizing) High Temperature Vulcanizing.

  In RT vulcanizing, in contact with the moisture in the air the material starts cross-linking right away. A small amount of acetic acid, amine or neutral by products are released depending on the nature of curing agent. They show elastomeric properties as natural rubber and can retain their original properties to temperature over 500 K to temperatures below 250 K.

  In HT vulcanizing silicone is cured with organic peroxides or platinum catalyst at an increased temperature. The HT vulcanized silicone contains long polymer chains with high molecular weight. This results in them having outstanding mechanical properties and can be applied to temperatures ranging from 200K to 500K. [20]

- **Silicone resin**

  These are formed by branched, cage like oligosiloxanes. The general formula of these resins is $R_nSiX_mO_y$. Where R (a non-reactive substituent) mostly Ph or Me, X (functional group) H, OH, Cl or OR.

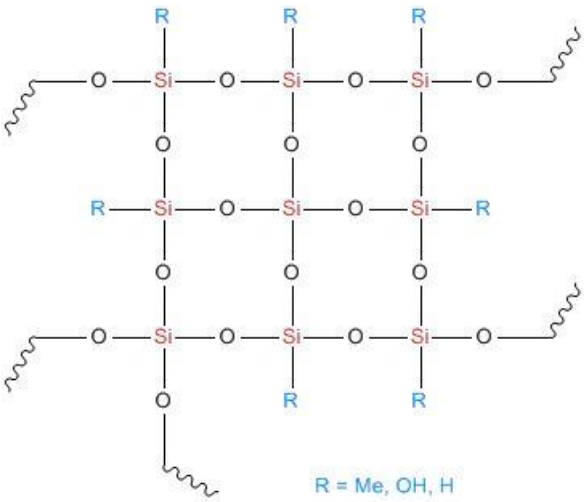

Figure 5: Structure of silicone resin [21]

These have a various range of applications and have properties like heat resistance, UV resistance, hardness, water repellency and dielectric properties. [21]

2.2 Fibrous additives

Fiber reinforced composite are now the premium choice of production in many industries during product development. Despite composite being hindered by their non-isotropic nature which makes them hard to work with during modelling and simulation, the physical properties combined with the unbeatable lightweight makes them undeniably attractive compared to materials like steel, aluminum, iron and titanium.

Glass fibers

Glass fibers are the most used fiber additive in the composite industry. They are commonly used in most end-market applications to replace heavier metal parts. They are commonly used in reinforcing polyester, epoxy and phenolic resins. Moisture has an negative effect on the strength of glass fiber and they are also susceptible to static fatigue i.e. they can undergo subcritical crack growth when subjected to a constant load for an extended period of time. [22] Although it weighs more than that of carbon fiber and is not as stiff as carbon fiber, it has more elongation at break and is more impact resistant. The type of the fiber, diameter of the filament, coating chemistry and the fiber form effect on the range of properties and the performance of the composite. They have a tensile strength of 1750 MPa, Young' modulus of 70 GPa and the coefficient of thermal expansion is $4.7\times10^{-6}\ K^{-1}$. [10]

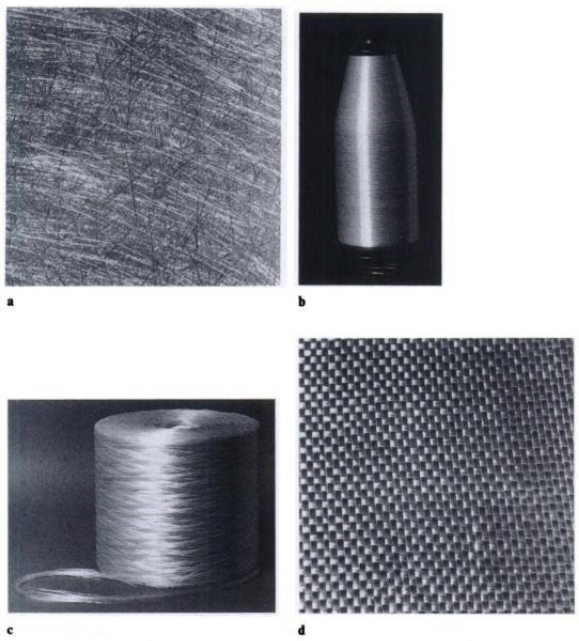

Figure 6: Glass fiber in a variety of its forms: a. chopped strand, b. continuous yarn, c. roving, d. fabric [22]

High-performance Fibers

These fibers are used in producing advanced composites and include carbon fiber, boron fibers, aramid fiber, high modulus polyethylene (PE) and hybrid combinations.

Carbon fibers are mostly used in high performance applications. They are exceptionally light with a density of just 2.268 g cm^{-3}. It is made from precursors such as polyacrylonitrile (PAN), rayon and pitch. High strength fibers are then created by chemically treating, heating and stretching and then carbonizing the precursors. Based on the heat treatment they are classified into 3 types. Type-I, where the final heat treatment temperature reaches above 2000 ^{o}C. They are also known as high-heat-treatment carbon fibers (HTT) and offer very high modulus. Type-II, where final heat treatment temperature should be 1500 ^{o}C or above. They are also known as intermediate-heat-treatment carbon fibers (IHT) and offer very high strength. Type-III, where final heat treatment temperature should not exceed 1000 ^{o}C. They are called low-heat-treatment carbon fibers and have low modulus and low strength. At first rayon based high performance carbon fibers were made but now it is replaced by PAN and pitch-based fibers in most applications. They have an excellent range of properties including excellent strength (upto 1000 ksi), high stiffness, low to negative axial coefficient of thermal expansion. They also offer biological inertness and x-ray permeability which allows them to have medical applications. They are chemically inert and provide high corrosion resistance. [23]Although having greater strength as compared to glass and aramid fibers, they are less impact resistant and experience galvanic corrosion in contact with metal. However, galvanic corrosion can be solved by using a barrier material (often fiber glass or epoxy) during laminate layup. [10]

Figure 7: Milled and chopped reclaimed carbon fiber [24]

Aramid fibers are another type of fibers used in composites. They are formed from aromatic polyamide. They offer exceptional impact resistance and elongation higher than carbon fibers but less than glass fibers. The standard high-performance aramid fiber has tensile strength of about 500 ksi, elongation of about 3% and modulus of about 20 Msi. They show good resistance to organic solvents, are nonconductive, have low flammability and good fabric integrity at elevated temperatures. With the high impact resistance, the demand of aramid fibers is quite high in developing armor and ballistic applications.

Figure 8: Aramid fiber and its forms [25]

Boron fibers provide exceptional strength. They are twice as stiff and five times as strong as steel. [10] Along with the exceptional strength boron fibers provide stiffness and light weight, and possess excellent bucking and compressive resistance. It has an average tensile strength of 3-4 GPa and Young’s modulus is between 380-400 GPa. [22]

2.3 The rule of mixtures

Rule of mixtures, in materials science, is a weighted mean used to anticipate properties of a composite made up of continuous or unidirectional fibers.

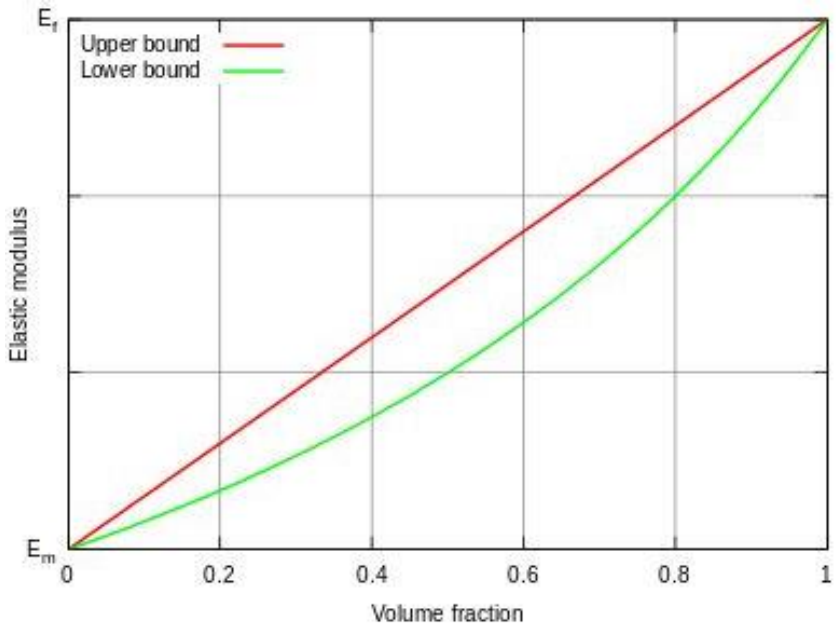

Figure 9: Projected upper and lower bound on the elastic modulus of a composite material [26]

The rule of mixtures provides the theoretical lower and upper bound on elastic modulus, ultimate tensile strength, mass density, thermal conductivity and electrical conductivity like properties of the composite material. There are two models based on loadings, Voigt model (axial loading) and Reuss model (transverse loading).

In direction parallel, the rule of mixture states, the overall property may be as high as [26]

$$E_c = f\,E_f + (1 - f)\,E_m \qquad \text{(Equation 1)}$$

Where;

- E_f (Young's modulus of the fibers)
- E_m (Young's modulus of the matrix)
- $f = \frac{V_f}{V_f + V_m}$ is the fiber volume fraction

It can also be known as upper bound modulus and corresponds to loading parallel to the fibers.

In the direction perpendicular to the fibers, the inverse rule of mixture states, the elastic modulus can be as low as [26]

$$E_c = \left(\frac{f}{E_f} + \frac{1-f}{E_m} \right)^{-1} \qquad \text{(Equation 2)}$$

Where;

- E_f (Young's modulus of the fibers)
- E_m (Young's modulus of the matrix)
- $f = \left(\frac{V_f}{V_f + V_m} \right)$ is the fiber volume fraction

  The elastic modulus acquired is also called the lower bound modulus and corresponds to transverse loading.

2.3.1 Derivation of upper bound modulus

Considering a composite material under uniaxial tension (σ_∞) for the material to stay intact the strain on the matrix, ϵ_m must equal to the strain of the fibers, ϵ_f. Under uniaxial tension Hooke's law gives [26]

$$\frac{\sigma_f}{E_f} = \epsilon_f = \epsilon_m = \frac{\sigma_m}{E_m} \qquad \text{(Equation 3)}$$

Where;

- $\sigma_\mathrm{f} =$ stress of the fibers
- $\mathrm{E_f} =$ elastic modulus of the fibers
- $\sigma_\mathrm{m} = stress\ of\ the\ matrix$
- $\mathrm{E_m} =$ elastic modulus of the matrix

Considering stress to be force per unit area, a force balance gives [26]

$$\sigma_\infty = f\sigma_\mathrm{f} + (1-f)\,\sigma_m \qquad \text{(Equation 4)}$$

Where;

- $\mathrm{f} =$ volume fraction of fibers
- $(1-\mathrm{f}) =$ volume fraction of the matrix

Abiding Hooke's law $\sigma_\infty = E_c\epsilon_c$, and considering that the composite material behaves as a linear-elastic material; for some elastic modulus of the composite E_c and some strain of the composite ϵ_c, the equation3 and equation 4 can be combined to give [26]

$$E_c\epsilon_c = f\ \mathrm{E_f}\epsilon_f + (1-f)\mathrm{E_m}\epsilon_m$$

Ultimately, since $\epsilon_c = \epsilon_f = \epsilon_m$, the elastic modulus of the composite can be expressed as [26]

$$E_c = f\ E_f + (1-f)\ E_m \qquad \text{(Equation 5)}$$

2.3.2 Derivation of Lower-bound modulus

In this case the composite material is loaded perpendicular to the fibers. By assuming $\sigma_\infty = \sigma_f = \sigma_m$, the overall distribution of the strain between the materials in the composite is such that [26]

$$\epsilon_c = f\ \epsilon_f + (1-f)\ \epsilon_m$$

The overall modulus of the material then can be given by [26]

$$E_c = \frac{\sigma_\infty}{\epsilon_c} = \left(\frac{\sigma_f}{f\ \epsilon_f + (1-f)\ \epsilon_m}\right) = \left(\frac{f}{E_f} + \frac{1-f}{E_m}\right)^{-1} \qquad \text{(Equation 6)}$$

Since $\sigma_f = E\epsilon_f$ and $\sigma_m = E\epsilon_m$

2.4 The method of casting

Casting is simply a manufacturing process where a mold with a hollow cavity of desired shape is filled with liquid material and then allowed to solidify. The casting (solidified part) is then taken out of the mold to complete the process. The materials that are used to produce casting by this process are metallic materials and other materials like concrete, epoxy, silicone etc. which cure after mixing two components together. Its easy and cheap to produce complex shapes using this method thus it is often chosen instead of other methods when it comes to produce complex shapes. Casting can be further sub divided into two types: metal casting and plastic, concrete, or resin casting. [27]

Resin casting is a method of casting where mold is filled with resin and let it cure. This casting method is generally used while producing small prototypes or test specimens. Single monomer resins might as well be used in this process with a curing to form homo-polymers. The curing agent contains a catalyst or is technically a source of free radicals and causes a free-radical chemical chain polymerization acting as an initiator. In contrast, resin casting can as well be done by using a nearly equal amount of hardener with the resin, which contains a functional second polymer which helps in forming a final product plastic copolymer. [28]

