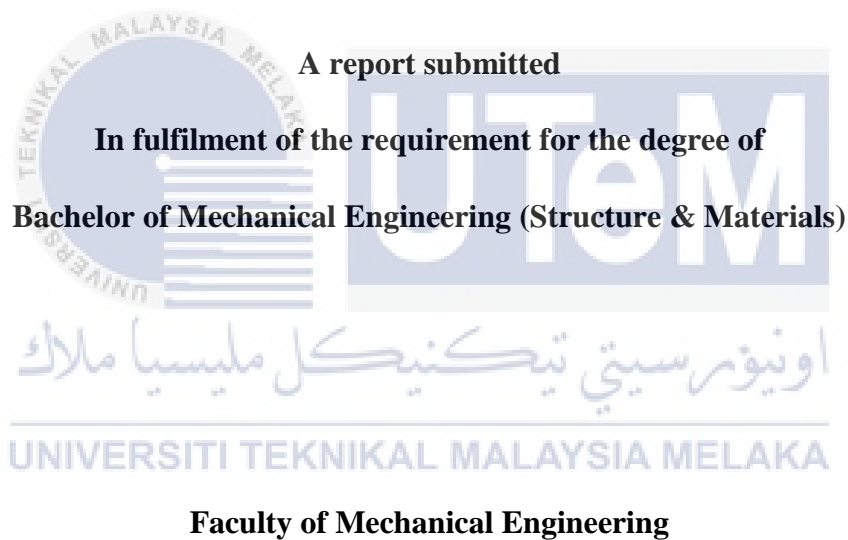


**EFFECT OF SHORT PINEAPPLE LEAF FIBER TREATMENT ON THE
PROPERTIES OF PINEAPPLE LEAF FIBER- STARCH COMPOSITE**

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DECLARATION

I declare that this project report entitled “Effect of Short Pineapple Leaf Fiber Treatment on The Properties of Pineapple Leaf Fiber- Starch Composite” is the result of my own study except as cited in the reference.



Signature :

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SUPERVISOR'S DECLARATION

I hereby declare that I have read this project report and in my opinion this report is acceptable in term of scope and quality of the award of the degree of Bachelor of Mechanical Engineering (Structure and Material).



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ABSTRACT

Nowadays, the natural fiber shown the greater performance in developing biodegradable composite to fix and figure out ecological problem. The result from previous study shows that, the usage of natural fiber has gained attention from industries in producing a new composite material as a replacement to use of synthetic fiber such as carbon fiber, glass fiber and carbon fiber. This is because as a reinforcement material due to their excellent in mechanical properties, eco-friendly, and less expensive. Besides, that why the industries looking deeply to it of this potential of fiber reinforcement composite especially form industries in plastic production. Pineapple leaf fiber (PLF) is one of a natural fiber and is the good to replace synthetic fiber. The usage of pineapple plants is limited only on its fruit. So that, the research come out from the leaf that has been wasted with no used. In this study pineapple leaf fiber (PLF) used as the reinforcement materials and starch (SH) used as a matrix material. The composition of PLF/ SH is 50PLF/50SH, 60PLF/40SH, and 70PLF/30SH. The composition that being selected is 60PLF/40SH. Another than that, the fiber has gone through an alkaline treatment to increase the strength and take out the impurities that contained in PLF. Thus, after done the treatment with the several time. This study is used different time of treatment to investigate the properties of PLF/SH composite. Therefore, eight samples that had been treated with this an alkaline treatment with 2 hours until 16 hours of treatment on PLF before it chopped finest and mixed with SH. Based on the result, the samples with long treatment has highest result of flexural stress which is 3.372 (MPa). Lastly, from the result of SEM analysis shows the structure of PLF/SH is perfect melt together and lowest among of void. This is because both of PLF/SH is mixed well and become homogeneous during fabrication process.