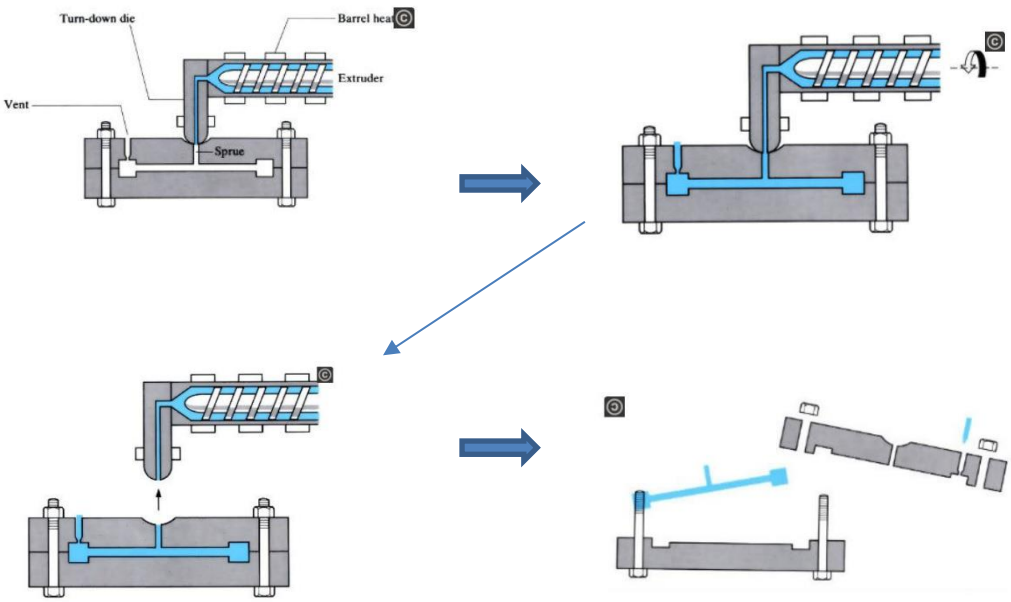

Figure 10: Melt Casting [48]

2.5 Universal testing machine

Universal Testing Machines (UTMs) are basically used in testing mechanical properties of a specimen. Most commonly UTMs are used to test tensile properties, compression properties and bending properties. The test specimen is put in the UTM with right apparatus for the kind of test that is to be done. The UTM measures the Force and deflection in Newtons and mm respectively. After the test the data and graphs collected by the computer is used to calculate and analyze the properties of the test specimen. There are other UTMs which have their own specialized names because of the marketing to a specific sector such as “top load compressor tester” for packaging and pipes, “peel tester” for adhesives, labels and tapes, “texture analyzer” for food. An UTM is a multipurpose equipment in quality control, research and development of material and products. [29]

2.5.1 Tensile test

To perform this test, the test specimen is clamped on each of its ends and pulled apart until it breaks. This test measures the tensile strength, break strength, yield strength and the tensile modulus of the specimen. The test helps to ensure product safety and integrity and helps to make certain of consistency during manufacture and assembly. The test is done under ASTM standards by designation C1341-13, C1368-10, C907-03, D1414-15 etc. The types of tensile tests are elongation and recovery, grab method and strip method. [30]

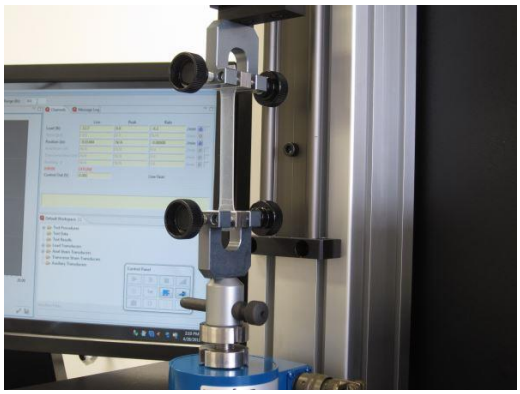

Figure 11: UTM performing a tensile test [29]

2.5.2 Compression Test

To perform this test, the test specimen is put in-between two-level plates and a certain load is applied until a certain distance is reached or the specimen breaks. The test measures from this test is the compressive force or the maximum force sustained before breakage. During product development this helps assure the quality and the performance of the finished products. The compression tests are done following the ASTM standards ASTM D6108-19, ASTM D6484/ D6484-14, ASTM D6742/D6742M-17 etc. The types of tests that can be done under compression tests are; box crush (BCT), cleavage strength, compressive hardness, edge crush test (ECT), compressibility and recovery etc. [31]

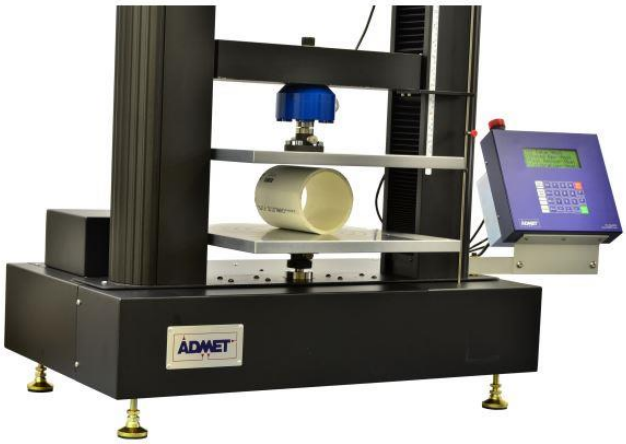

Figure 12:UTM performing a compression test [29]

2.5.3 Peel Test

This test is done to assess the bond quality to determine the adhesion strength of the material generally in cases where thin film is deposited on the substrate. This helps analyze the tension characteristics between the adhesive and adherent surfaces. The test is very useful in accessing the effects of surface treatment or performance of the material in real life such as environmental exposure. The peel tests are done following the ASTM standards by designation D3330-04, C297, C393, D6252/D6252M-98 etc. The types of peel tests are; 135-degree peel, 180-degree peel, 90-degree peel, climbing drum peel, floating roller peel, peel wheel test and T-peel. [32]

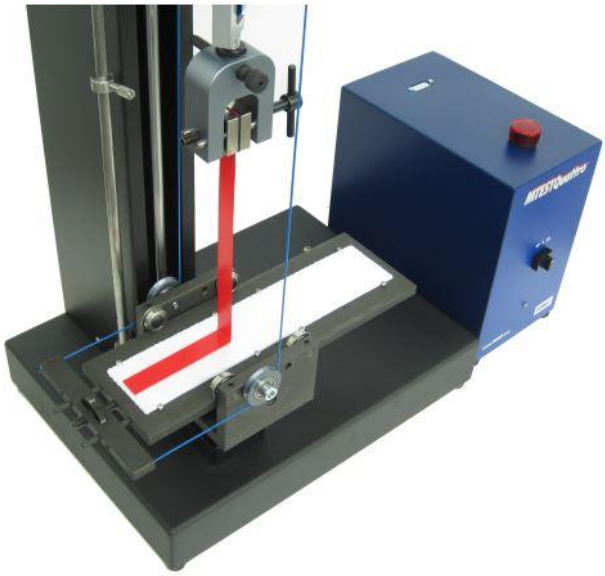

Figure 13: UTM performing a peel test [29]

2.5.4 Bend Test

This is a type of compression set but in this test the test specimen lies horizontally and is supported by two supports on each end while there is no support in the middle at all. The machine presses the specimen from above until it breaks or reaches a specific distance. [29] This test helps to analyze the flexural strength and the flexural modulus of the test specimen. The ASTM standards to do the bending test are of designation C1161-02C, C1341-13, C1684-08, C203-05A etc. [33]

Figure 14: UTM perfoming a bending test [29]

2.5.5 Puncture Test

This test is similar to compression test but instead of compression of the material a penetrometer probe is passed through the specimen being tested. This test can be performed in a wide range of products and helps analyzing the energy required to rupture or deform the material. The force at which the product breaks is called the puncture resistance. [29]

2

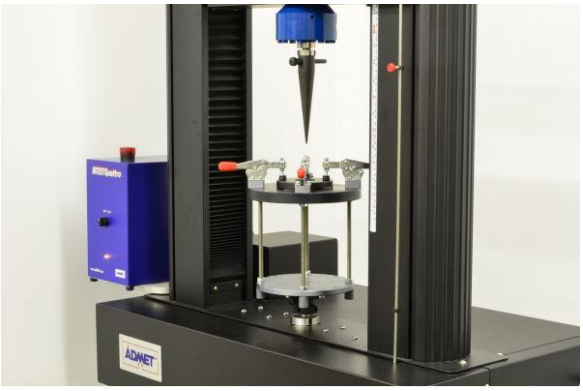

Figure 15: UTM performing a puncture test [29]

2.5.6 Tear test

In this testing method a force is applied to a material that already contains a partial fracture to bring the material to complete fracture. The purpose of this test is to test how much the material can resist the grown of the initial tear and calculate its tear resistance. [34] The ASTM standards to do the bending test are of designation ASTM D1424, ASTM D689 etc.

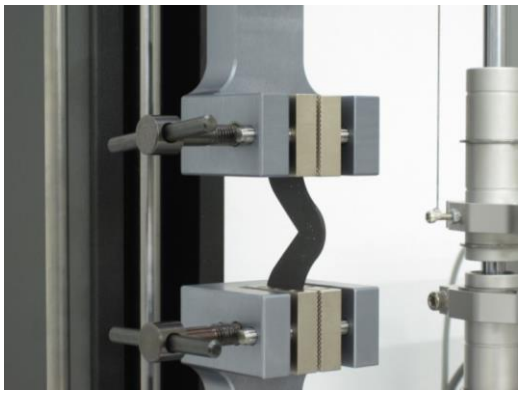

Figure 16: Die C specimen in Tensile grips [35]

3 EXPERIMENTS

This chapter contains information about the preparation of the casting mould, specimen geometry material preparation, injection of the specimen into the mould, specimen curing, specimen unloading from the mould, mechanical testing of the specimen and analysis of the data form the tests.

3.1 Materials and Equipment Used

a) *Mold silicone 1540A and Mold silicone 1540B (from Kevra Oy)*

   Mold Silicone is high tensile additive crosslinking RTV silicone. Two components of mold silicone 1540 were used. The mixing ratio of component A and component B is 1:1 and they cure at room temperature.

Figure 17: Mold silicone 1540 A (on the left) and mold silicone 1540 B (on the right)

b) *Glass fibers*

c) *Carbon fibers*

d) *Cellulose*

e) *Casting molds*

   i. *Mold for teartest specimen*

      The top, front and the bottom layer were designed in the inkscape software. The middle layer has a cut-out of tear test sample according to the standard ASTM D624-00(2012), the top layer has the inlet and the outlet, and the bottom layer is plain.

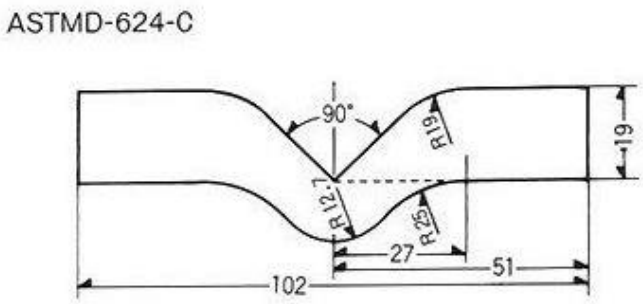

Figure 18: ASTM D624-C tear test specimen dimensions [36]

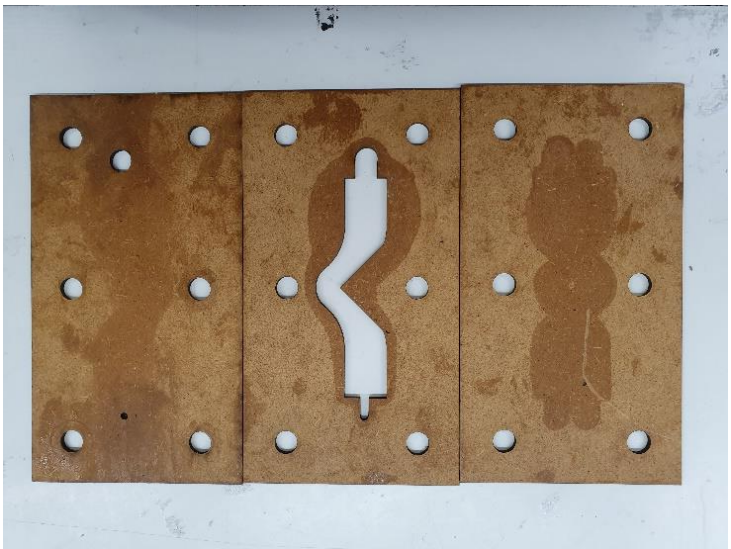

Figure 19: Cast for tear test specimen; top layer(left), middle layer(middle) and bottom layer(right)

ii. Mold for tensile specimen

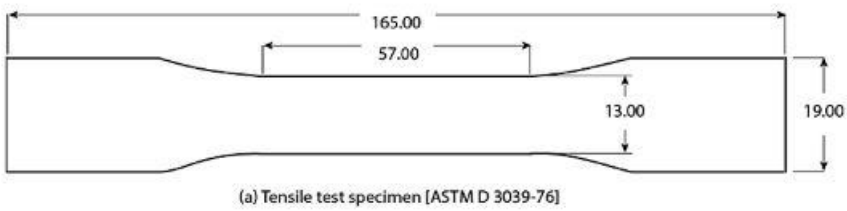

Figure 20: Tensile test specimen ASTM D3039 [37]

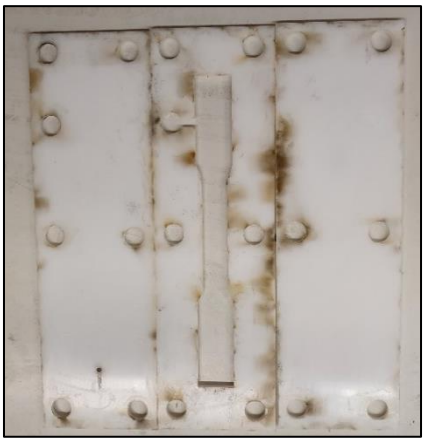

Figure 21:Cast for tensile test specimen; top layer(left), middle layer(middle) and bottom layer(right)

The top, front and the bottom layer were designed in the inkscape software. The middle layer has a cut-out of tensile test sample according to the standard ASTM D3039, the top layer has the inlet and the outlet, and the bottom layer is plain.

f) *Universal Testing machine (Model M350-5CT)*
g) *Vernier calipers*
h) *Injection syringe: to inject the silicone fiber mix into the cast*

Figure 22: Syringe used to inject and measure composite mix in the moulds

The syringe was used to measure the amount of silicone fiber mix and also to inject the mix into the molds.

i) *Nuts and bolts: to hold the layers of cast together*
j) *Vacuum pump*
k) *Weighing balance precise to 0.1 gm*
l) *Microscope*
m) *Paper cups*

3.2 Preparation of the silicone fiber compound

Mold silicone A and Mold silicone B were mixed in the ratio of 1:1 and weighted with a scale of precision $\pm 0.1\ gm$ and then the fibers were added to make a mechanical compound of silicone and fibers. The compound of four different fiber volume fractions for each fiber were made. [38] Fibers were also weighted in the same weighing scale and added into the mix and the total volume of the mix was recorded afterwards. The total mass of the mix was calculated by adding the mass of the fiber and the silicone used.

Density of carbon fiber, glass fiber and cellulose were available from the manufacturers data. The composite mix was then stirred manually until the mix became homogenous. The mix was then put in a vacuum chamber until the air bubbles were taken away. With mass and density of the fiber available, the volume of the fiber was calculated as.

$$\rho = \frac{M}{V} \text{ [39]} \qquad \text{(Equation 7)}$$

$$\text{i.e. } v = \frac{M}{\rho}$$

where.

$\rho = density$

$m = mass$

$v = volume$

The total volume of the mix was measured in the lab with a labelled syringe. The fiber volume fraction of the mix was than calculated using.

$$V_f = \frac{v_f}{v_c} \text{ [40]} \qquad \text{(Equation 8)}$$

Where;

$V_f = fiber\ volume\ fraction$

$v_f = volume\ of\ fibers$

$v_c = total\ volume\ of\ the\ composite$

The composite mix of known fiber volume fraction was then injected into the casting mold with a syringe and left for almost 12 hours each time at room temperature.

3.3 The casting mould

The moulds for casting were designed using the solidworks software. Three different types of casting moulds were designed and later laser cut using a laser cutter. Top layer, middle layer and a bottom layer were designed for each mould. The top having the inlet and outlet, the middle layer having the cutout of the shape of the specimen and the bottom layer with holes for to hold the screws.

3.3.1 Tensile Test Specimen (ASTM D3039)

ASTM 3039 tensile test is a standard used to measure the breaking force of a polymer composite specimen and the specimen's elongation at break. Considering the specimen for our experiment is a composite of silicone and fibers, this test was chosen as the testing method for the tensile properties of the specimen produced.

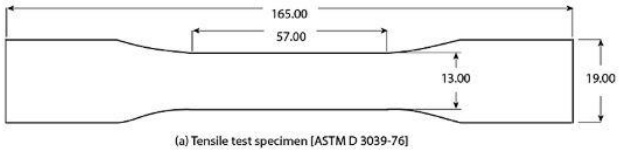

Figure 23: Tensile test specimen ASTM D3039 [37]

3.3.2 ASTM D624 – 00(2012)

This test method is used to measuring the tearing properties of conventional vulcanized rubber. [41] The test specimens from ASTM D3039 and ISO 37-1 didn't break during the test, so this standard was used to produce specimen for studying the tearing properties of the composite material we were working with.

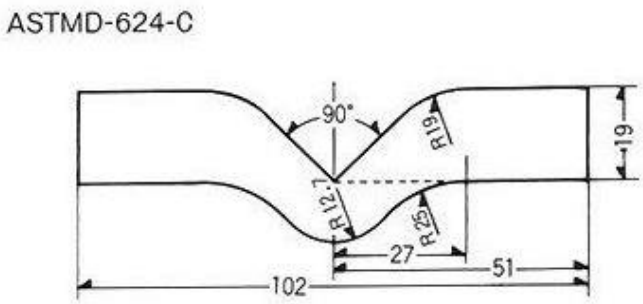

Figure 24: ASTM D624-C tear test specimen dimensions [36]

3.4 Microscopic analysis of fibers

The microscopic analysis of the fibers is done to examine the shape, size and orientation of the fibers. The intend of this analysis is to examine what kind of advantages/disadvantages we might have during the reinforcement because of these factors.

3.4.1 Fiber length distribution of fibers

Fiber lengths were measured for each fiber strand under the microscope.

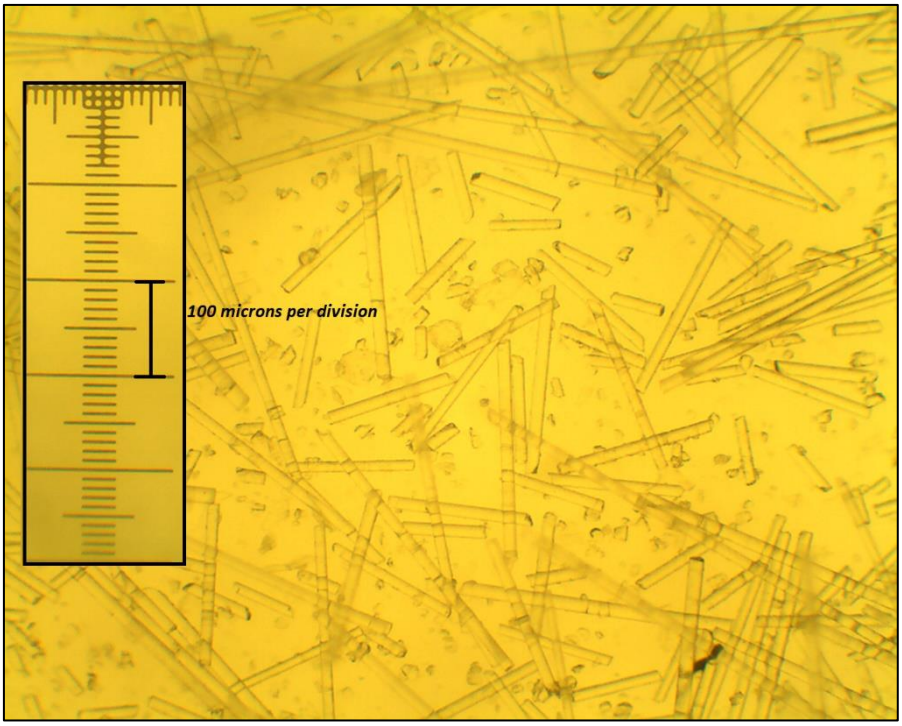

Figure 25: 10x glass fiber transmission image with a reference scale

The shapes of glass fibers were observed to be cylindrical. Using the reference scale which was taken at 10x magnification as well we can measure the size of the fibers. The reference scale has 100 microns per 1 division.

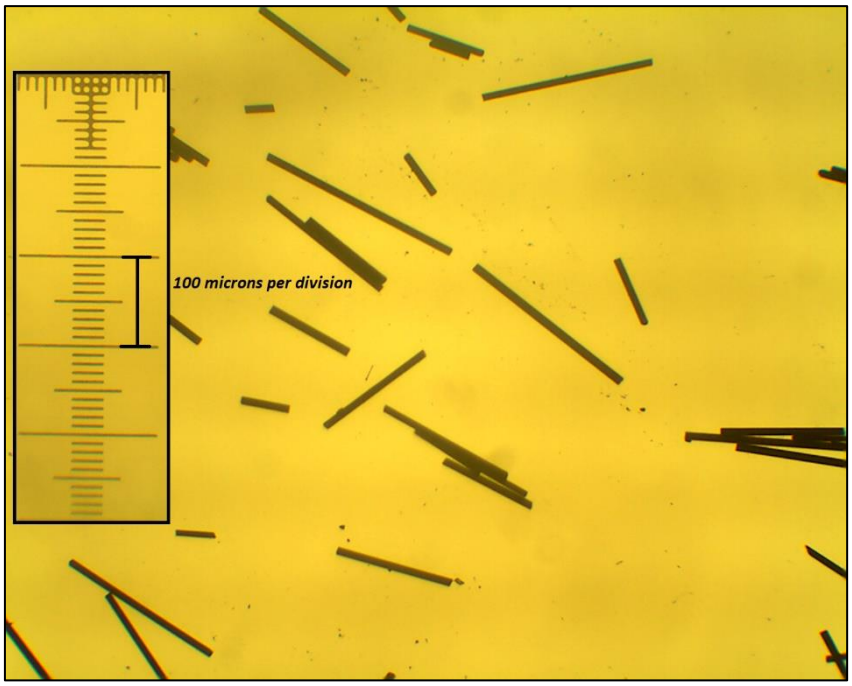

Figure 26: 10x carbon fiber transmission image with a reference scale

The shapes of carbon fibers were observed to be cylindrical as well. Using the reference scale which was taken at 10x magnification as well we can measure the size of the fibers. The reference scale has 100 microns per 1 division.

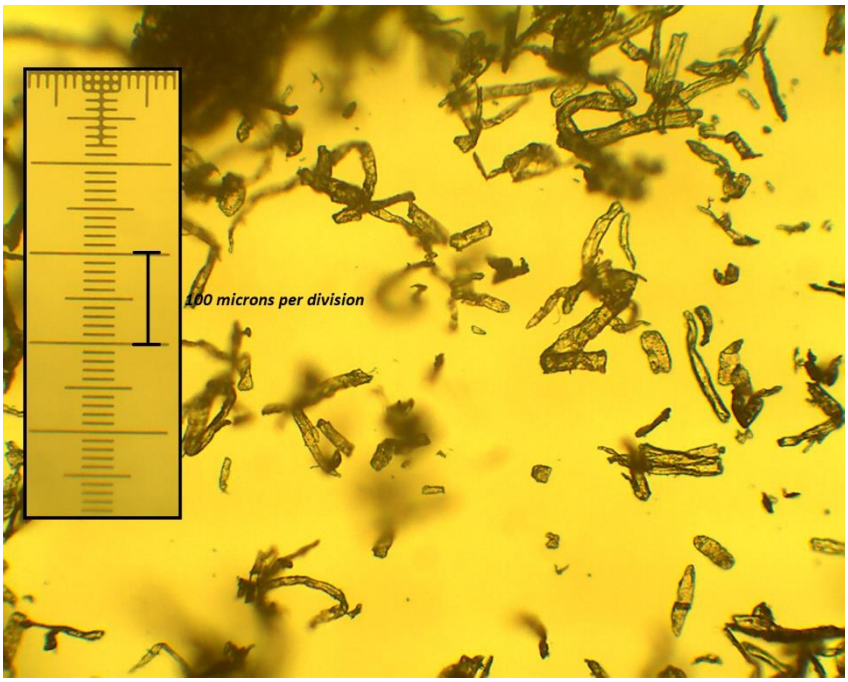

Figure 27: 10x cellulose fiber transmission image with a reference scale

The shapes of carbon fibers were observed to be irregular. These are the only bio-fibers used during the experiments. Using the reference scale which was taken at 10x magnification as well we can measure the size of the fibers. The reference scale has 100 microns per 1 division as shown in the figure above.

Below is the graphical representation of fiber length distribution of each type of fiber and the percentual distribution of fibers according to the length.