ABSTRAK

Pada masa kini, serat semula jadi menunjukkan prestasi yang lebih besar dalam membangun komposit biodegradasi untuk membetulkan dan memikirkan masalah ekologi. Hasil daripada kajian terdahulu menunjukkan bahawa penggunaan serat semula jadi telah mendapat perhatian dari industri dalam menghasilkan bahan komposit baru sebagai pengganti penggunaan serat sintetik seperti serat karbon, gentian kaca dan serat karbon. Ini kerana sebagai bahan pengukuhan dan kerana sifatnya yang sangat baik dalam sifat mekanik, mesra alam, dan lebih murah. Di samping itu, industri-industri juga melihat potensi komposit tetulang serat ini terutamanya industri dalam pengeluaran plastik. Serat daun Nanas (PLF) adalah salah satu serat semula jadi dan sangat baik untuk menggantikan serat sintetik. Penggunaan tumbuhan nanas hanya terhad kepada buahnya. Jadi, penyelidikan itu keluar dari daun yang telah dibazirkan tanpa digunakan. Dalam kajian ini serat daun nenas (PLF) yang digunakan sebagai bahan pengukuhan dan kanji (SH) digunakan sebagai bahan matriks. Komposisi PLF / SH adalah 50PLF / 50SH, 60PLF / 40SH, dan 70PLF / 30SH. Komposisi yang dipilih ialah 60PLF / 40SH. Selain itu, serat telah melalui rawatan alkali untuk meningkatkan kekuatan dan mengeluarkan kekotoran yang terkandung dalam PLF. Oleh itu, selepas melakukan rawatan dengan beberapa kali. Kajian ini menggunakan masa rawatan yang berbeza untuk menyiasat sifat komposit PLF / SH. Oleh itu, lapan sampel yang telah dirawat dengan rawatan alkali iaitu dengan 2 jam sehingga 16 jam rawatan pada PLF sebelum ia dicincang dengan baik dan dicampur dengan SH. Berdasarkan hasilnya, sampel dengan rawatan panjang mempunyai hasil tertinggi tekanan lentur iaitu 3.372 (MPa). Akhir sekali, dari hasil analisis SEM menunjukkan struktur PLF / SH cair dengan sempurna antara satu sama lain. Ini kerana kedua-dua PLF / SH bercampur dengan baik dan menjadi homogen semasa proses fabrikasi.

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LIST OF ABBREVIATION

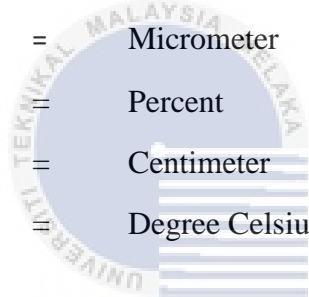
PLF	=	Pineapple Leaf Fiber
PP	=	Polypropylene
FRM	=	Fiber Reinforced Plastic
PMC	=	Polymer Matrix Composites
MMC	=	Metal Matrix Composites
NaOH	=	Sodium Hydroxide
CMC	=	Ceramic Matrix Composites
SH	=	Starch
FRP	=	Fiber reinforced Polymer
SEM	=	Scanning Electron Microscope

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LIST OF SYMBOLS

MPa	=	Mega Pascal
GPa	=	Giga Pascal
m	=	Meter
kg	=	Kilogram
g	=	Gram
/	=	per
mm	=	Millimeter
μm	=	Micrometer
%	=	Percent
cm	=	Centimeter
$^{\circ}\text{C}$	=	Degree Celsius



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CHAPTER 1

INTRODUCTION

1.0 Background

A composite is a material from two or more material to produce a new material that has a new or improved strength ability from its original individual components. Basically, most of the materials that exist, or we see is made of a composite material. For examples, bones, wood and stone are natural composite items that develop by a natural process. The leaf itself consist of natural fiber and usually use in making a new composite material because of their unique characteristics which have a good mechanical property, stronger, lighter, biodegradable and less expensive compared to the synthetic fiber. Because of that, natural fiber has potential to be an alternative to synthetic fiber such as glass fiber and carbon fiber.

There are a few examples of natural fibers that can be extracted from a plant such as pineapple leaf, banana leaf, palm leaf, hemp, kenaf, bamboo and coconut shell fiber. Furthermore, all natural fiber, Pineapple leaf fiber (PLF) seems to have the highest cellulose content which makes the fibers can produce good mechanical properties. In order to give the unique ability for natural fiber, binder such as the starch composite are added to enhance the existing mechanical properties or and other words called as a reinforcement of the materials.

Reinforcement mean is strengthening the structure or material itself. For example, back in ancient Greek civilization years, clay was reinforced by the straw to build walls. In this case, clay will become the binder holding the straw together thus, make the construction become stronger.