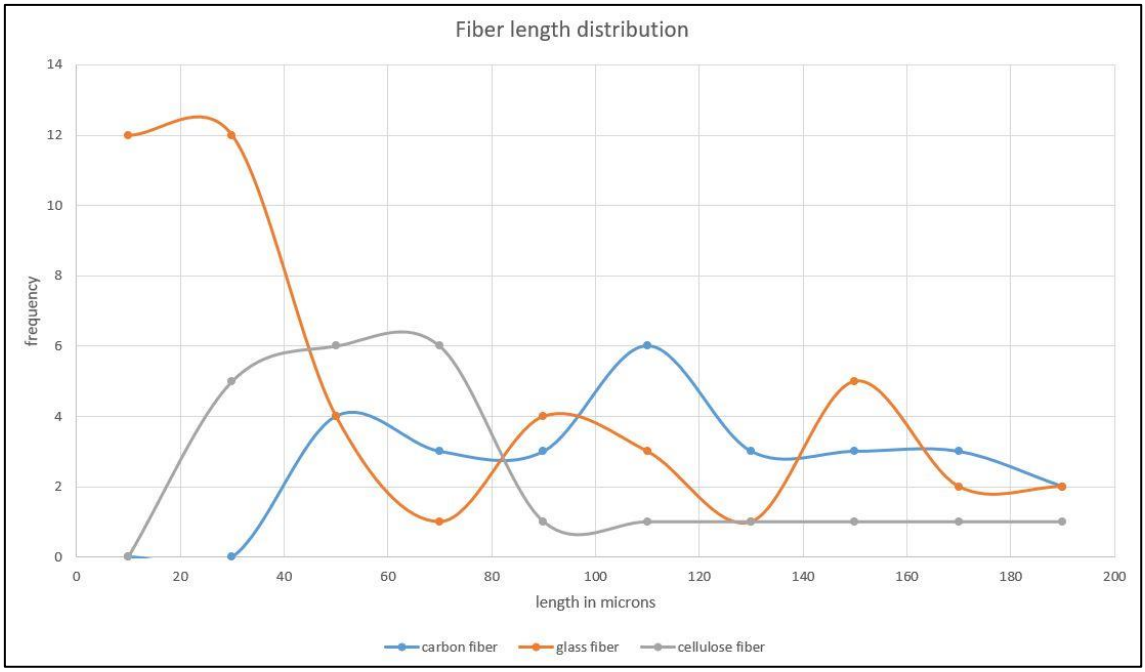

Figure 28: Length distribution of fibers

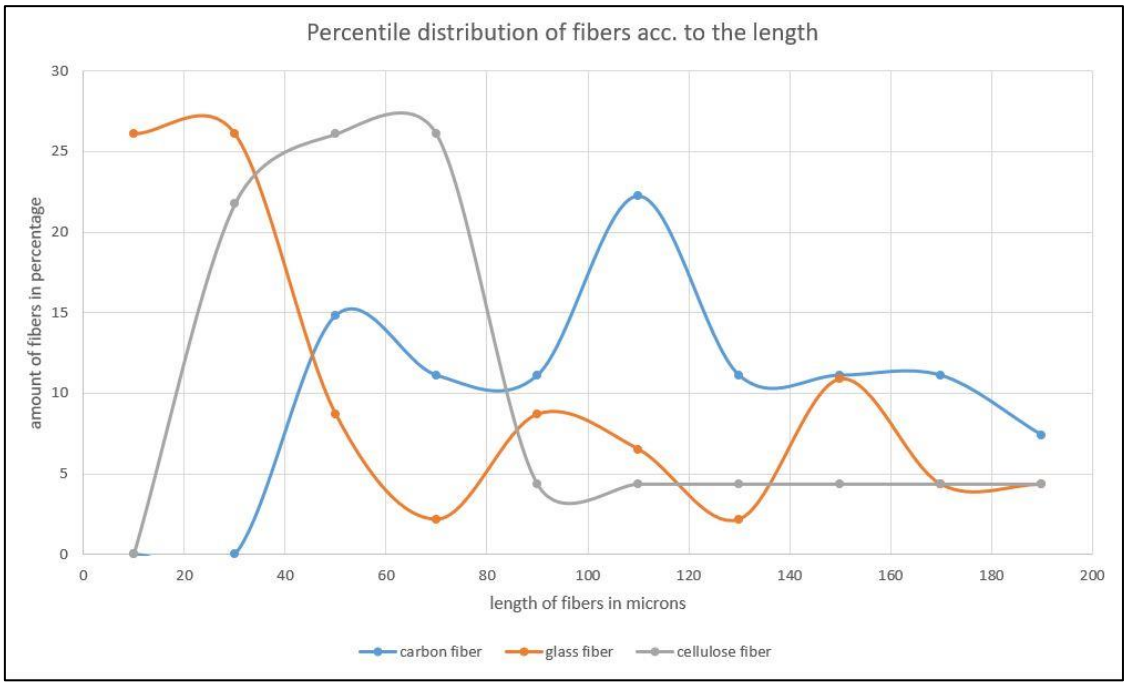

Figure 29: Percentile distribution of fibers according to length

Table 1: Percentile distribution of fibrous additives according to length

Length (microns)	***carbon fiber (%)***	***glass fiber (%)***	***Cellulose (%)***
10	*0*	*26.08*	*0*
30	*0*	*26.08*	*21.73*
50	*14.81*	*8.69*	*26.08*
70	*11.11*	*2.17*	*26.08*
90	*11.11*	*8.69*	*4.34*
110	*22.22*	*6.52*	*4.34*
130	*11.11*	*2.17*	*4.34*
150	*11.11*	*10.86*	*4.34*
170	*11.11*	*4.347*	*4.34*
190	*7.40*	*4.347*	*4.34*

The figure 28, figure 29 and Table 1 show the fiber length distribution and the percentile distribution of fibers according to length. Carbon fibers were observed to have almost about 22% of its fiber length in between 100 microns-120 microns. While glass fiber had some very long fibers, most of the fibers were very short. Almost 52% of the total fibers were below the length of 30 microns. Cellulose had almost 20% fibers below 30 microns, 26% between 30 microns- 50 microns and 26% between 50 microns – 70 microns. Fibers longer than 70 microns were rarely occurring in cellulose additive.

From figure 25, Figure 266 and Figure 277 the shapes of the fiber can be observed. Glass fibers were observed to have cylindrical shape and a diameter slightly larger than that of carbon fibers. Carbon fibers had a smaller radius than that of glass fiber and were also observed to be cylindrical as well. Though cellulose fiber had more fibers at about 70 microns the fibers had a bended shape.

3.5 Modelling of modulus

3.5.1 Hooke's Law

When a force F is applied at each end of a bar with cross-section A, it elongates from its original length L_0 to L_n meanwhile decreasing the cross-section.
The stress is given by the quotient of tensile force divided by the cross-section.

$$\delta = \frac{F}{A} \text{ [42]} \qquad \text{(Equation 9)}$$

Where,
$\delta = stress$
$F = tensile\ force$
$A = cross\ section\ area$

The strain is given by.

$$\epsilon = \frac{L_n - L_0}{L_0} \text{ [42]} \qquad \text{(Equation 10)}$$

Where,
$L_n - L_0 = \Delta L = change\ in\ length$
$L_0 = original\ length$

The Young's modulus can be thus measured from the slope of the force over elongation data as;

$$E = \frac{stress}{strain}$$

$$E = \frac{\frac{F}{A}}{\frac{\Delta L}{L_0}}$$

$$E = \frac{F \times L_0}{A \times \Delta L}$$

$$E = \frac{F}{\Delta L} \times \frac{L_0}{A}$$

$$E = slope \times \frac{L_0}{A}$$

$$E = \frac{slope \times L_0}{A} \qquad \text{(Equation 11)}$$

3.5.2 Derivation of Experimental modulus

The modulus was modelled with respect to the rule of mixture

The rule of mixture suggests the modulus of a composite when the fiber transfers the stress in the direction of the forces can be as high as;

$$E_{upper\ bound} = f\ E_f + (1 - f)\ E_m$$

Where;

- E_f (material property of the fibers)
- E_m (material property of the matrix)
- $f = \frac{V_f}{V_f + V_m}$ is the fiber volume fraction

That can also be written as;

$$E^1{}_{upper\ bound} = f\ E_f^1 + (1 - f)\ E_m^1$$

Whereas, the rule of mixture suggests the modulus of a composite when the fiber does not transfer the stress in the direction of the force can be as low as;

$$E_{lower\ bound} = \left(\frac{f}{E_f} + \frac{(1 - f)}{E_m}\right)^{-1}$$

- E_f (material property of the fibers)
- E_m (material property of the matrix)
- $f = \frac{V_f}{V_f + V_m}$ is the fiber volume fraction

Which can also be written as;

$$E^{-1}{}_{lower\ bound} = f\ E_f^{-1} + (1 - f)\ E_m^{-1}$$

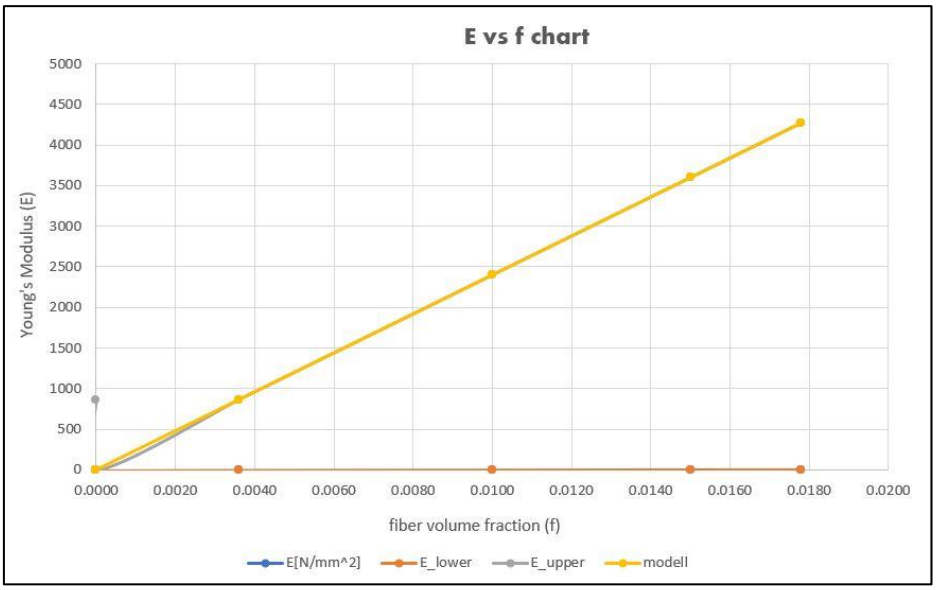

Figure 30: p-value graph of carbon reinforced silicone when p=1

The figure 30 shows when the p-value reaches 1, the modulus of the specimen becomes as high as upper bound modulus and the graphs for the upper bound modulus and the modelled modulus overlap.

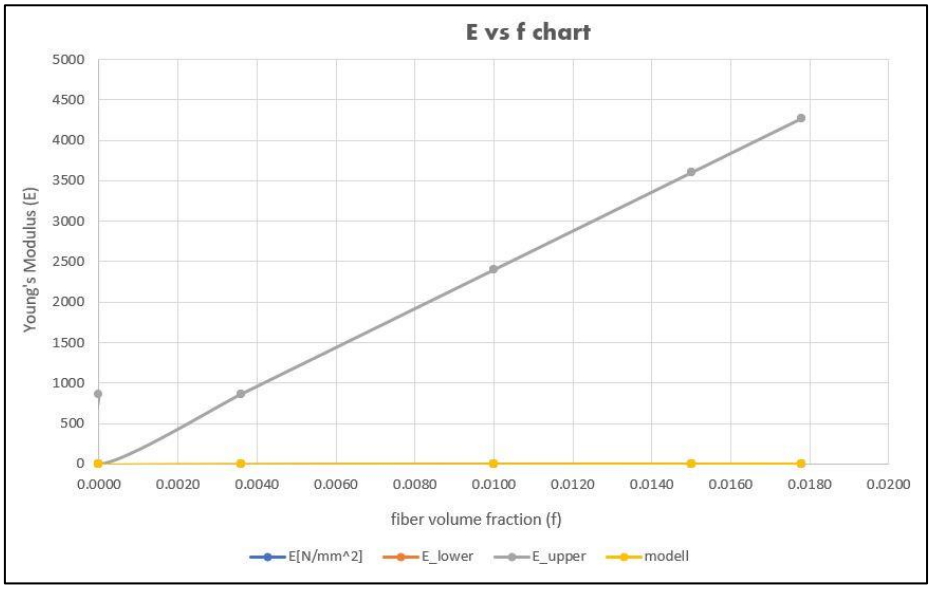

Figure 31: p-value graph of carbon fiber reinforced silicone at p= -1

The figure 31 shows when the p-value reaches -1, the modulus of the specimen becomes as low as lower bound modulus and the graphs for the lower bound modulus and the modelled modulus overlap.

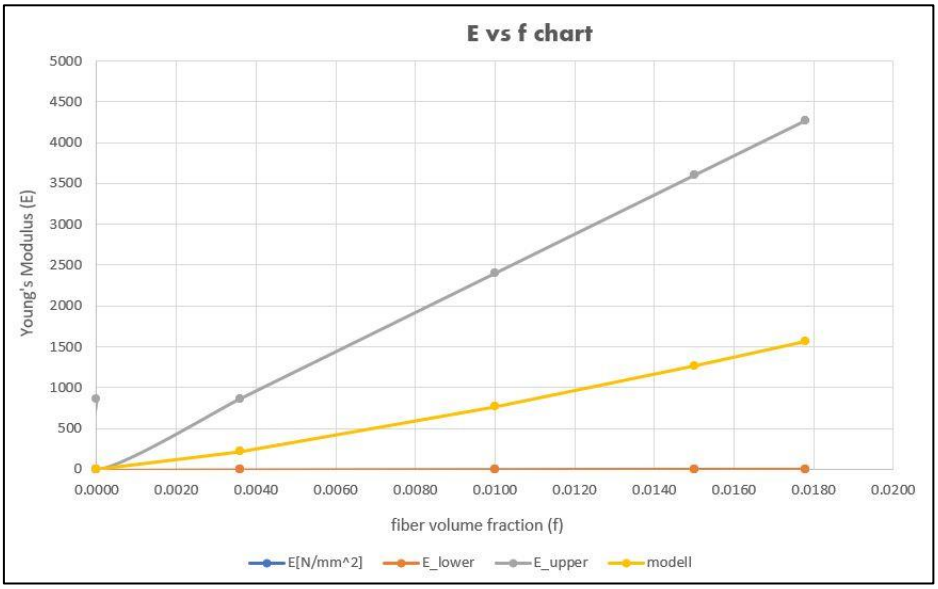

Figure 32: p-value graph of carbon fiber reinforced silicone at p= 0.8

When the p-value is in-between 1 and -1 the graph for the modelled modulus lies in-between the upper bound and lower bound modulus'. The graph approaches more towards the upper bound modulus when the p-value approaches +1.

The rule of mixture generalizes:

Modulus	***Equation***	***p-value***
Upper	$E^{1}{}_{upper\ bound} = f\ E_f^1 + (1-f)\ E_m^1$	$p = 1$
Lower	$E^{-1}{}_{lower\ bound} = f\ E_f^{-1} + (1-f)\ E_m^{-1}$	$p = -1$
Interme-diate	$E^{p}_{experimental} = f\ E_f^p + (1-f)\ E_m^p$	$-1 \leq p \leq 1\,, p \neq 0$

Thus, from the above equations, while modelling our experimental modulus of our composite the general formula for the experimental modulus is derived as;

$$E_{experimental}^{p} = f\ E_f^p + (1-f)\ E_m^p$$

$$or,\ \ E_{experimental} = \sqrt[p]{f\ E_f^p + (1-f)\ E_m^p}$$

$$or,\ \ E_{experimental} = \left(f\ E_f^p + (1-f)\ E_m^p\right)^{\frac{1}{p}}$$

The experimental young's modulus is then derived as;

$$E_{experimental} = \left(f\ E_f^p + (1-f)\ E_m^p\right)^{\frac{1}{p}} \qquad \text{(Equation 12)}$$

- E_f (material property of the fibers)
- E_m (material property of the matrix)
- $f = \left(\frac{V_f}{V_f+V_m}\right)$ is the fiber volume fraction
- $p(\ reinforcement\ factor)\ where\ -1 \leq p \leq 1\ , p \neq 0$

3.6 Mechanical testing

Mechanical testing of the test samples was done at Arcada University Laboratory using Universal Testing machine (Model M350-5CT). Tensile tests were done with Tensile Test Specimen (ASTM D3039) and ISO 37-1 specimen while tear test was done with ASTM D624 – 00(2012) specimen. The clamps used for the testing were model: PWG25.

Figure 33: Clamps used for mechanical testing. Model: PWG25

Figure 34: Tensile test specimen during testing

The figure 31 shows how the tensile test specimen was loaded in the UTM during tensile testing.

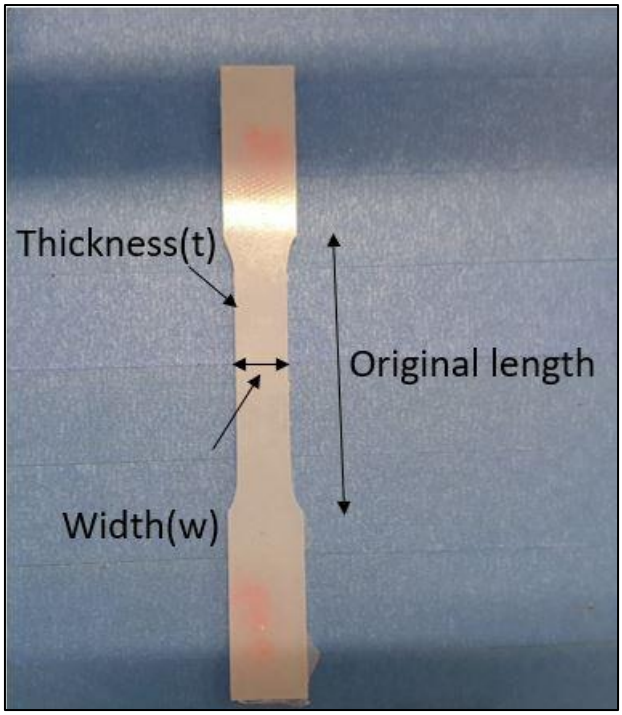

Figure 35: Tensile test specimen detailing where the measurements were taken from

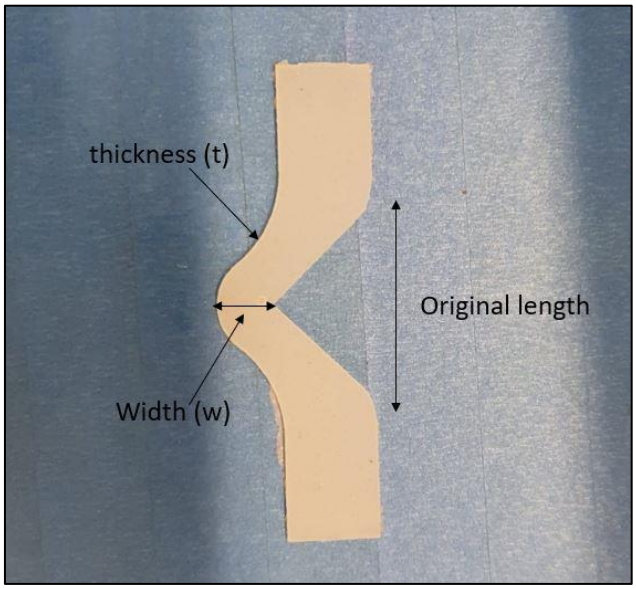

Figure 36: Tear test specimen detailing where the measurements were taken from

The figures 35 and figure 36 show how the specimen looked before testing and where exactly the dimensions for the testing were taken from. The dimensions were measured using Vernier calipers.

3.6.1 Test specifications for Tensile Test Specimen (ASTM D3039)

A total of 10 test specimen were made with each fiber. 30 different specimens of silicone with glass fiber, carbon fiber and cellulose fiber were casted and tested. The fiber volume fraction of the specimen 0.3%-1.7% for carbon fiber reinforced silicone, 2%-20% for glass fiber reinforced silicone and 3%-27% for cellulose fiber reinforced silicone. The test specifications used during tensile testing are:

- Abs: 375mm
- Width: 13.7mm
- Thickness: 3.2mm
- Test speed: 100 mm/min

3.6.2 Test specifications for ASTM D624 – 00(2012) specimen

Two different fiber volume fraction sample with each fiber were made. A specimen with just silicone was also made to see what effects the fiber has on the tear properties. The test specifications used during the tear test are:

- Abs: 355mm
- Width: 12.7mm
- Thickness: 3.2mm
- Test speed: 100 mm/min

3.6.3 Tear test

Tear test were performed with ASTM D624 – 00(2012) specimen to investigate if fiber reinforcement had an effect in improving the tearing properties of the silicone. Two fiber volume fraction samples of each fiber were made and tested.

Figure 37: Tear test specimen during testing

Tear test were performed with ASTM D624 – 00(2012) specimen to investigate if fiber reinforcement had an effect in improving the tearing properties of the silicone. Two fiber volume fraction samples of each fiber were made and tested.

Tensile stress is given by;

$$\delta_{tear} = \frac{F}{A}$$

Where;

$F = \ force\ at\ tear$

$A = \ cross - section\ area\ of\ the\ specimen$

Tear strain is given by;

$$\varepsilon_{tear} = \frac{\Delta L}{L_0}$$

Where;

$\Delta L = \ elongation\ at\ break$

$L_0 = \ original\ length$

4 RESULTS

4.1 Comparison of modelled and tested modulus

Data from mechanical testing of tensile test ASTM 3039 specimen were saved as *.csv files while enabled the file to be opened with Ms. Excel. Graphs were then created using the data in the excel and then the slope of the graphs on the elastic region were recorded. Width, thickness and the length of the specimen were measured with the help of Vernier calipers and recorded prior to the testing.

4.1.1 Carbon fiber

The p-value was switched from -1 to +1 until the graph of the experimental modulus was aligned with the graph of tested modulus. For the carbon fiber reinforced silicone, the experimental modulus aligned with the tested modulus at $p = 0.3$.

$$Young's\ modulus\ of\ the\ matrix(E_m) = 0.98\ MPa$$

$$Young's\ modulus\ of\ the\ fiber(E_f) = 240\ GPa$$

The p-value whereas ranges from -1(lower bound) being completely unreinforced to +1(upper bound) completely reinforced i.e. the p value range is 2. Having a p=0.3 means that the position of p at 0.3 is 1.3 units higher from -1. So, the percentual reinforcement of the carbon fiber in this case would be;

$$percentual\ reinforcement = \frac{1.3}{2} \times 100\% = 65\%$$

4.1.1.1.1 Progression of the p-value

The above graph illustrates Young's modulus vs fiber volume fraction graph of Tested modulus, Lower bound modulus, upper bound modulus, and theoretical modulus' at different p-values. p-value was switched from -1 to +1 excluding 0, in the excel file until the p-value which aligns the tested modulus graph and the modelled modulus graph. The p-value progression shows how the modelled E vs f graph gradually falls from upper bound to lower bound from +1 to -1 aligning to the tested E vs f graph just at p= 0.3.

Below is the graphical representation of the progression of the p-value graph.

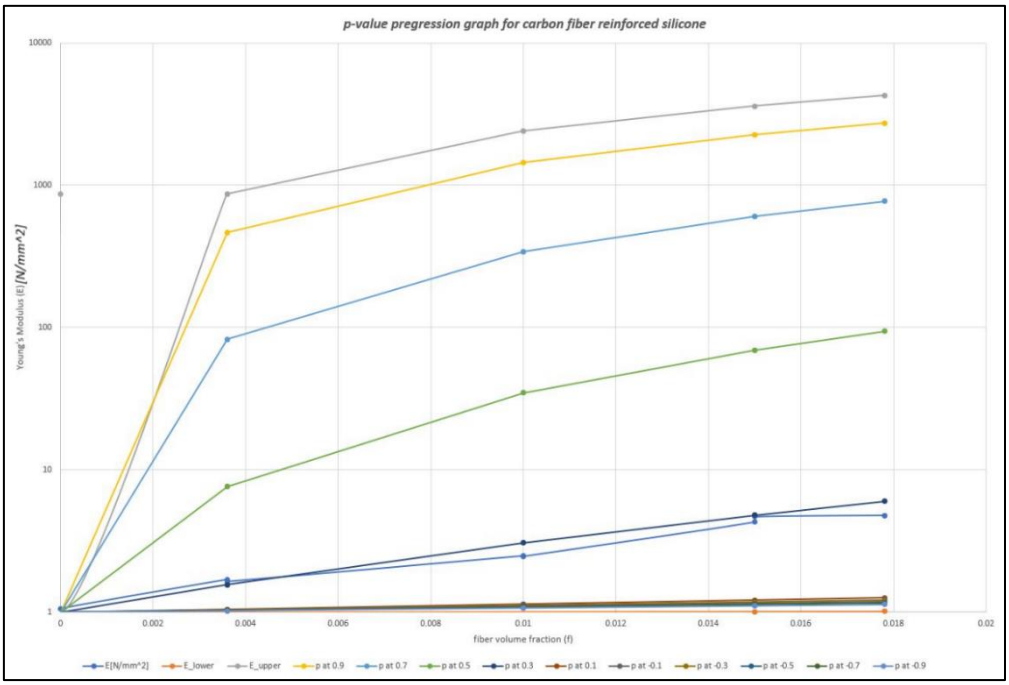

Figure 38: Progression of p-value from 0.9-0.1 for carbon fiber reinforced silicone

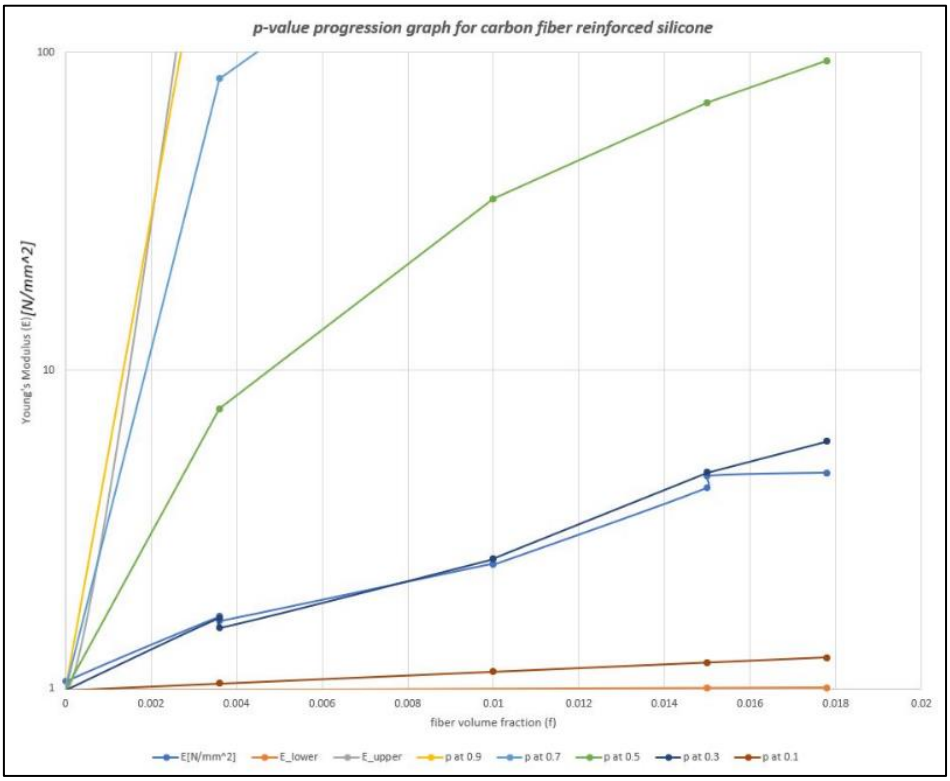

Figure 39: p-value graph for carbon fiber reinforced silicone aligns to the tested modulus graph at p=0.3

Table 2: Data of carbon fiber reinforced specimens at p=0.3

f	slope[N/mm]	E_{tested} [N/mm^2] [$E_{tested} = \frac{slope \times L_0}{A}$]	E_{lower} [N/mm^2] $E_c = (\frac{f}{E_f} + \frac{1-f}{E_m})^{-1}$	E_{upper} [N/mm^2] $E_c = f\,E_f + (1-f)\,E_m$	$E_{modelled}$ l[N/mm^2] $E_{experimental} = (f\,E_f^p + (1-f)\,E_m^p)^{\frac{1}{p}}$
0.0000	0.56	0.98	0.98	864.98	0.98
0.0000	0.6	1.05	0.98	864.98	0.98
0.0036	0.96	1.68	0.99	864.98	1.55
0.0036	0.93	1.6	0.99	864.98	1.55
0.0100	1.41	2.4	0.99	2400.98	3.05
0.0100	1.40	2.4	0.99	2400.98	3.05
0.0150	2.44	4.279	1.00	3600.97	4.77
0.0150	2.66	4.65	1.00	3600.97	4.77
0.0178	2.73	4.77	1.007	4272.97	5.99
0.0178	2.72	4.74	1.007	4272.97	5.99