As the previous studies that fiber reinforced plastic (FRP) is a very well-known composite that being used in structure engineering, mostly in the field of aerospace, building and offshore platforms. This is because, there are considered to have more strength, non-corrosive, light in weight and most important is easily moulded or constructed. But the materials or fibers are usually from the glass and carbon combining with the plastic polymer as the binder. While in this study, PLF will be used as the reinforce materials and starch as the binder which may potentially give a good result in mechanical properties, besides it characteristic which is an environmentally friendly, renewable, recyclable and biodegradable. Fibers can be altered by alkaline treatment. In general, alkaline treatment will improves surface roughness and increases the number of celluloses on the surface of fibers. Therefore, Pineapple leaf fibers are conducted with alkaline compound to improve their physical and mechanical properties. In fact, it will exceed mechanical interlocking. Furthermore, in previous studies have collected various trials elaborating with alkaline treatment for natural fiber. Atiqah et al. [4] evaluate the kenaf fiber with 6% sodium hydroxide (NaOH) compound for 3 hours and showed excellent outcome for flexural, tensile and impact strengths. Claudia Merlini et al. [5] experiment alkaline treatment on banana short fibers with 10% NaOH solution for 1 hour.

1.1 Problem Statement

Nowadays, the usage of natural fibers as an alternative reinforce in composites materials are still in research phase. Some of the problems arise is that synthetic is widely use are hardly to decompose and are not sufficiently eco- friendly. Besides that, by using natural fibers such as pineapple leaf, hemp, kenaf, and jute fibers with certain type of binder to create a composite material seems can be compete with existing synthetic composites which they have a good mechanical property. Just at a certain time, natural fibers have been awakening the industry to substitute their products by using the natural composite. For example, it has been widely used in the automotive industry. Thus, by using the natural fibers they can produce higher strength of automotive interior components such as dashboard, door trim, but cheaper in price. The achievement of natural fiber and fiber reinforce composites. The chemical alteration of natural fiber such as alkaline treatment has acknowledged various levels of success in improving fiber strength in nature fiber composite. An Alkaline treatment of pineapple leaf fiber is commonly method that often used by some researches produce a high-quality fiber for reinforcement materials. The alkaline treatments showed improved behaviour in mechanical properties as compared to untreated fibers. Panyasart et. al [6] attempt test on pineapple leaf fiber (PLF) with 5% NaOH compound and 5 hours engagement period at room temperature. Previous studies by Asim et. al [2] on alkali treatment for pineapple fiber exhibit reinforce in mechanical properties for fibers treated with 6% NaOH. The alkaline treatments showed improved behaviour in mechanical properties as compared to untreated fibers.

In this project, the aim is to study the effect of Short Pineapple Leaf Fibers Treatment on the properties of pineapple leaf fiber (PLF). Starch composite was used as the reinforce material. The various ratio of PLF/SH composite was be selected and the ratio of composition in the PLF/SH composite was fixed at, 70:30, 60:40, 50:50. An alkaline

treatment will be conducted with various hours (2, 12, 24) to extract thin PLF bundles and enhance the PLF properties before the formation process of PLF/SH composite used hot press. The test that will be covered used tensile test, flexure test, hardness test, density measurement and macrostructure analysis. The composite seems to have a good potential that can widely use in industry like for an example for the plastic industries product more benefit to the environment.

1.2 Objectives

The objective of this project is:

1. To determine the effect of Pineapple Leaf Fiber (PLF) treatment on the properties PLF/ Starch (SH) composite.
2. To study the effect of PLF loading on the properties of PLF /SH composite.

1.3 Scope Of Project

This research studied the effect PLF loading on the mechanical properties of PLF/ SH composite had been carrying out. The various ratio of PLF/ SH composite was be selected and the ratio of composition in the PLF/ SH composite was fixed at 70:30, 60:40, and 50:50. An alkaline treatment will be conducted with various hours (2, 12, and 24) to extract thin PLF bundles and enhance the PLF properties before the information process of PLF/ SH composite used hot press. The mechanical properties of PLF/ SH composite will be determined used tensile test, Flexure test, hardness test, density measurement and macrostructure analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Natural fibers composites have gained a reputation in renewal the synthetic fibers such as glass fibers reinforced composites that commonly known to their non eco-friendly to the natural system. There are examples of natural fibers can be from a numerous source such as pineapple leaf, bamboo, banana leaf and kenaf [1].