Where;

- Width (w) = 12.7 mm
- Thickness (t) = 3.1 mm
- Area of cross-section (A) = 39.3 mm^2
- Original length (L_0) = 68.7 mm

4.1.2 Glass fiber

The p-value was switched from -1 to +1 until the graph of the experimental modulus was aligned with the graph of tested modulus. For the carbon fiber reinforced silicone, the experimental modulus aligned with the tested modulus at $p = 0.075$.

$$Young's\ modulus\ of\ the\ matrix(E_m) = 0.98\ MPa$$
$$Young's\ modulus\ of\ the\ fiber(E_f) = 70\ GPa$$

4.1.2.1.1 Progression of the p-value for glass fiber

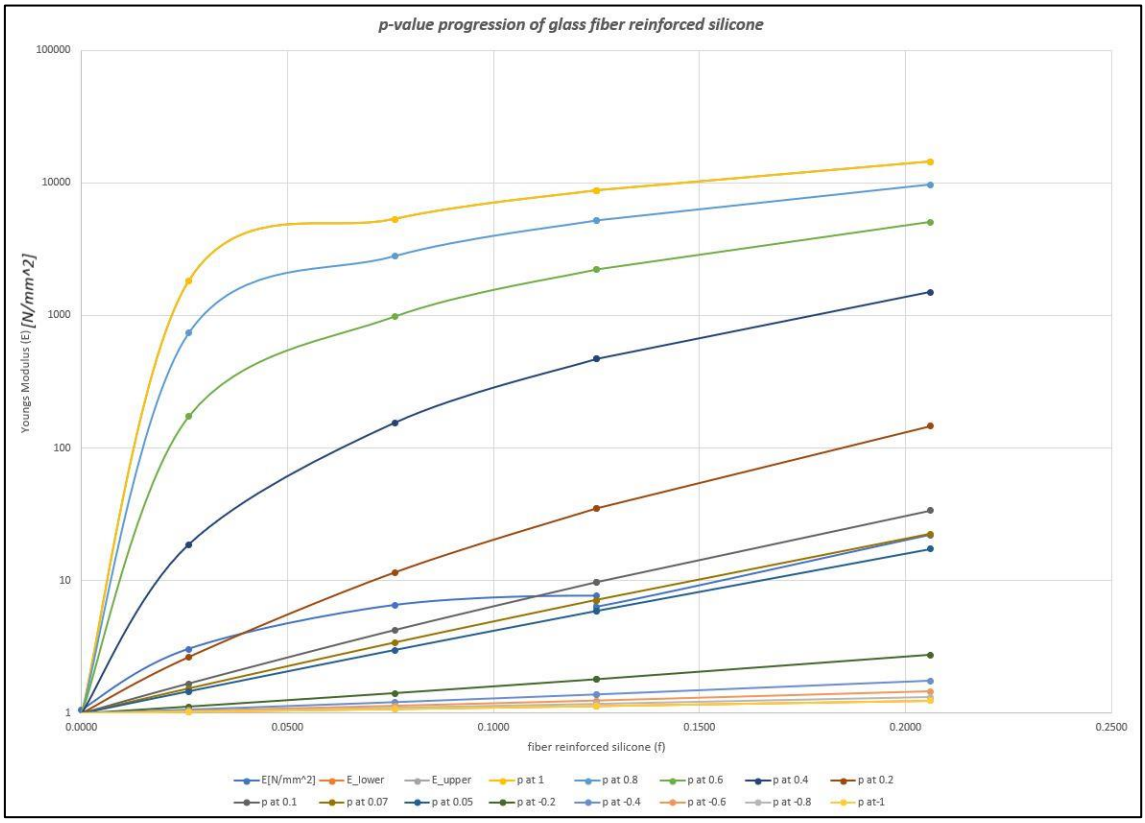

Figure 40: p-value graph progression of glass fiber reinforced silicone

The above graph illustrates Young's modulus vs fiber volume fraction graph of Tested modulus, Lower bound modulus, upper bound modulus, and theoretical modulus' at different p-values. p-value was switched from -1 to +1 excluding 0, in the excel file until the p-value which aligns the tested modulus graph and the modelled modulus graph. The p-value progression shows how the modelled E vs f graph gradually falls from upper bound

to lower bound from +1 to -1 aligning to the tested E vs f graph just at p= 0.07.

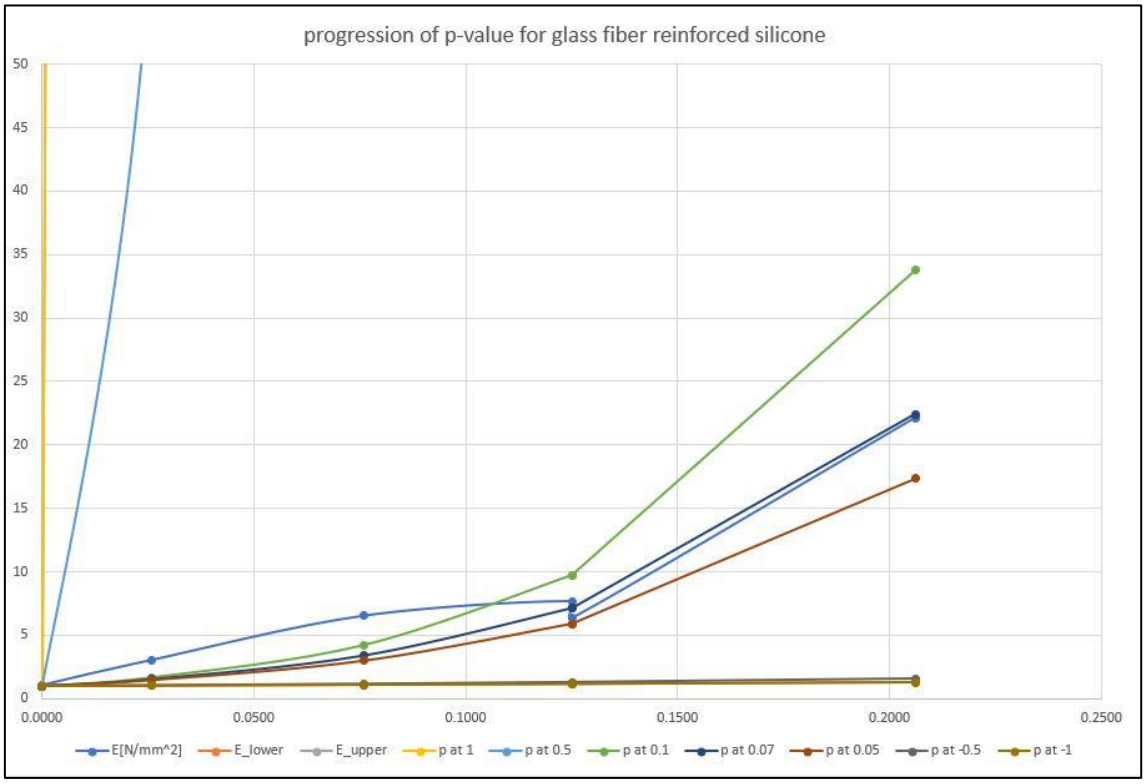

Figure 41: p- value graph of glass fiber reinforced silicone aligns with the tested modulus graph at p= 0.07

In figure 38 the tested modulus' graph aligns to that of p-value graph at p= 0.07. Thus, the percentual reinforcement of the silicone matrix due to glass fiber can be calculated as;

$$percentual\ reinforcement = \frac{1.075}{2} \times 100\% \ = 53.75\ \%$$

Table 3: Data of glass fiber reinforced silicone at p=0.075 [N/mm^2]

f	slope[N/mm]	E_{tested} [N/mm^2] $[E_{tested} = \frac{slope \times L_0}{A}]$	E_{lower} [N/mm^2] $E_c = (\frac{f}{E_f} + \frac{1-f}{E_m})^{-1}$	E_{upper} [N/mm^2] $E_c = f\,E_f + (1-f)\,E_m$	$E_{modelled}$ l[N/mm^2] (p-value) $E_{experimental} = (f\,E_f^p + (1-f)\,E_m^p)^{\frac{1}{p}}$
0.0000	0.56	0.98	0.98	0.98	0.98
0.0000	0.60	1.05	0.98	0.98	0.98
0.0260	1.74	3.04	1.01	1820.96	1.54
0.0760	3.74	6.52	1.07	5320.91	3.50
0.1250	4.39	7.66	1.13	8750.86	7.48
0.1250	3.62	6.33	1.13	8750.86	7.48
0.2060	12.64	22.12	1.24	14420.79	23.95

Where;

- Width (w) = 12.7 mm
- Thickness (t) = 3.1 mm
- Area of cross-section (A) = 39.3 mm^2
- Original length (L_0) = 68.7

4.1.3 Cellulose fiber

The p-value was switched from -1 to +1 until the graph of the experimental modulus was aligned with the graph of tested modulus. For the carbon fiber reinforced silicone, the experimental modulus aligned with the tested modulus at $p = -0.5$.

$$Young's\ modulus\ of\ the\ matrix(E_m) = 0.98\ MPa$$

$$Young's\ modulus\ of\ the\ fiber(E_f) = 20\ GPa$$

4.1.3.1.1 Progression of the p-value

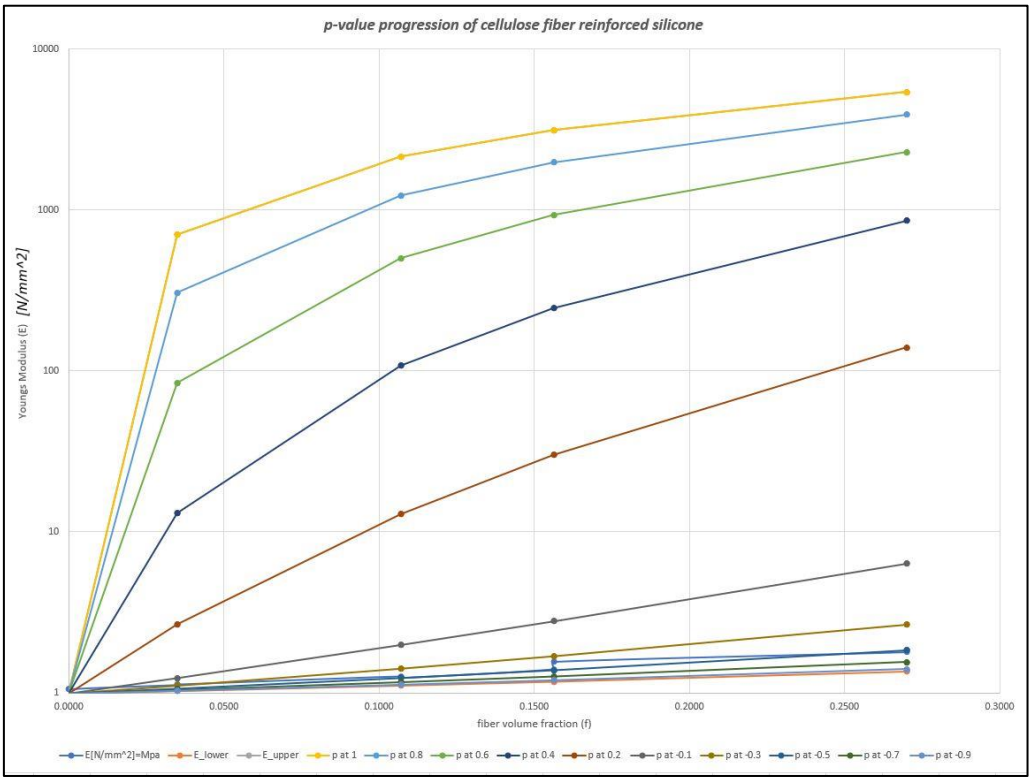

Figure 42: Progression of p-value graph for cellulose fiber reinforced silicone

The above graph illustrates Young's modulus vs fiber volume fraction graph of Tested modulus, Lower bound modulus, upper bound modulus, and theoretical modulus' at different p-values. p-value was switched from -1 to +1 excluding 0, in the excel file until the p-value which aligns the tested modulus graph and the modelled modulus graph. The p-value progression shows how the modelled E vs f graph gradually falls from upper bound to lower bound from +1 to -1 aligning to the tested E vs f graph just at p= -0.5.

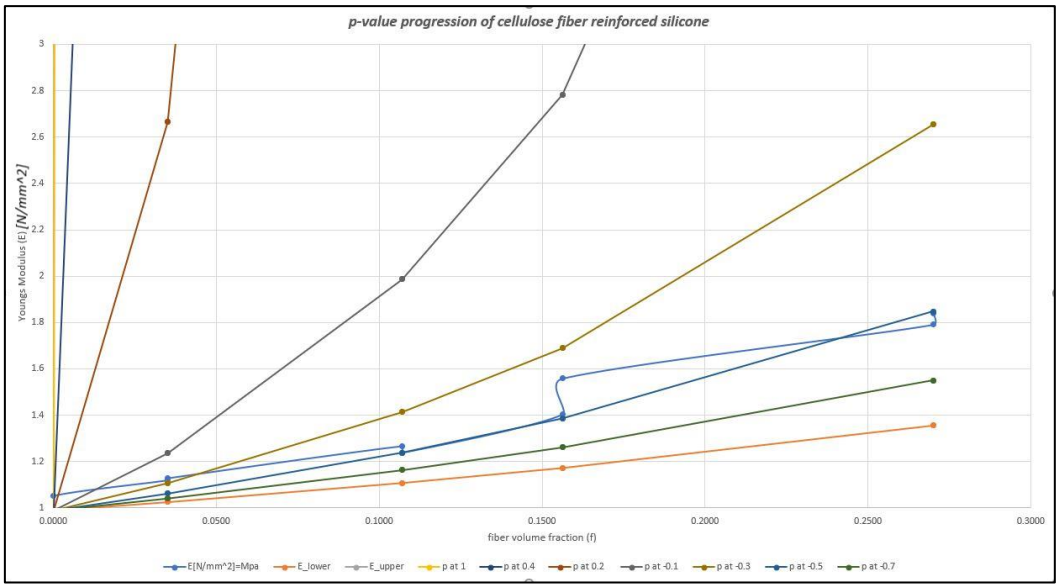

Figure 43: p-value graph aligns with tested modulus at p= -0.5 for cellulose fiber reinforced silicone

The figure 40 shows how the p-value graph aligns with the tested modulus graph at p=-0.5. The percentual reinforcement of the cellulose fiber can be calculated as.

$$percentual\ reinforcement = \frac{0.5}{2} \times 100\% = 25\%$$

Table 4: Data of cellulose fiber reinforced silicone at p= -0.5

f	slope[N/mm]	E_{tested} [N/mm^2] [$E_{tested} = \frac{slope \times L_0}{A}$]	E_{lower} [N/mm^2] $E_c = (\frac{f}{E_f} + \frac{1-f}{E_m})^{-1}$	E_{upper} [N/mm^2] $E_c = f\,E_f + (1 - f)\,E_m$	$E_{modelled}$ l[N/mm^2] $E_{experimental} = (f\,E_f^p + (1 - f)\,E_m^p)^{\frac{1}{p}}$
0.0000	0.56	0.98	0.98	0.98	0.98
0.0000	0.60	1.05	0.98	0.98	0.98
0.0350	0.64	1.11	1.02	700.95	1.06
0.0350	0.64	1.12	1.02	700.95	1.062
0.1070	0.72	1.26	1.10	2140.88	1.23
0.1070	0.70	1.23	1.10	2140.88	1.23
0.1562	0.80	1.40	1.17	3124.83	1.38
0.1562	0.89	1.55	1.17	3124.83	1.38
0.2700	1.02	1.79	1.35	5400.72	1.84
0.2700	1.05	1.83	1.355	5400.72	1.84

Where;

- Width (w) = 12.7 mm
- Thickness (t) = 3.1 mm
- Area of cross-section (A) = 39.3 mm^2
- Original length (L_0) = 68.7 mm

4.2 Reinforcement and tear properties

The force and elongation at tear were obtained via tear test of ASTM D624 – 00(2012) specimen. The length, width and the thickness of the specimen were measured with the Vernier calipers prior to the testing.

$$width\ of\ teartest\ specimen\ (w) = 12.7mm$$
$$thickness\ of\ tear\ test\ specimen\ (t) = 3.2\ mm$$

$$cross - section\ area\ of\ tensile\ test\ specimen(A) = width \times thickness$$
$$= 12.7mm \times 3.2mm$$
$$= 40.64\ mm^2$$

$$original\ length\ (L_0) = 54mm$$

4.2.1 Silicone with 0% fiber

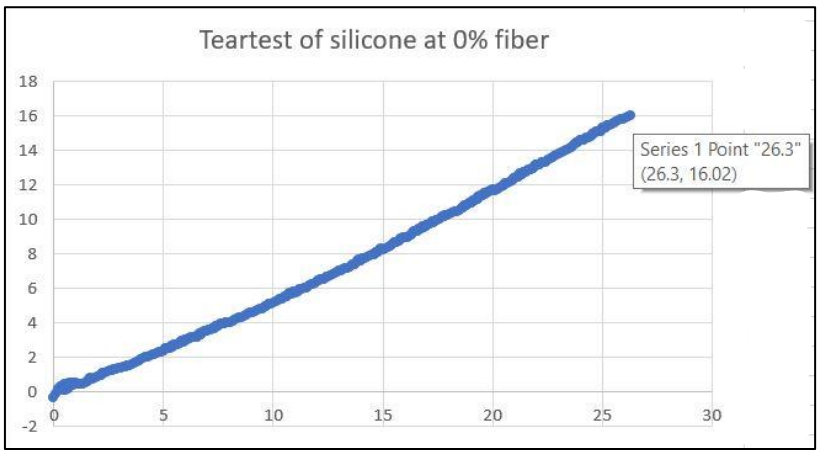

Figure 44: Tear test of silicone with 0% fiber (force vs elongation)

From the graph;

$$F = 16.05\ N$$

$$\Delta L = 26.3mm$$

Thus;

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{16.05}{40.64} = 0.39\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{26.3mm}{54mm} = 0.48 = 48\%$$

4.2.2 Carbon fiber

4.2.2.1.1 Tear properties at 0.66% fiber volume fraction

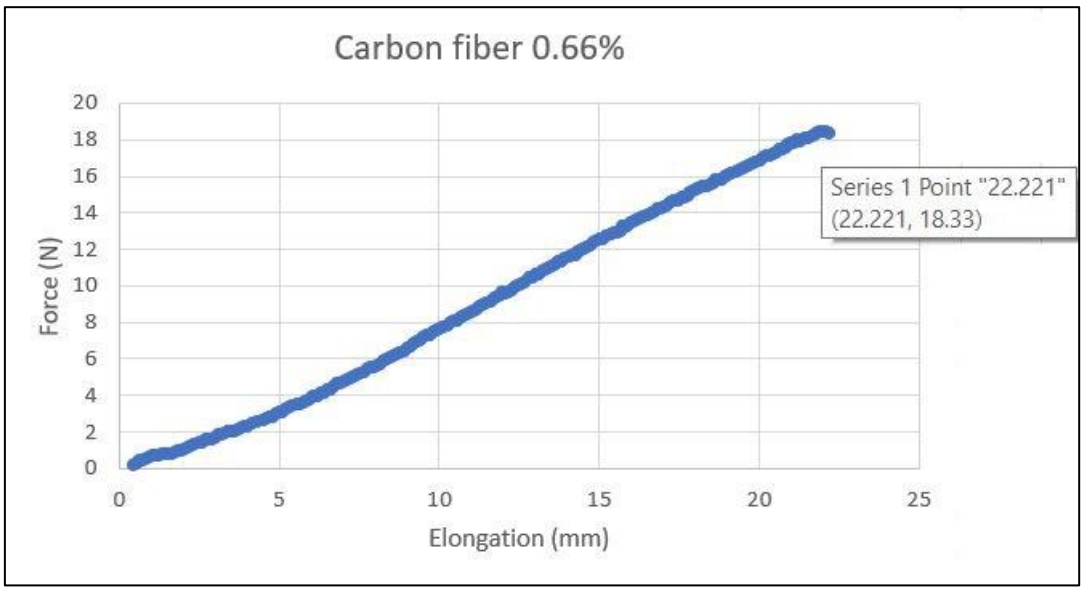

Figure 45: Force vs elongation graph of 0.66% carbon fiber reinforced silicone

From the graph;

$$F = 18.33\ N$$

$$\Delta L = 22.22\ mm$$

Thus;

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{18.45}{40.64} = 0.45\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{22.013mm}{54mm} = 0.40 = 40\%$$

4.2.2.1.2 Tear properties at 1.78% fiber volume fraction

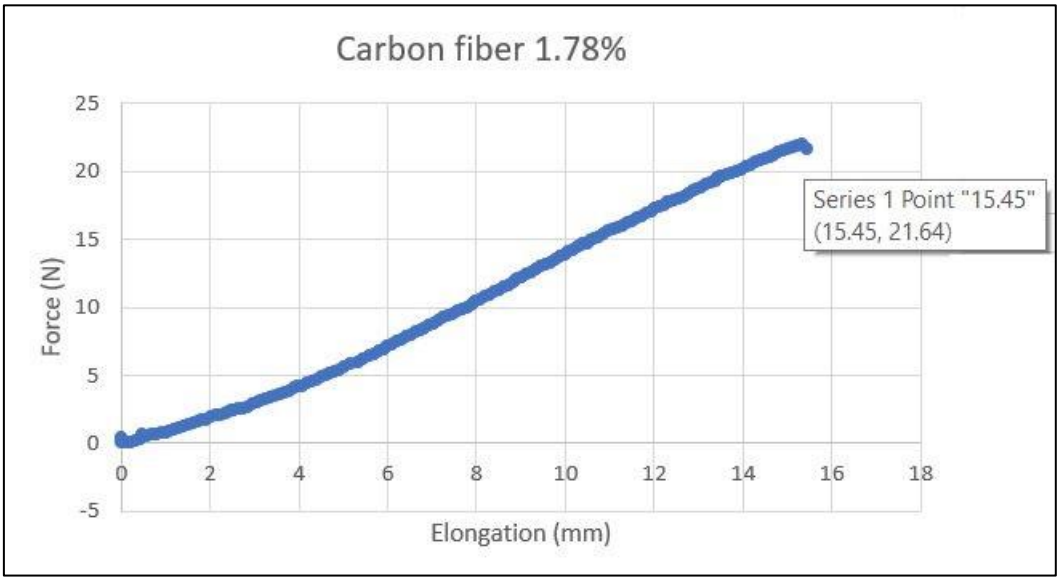

Figure 46: Force vs elongation graph of 1.78% carbon fiber reinforced silicone

From the graph;

$$F = 21.64\ N$$

$$\Delta L = 15.45mm$$

Thus;

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{21.64}{40.64} = 0.53\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{15.45mm}{54mm} = 0.28 = 28\%$$

4.2.3 Glass fiber

4.2.3.1.1 Tear properties at 0.6% fiber volume fraction

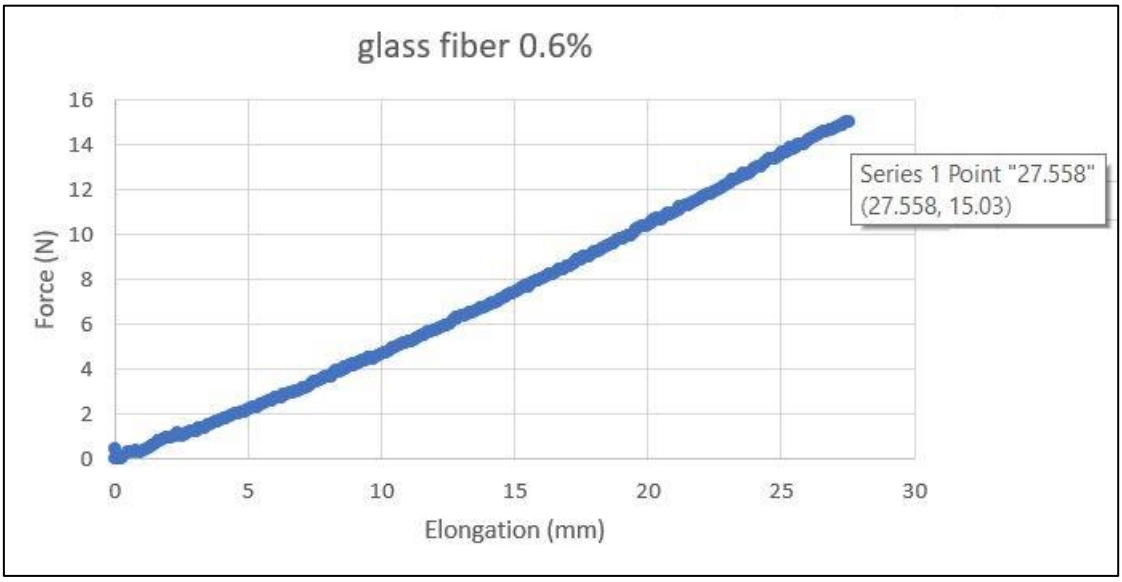

Figure 47: Force vs elongation graph of 0.6% glass fiber reinforced silicone

From the graph;

$$F = 15.03\ N$$

$$\Delta L = 27.55mm$$

Thus;

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{15.03}{40.64} = 0.36\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{27.55mm}{54mm} = 0.51 = 51\%$$

4.2.3.1.2 Tear properties at 2,7% fiber volume fraction

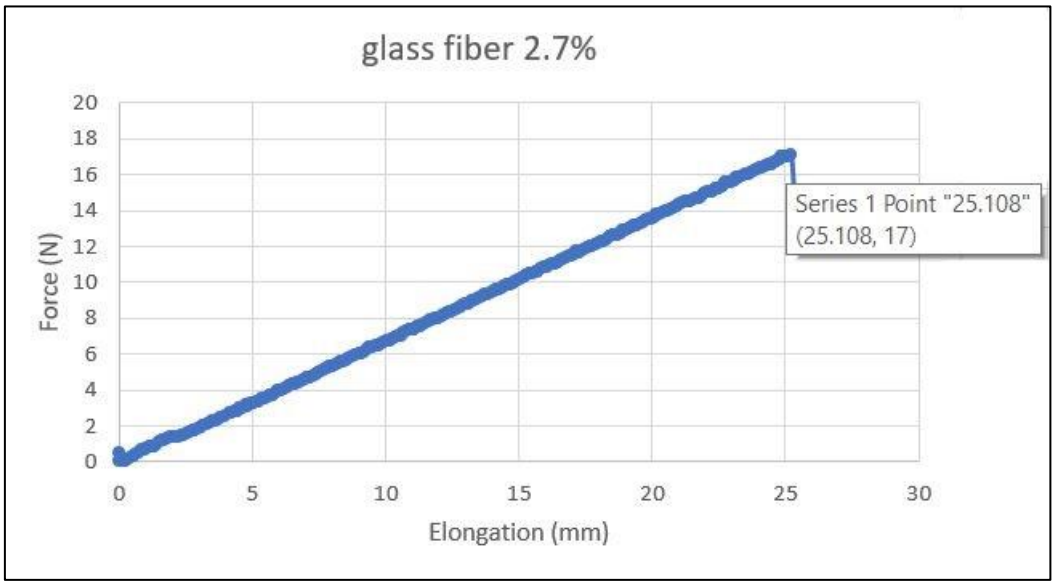

Figure 48: Force vs elongation graph of 2.7% glass fiber reinforced silicone

From the graph;

$$F = 17\ N$$

$$\Delta L = 25.10mm$$

Thus;