Biodegradable fiber- reinforced polymer (FRP), is a composite material that produce of a polymer matrix reinforced with fiber. There a lots usage or various of nature fiber. Many industries are interested used in development for their product of natural fiber composites. There are numerous types used to build up of natural fiber composite such as bamboo, coconut, rice husk, wood and pineapple leaf. Biodegradable composite material or Bio composite is a composite material shaped by a grid and support of common fiber. Biocompatibility is linked to the action of biomaterial in various contexts. The capability of a material to act with an appropriate host response in a precise position [29].

The polymer is a macromolecule that composed of many bounding materials. Commonly, an epoxy and polyester thermosetting plastic are the normally favourite choice to used. Biodegradable fiber- reinforced polymer (FRP) is often used in automotive, aerospace, marine and construction industries. There are two type of fiber which is being used for reinforced the composite materials.

- i. Synthetic fiber
- ii. Natural fiber

Generally, synthetic fiber are using to reinforce plastic due to superior performance of mechanical properties and low cost of production but it very worth it. However, synthetic fiber have big significant as high energy consumption, exposed to damage by hot washing, non- renewability and high density. Furthermore, different side with fiber reinforced polymer composite which is, it earned the world-wide attention due to high specific strength and modulus. In addition, material composite that have great strength fiber such as glass and graphite are commonly used in aerospace, automotive components are highly expensive cost to produce. This condition or standpoint will lead the industry to use of the other option materials composites.

Natural fiber has create a huge ability to replace for example glass fiber in composite due to more economical characteristic and good mechanical properties compare to synthetic fiber [30].

A composite material is a constituent material that made from two or more micro or macro material with different chemical and physical properties. Natural fibers can help to develop the mechanical properties of a product since it has profit to environment. In addition, the comparison price between the natural fibers are more economical compared to synthetic fiber such as glass fiber and carbon fiber that have been extensively used in the industries.

In this literature review, pineapple leave fiber starch (PLF/ SH) composite is used with the various ratio was be selected fixed at 70:30, 60:40, and 50:50 and with an alkaline treatment will be conducted with various hour (2, 12 and 24).

2.1.1 Types Of Composite

There are there types of composite depend on their matrix type. These are known by their natural behaviour and the properties. These consist of Metal Matrix Composites (MMC), Ceramic Matrix Composites (CMC) and Polymer Matrix Composites (PMC).

2.1.1.1 Metal Matrix Composites

Metal matrix composites are commonly used in the production of chamber nozzle for aircraft applications, tubing, cables, heat exchangers, space shuttle, automotive industries and structural members. This is due to the properties of the metal matrices that have higher strength, fracture toughness and stiffness. Besides, metal also have the qualities that can withstand high temperature in corrosive environment compared to the polymer composites [12].

2.1.1.2 Polymer Matrix Composites (PMC)

Generally, the strength and stiffness of a polymer is low compared to the metal and ceramic, but these complications had been overcome by reinforcing them with other materials. However, the process for producing the composites much simple and cheaper compared to other types of composites. This make the polymer matrix composites gained it demand in the industries [12].

2.1.1.3 Ceramic Matrix Composites (CMC)

The primary goals in producing the ceramic matrix composites is to increase strength or toughness of a materials. Normally it is found that there is a good outcome in the improvement in strength and stiffness of a material by using ceramic matrix composites [12].

2.2 Reinforcement

Reinforcement produce strength and rigidity, helping to support structural load [23]. Based on a journal, fiber-reinforced polymeric composites have gained so much acclaim because of their great mechanical properties like high specific strength and modulus [20]. Nowadays, natural fiber is used as a good restoration of synthetic fibers as reinforcement in plastic to reduce cost, increase the productivity of material and to improve mechanical properties of a product. The famous examples of the natural fibers such as rice straw, wood, bamboo, hemp and others [21].

2.2.1 Natural Fiber

Natural fiber-reinforced polymer composites have gained an excellent reputation among the engineers and material scientists in these days because of the ability of the composites to produce great mechanical properties, dielectric properties and giving many advantages to the environment such as it is renewability and biodegradability. Besides that, by using these natural fibers, many environmental problems can be solved. These composites are also can be well used as a wood replacement in the construction industry. Furthermore, natural fibers have raised an attention due to various disadvantages of the conventional petroleum-based plastic,

glass or carbon fiber that not an eco-friendly, very expensive and must use high progressing technologies. There numerous natural fibers that are used as reinforcement of polymer composite such as the pineapple leaf, bamboo, jute, banana, and coir [22].