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{17.06}{40.64} = 0.41\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{25.21mm}{54mm} = 0.46 = 46\%$$

4.2.4 Cellulose fiber

4.2.4.1.1 Tear properties at 4.7% fiber volume fraction

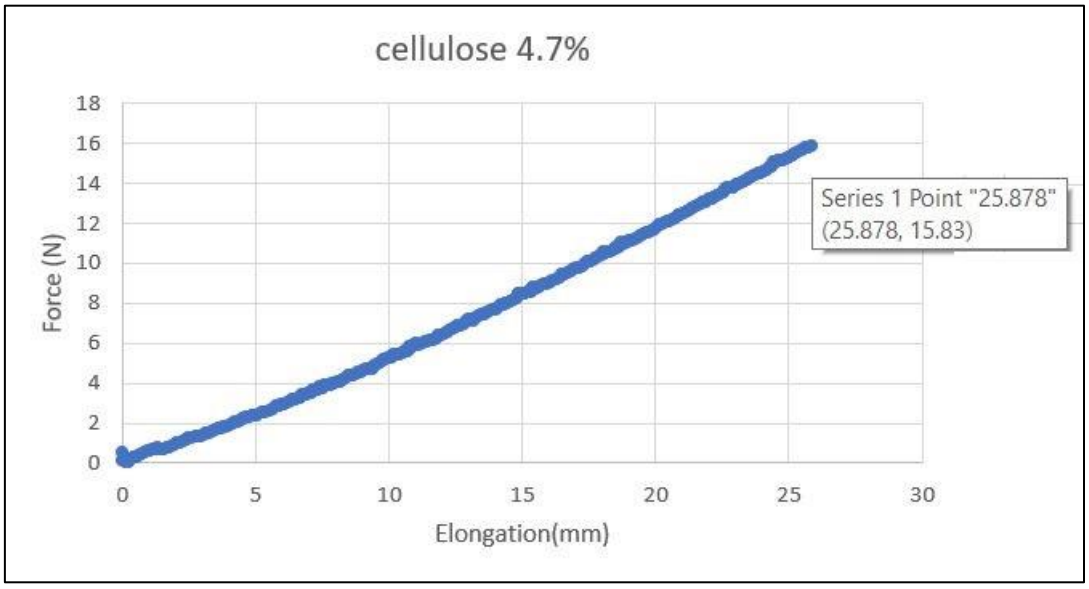

Figure 49: Force vs elongation graph of 4.7% cellulose fiber reinforced silicone

From the graph;

$$F = 15.83\ N$$

$$\Delta L = 25.87mm$$

Thus,

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{15.83}{40.64} = 0.38\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{25.87mm}{54mm} = 0.47 = 47\%$$

4.2.4.1.2 Tear properties at 24% fiber volume fraction

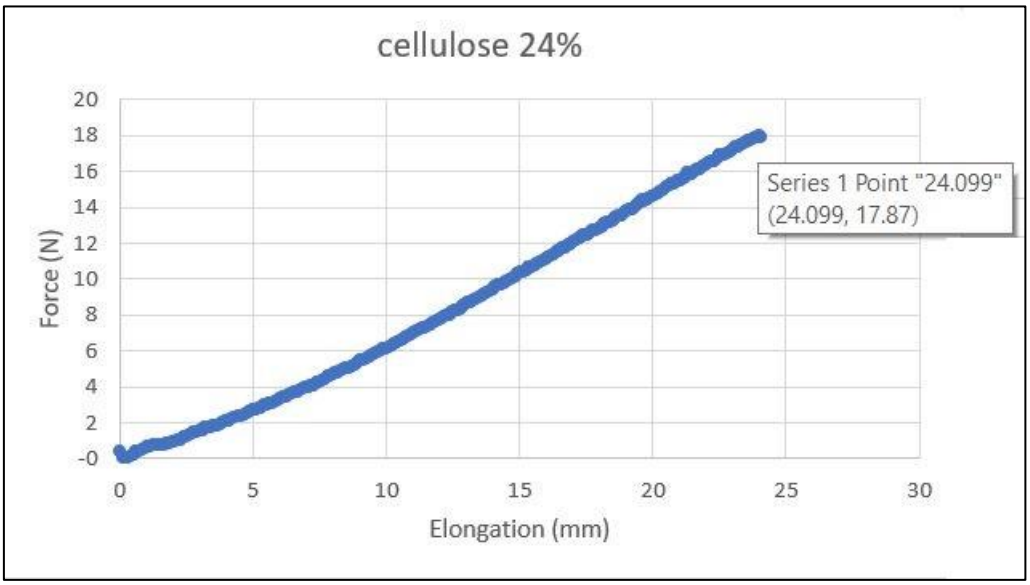

Figure 50: Force vs elongation graph of 24% cellulose fiber reinforced silicone

From the graph;

$$F = 17.87\ N$$

$$\Delta L = 24.09mm$$

Thus;

$$Tear\ stress = \delta_{tear} = \frac{F}{A} = \frac{17.87}{40.64} = 0.43\ MPa$$

$$Tear\ strain = \varepsilon_{tear} = \frac{\Delta L}{L_0} = \frac{24.09mm}{54mm} = 0.44 = 44\%$$

Table 5: Comparison of Tear properties of the fiber reinforcement

Specimen	***Fiber volume fraction***	***Tear stress***	***Tear strain***
Silicone	*0%*	*0.39 MPa*	*48%*
Carbon fiber reinforced silicone	*0.66%*	*0.45 MPa*	*40%*
	1.78%	*0.53 MPa*	*28%*
Glass fiber reinforced silicone	*0.6%*	*0.36 MPa*	*51%*
	2.7%	*0.41 MPa*	*46%*
Cellulose fiber reinforced silicone	*4.7%*	*0.38 MPa*	*47%*
	24%	*0.43 MPa*	*44%*

The Table 5 shows how each of the fiber influences the tear properties of the silicone. The tear stress of the silicone can be observed to be improved with the addition of the fibers. The tear stress was observed to be the highest in 1.78% fiber volume fraction carbon reinforced silicone. While glass fiber showed a little jump in improving tear stress by just 0.05 MPa with 2.7% fiber volume fraction. Cellulose had very less effect in improving tear properties of the silicone because with the 24% fiber volume fraction it merely improved the tear stress by 0.05 MPa.

However, the tear strain was reduced with the addition of fibers in exception with glass fiber. The tear strain was improved with 0.6% of glass fiber. Even when the fiber volume fraction was increased upto 2.7%, the tear strain just merely took a dip of 5%. Meanwhile with carbon fiber the tear strain took a huge fall. With just 0.66% fiber volume fraction of carbon, tear strain was reduced by 8% and with 1.78% of carbon fiber the tear strain went down by 20%. Cellulose fibers did not show massive changes in the tear strain properties. Even with 24% fiber volume fraction the tear strain was similar as that of pure silicone.

4.3 Interface relation between the matrix and fiber

The rule of mixture generalized above as Equation 12 allowed us to test changes in the Young's modulus of the composite material when the p-value changes from +1 to -1 i.e. from upper bound to lower bound. Table 2, Table 3 and Table 4 show how the Young's modulus increased with each fiber when the fiber volume fraction was increased.
From 25, Figure 266 and Figure 277 the shapes of the fiber can be observed. Glass fibers were observed to have cylindrical shape and a diameter slightly larger than that of carbon fibers. Carbon fibers had a smaller radius than that of glass fiber and were also observed to be cylindrical as well. Though cellulose fiber had more fibers at about 70 microns the fibers had a bended shape.

The figure 28, figure 29 and the Table:1 show the fiber length distribution and the percentile distribution of fibers according to length. Carbon fibers were observed to have almost about 22% of its fiber length in between 100 microns-120 microns. While glass fiber had some very long fibers, most of the fibers were very short. Almost 52% of the total fibers were below the length of 30 microns. Cellulose had almost 20% fibers below 30 microns, 26% between 30 microns- 50 microns and 26% between 50 microns – 70 microns. Fibers longer than 70 microns were rarely occurring in cellulose additive.

Carbon fiber was found to be having the highest p-value of 0.3, glass fiber of 0.07 and cellulose fiber of -0.5. Higher p-value suggests a better reinforcement thus among the fibers that were tested for the experiment carbon fiber was the best choice of all.
In conclusion, the fact that the carbon fiber additive has the large percentual amount of particles which are long rather than short as of in the glass fiber is the reason that the carbon fiber additive used during the experiment resulted in higher p-value i.e. better reinforcement.

The tensile tests and tear tests conducted during this thesis can be summarized with the table below.

Table 6: Tensile test and Tear test results

Tensile Test						***Tear Test***								
Carbon fiber		*Glass fiber*		*Cellulose fiber*		*Carbon fiber*			*Glass fiber*			*Cellulose fiber*		
f (%)	E (MPa)	f (%)	E (MPa)	f (%)	E (MPa)	f (%)	δ_{tear} (MPa)	ε_{tear} (%)	f	δ_{tear} (MPa)	ε_{tear} (%)	f	δ_{tear} (MPa)	ε_{tear} (%)
0	0.98	0	0.98	0	0.98	0	0.39	48	0	0.39	48	0	0.39	48
0.36	1.68	2	3.04	3.5	1.12	0.66	0.45	40	0.6	0.36	51	4.7	0.38	47
1	2.4	7	6.52	10.7	1.23	1.78	0.53	28	2.7	0.41	46	24	0.43	44
1.5	4.65	12	6.33	15	1.55									
1.78	4.77	20	22.12	27	1.79									

For tensile testing, a specimen was made with 0% fiber volume fraction to calculate the Young's modulus of pure silicone and was found to be 0.98MPa. With just 1.78% fiber volume fraction of carbon fiber the Young's modulus of the specimen was observed to be about 4.77 MPa while at 2% fiber volume fraction of glass fiber the Young's modulus just went upto 3.04 MPa. Cellulose fiber was observed to be having least reinforcement with Young's modulus going up to just 1.78 MPa with fiber volume fraction of 27%.

For pure silicone, the tear stress and tear strain were observed to be 0.39 MPa and 48% respectively. However, with addition of fibers the tear stress was improved but tear strain was drastically decreased in some cases. At 1.78% carbon fiber, tear stress went as high as 0.53 MPa while the tear stress took a dip of 20%. However, glass fiber showed improvement of tear strain with 0.6% fiber volume fraction rising the tear strain up to 51% from 48% but in addition of more fibers the tear strain decreased again. Cellulose fiber did not have any significant improvement in both tear stress and tear strain. With fiber volume fraction as high as 24% the tear stress went up by 0.4 MPa and the tear strain fell by just 4%.

5 DISCUSSION

The main objective of this this thesis was to establish if the rule of mixtures is valid for particle fibers and based on the results it seems that it is valid because our model followed the rule of mixtures and we got p-value for each type of fiber. In the results section we discussed how the carbon fiber having more fibers of higher length was contributing to the higher p-value i.e. better reinforcement. However, it is not clear if the thickness of the fibers has anything to do with improving the p-value at all. The fibers we used during this project had very high variation in length. In figure 48, glass fiber had some very long fibers while carbon fiber's mean length was higher. Also, we had only had 8 different specimens for each kind of fiber and 4 different fiber volume fraction which limits how accurately the p-value can be measured. Having more specimen with more different fiber volume fraction could help getting p-value of the fibers more accurately.

While making the mix of silicone and fiber it was mixed in a cup and weighed in a scale of precision of $\pm 0.1 gm$. The syringe in figure 21 was used to inject and measure the volume of the mix. The volume and the weight of the mix were not quite precise which in-turn might have affected how accurately the fiber volume fraction was measured.

The microscopic studies were done with small amount of each different kind of fibers. The fibers that were analyzed in the microscope could not well account for the exact length distribution of the fibers that were used during specimen production. For further studies all the fibers that are used could be measured to get more precise p-value.

For further studies, the mean length of the fibers could be improved by removing the very small particle fibers, more precise equipment could be used for measuring weight and volume of the mix so that more accurate fiber volume fraction of the mix can be calculated and also taking more number of specimens for experiment would help getting more data points meaning more precise p-value.

The industrial relevance of the outcome of this thesis can be very huge. As I conclude on the results section that the higher average length of carbon fibers contributed to the higher p-value, this can be used to choose fibers more efficiently. The average length contributing to higher p-value would mean if we can increase the average length of glass fiber for an example, we can drastically increase the reinforcing properties it. This could help

expensive and more strong fibers to be substituted by less expensive ones just by removing the very small length particle fibers. This would help in using cheaper fibers but get equal or even better mechanical properties than that of expensive ones.

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