There are few types of natural fibers, for example is the lignocellulosic fiber. The fibers are held by binder agents called “lignin” and “hemicellulose” in the fiber cell. The fiber also can be found on the outer layer of the fiber bundles and leaves. The fiber cells are structured in different layers, formed typically by groups of Nano-scale cellulose chains extending helically along the axis of the fiber cells and interconnected by amorphous regions composed of lignin and hemicellulose [25]. On the other hands, for vegetable fibers which are considering to be more complex because they are construe by the wide variety of organic compound in the fiber such as the lignin, hemicellulose, fatty acids, fats, waxes and many more [24].

Moreover, natural fiber as widely spread in many industries such as the building industry. This is because of the characteristics of natural fiber that is good in thermal insulation. The purpose why the natural fiber is used because of it is an environmentally friendly, energy saving and giving a long term of favour to the aspects of financial as it is low cost and does not requires skilled labour and not harmful to the human health compared to commercial thermal buildings insulators that mostly made from minerals wools, glass foam, and rock wools [26].

2.2.1.1 Pineapple Leaf fiber

Nowadays, natural fibers have got many intentions among the researchers as it has high potentials in replacing the synthetic fibers in fiber-reinforced plastics. The

most reason of this replacement is because of natural plants fiber that used as reinforcement to plastics are low in cost, can save much more energy during the production, have lower density, no interference to the processing machines, and not dangerous for the consumers (health). Thus, it is also easy to get, renewable, recycle and biodegradable [27]. There are so many types of natural fibers that can be found, pineapple leaf fiber (PLF) believed to have the highest cellulose contents which make the fiber have an excellent mechanical property [18].



Figure 2.1: Pineapple Plant

Essentially, the pineapple leaf fiber is taken from the leaf of pineapple plant.

In addition, in Malaysia, pineapple is one of the plants that widely available in the market. But the usage of the pineapple plant is not totally used as in Malaysia or other countries, they focus is only on the fruits. However, the other part of the plant wasted with no use [16]. But recently, in previous studies have found the benefits, superiority and the potentials of the pineapple leaf fiber as it can help to reduce the pollutions and save the environmental. This is because mostly of the wastage of the natural resources will be burn [15]. In Malaysia, there are three types of pineapple cultivars and each had shown various physical properties. Table 2.1 shows the three types of pineapple cultivars and their physical properties [19].

Table 2.1: The types of pineapple cultivars and physical properties [19]

Property	Cultivar		
	Moris Gajah	Josapine	Sarawak
Length of leaves (mm)	63	61	70
No. Of fiber bundles per leaf	90	80	>100
Diameter	120- 440	105- 300	170- 340
Average no. of leaves (mm)	50	65- 70	65- 70
Width of leaves (mm)	61	46	65

In recent past, many researchers have seen the capability of the PLF and many studies have been carried out to investigate many aspects of the PLF. For example, an experiment has been conducted where to study the tensile properties of fibers from the pineapple leaves, betel nut fruits and bark of lady fingers. It is surprisingly shown that PLF have the highest tensile strength and the elongation at break. [14]

Moreover, the mechanical properties of the PLF is mostly be studied by using them as a reinforcement material to a composite which is combined with certain types of binder like polypropylene, polyethylene, polycarbonate and polyester. The composites usually were evaluated by the fiber modifications, fiber length and fiber loading. However, the studies showed the mechanical properties of a pineapple leaf fiber may not good as other composites as it may vary but it must be remembered that there are different conditions that must be considered such as the plant types, age, locations of the plants [12]. Figure 2.2 shows the pineapple leaf fiber (PLF) and table 2.2 shows the properties of the pineapple leaf fiber.



Figure 2.2: The Pineapple Leaf Fiber (PLF)

Table 2.2: The Pineapple Leaf Fiber properties.

Property	Value		
	George et al. [12]	Mohanty et al. [18]	Arib et al. [20]
Moisture regain (%)	12.0	11.8	-
Microfibrillar angle (°)	-	14	-
Diameter (µm)	-	20- 80	-
Elongation at break	3.0	1.6	2.2
Young' Modulus (GPa)	6260	34.5- 82.5	4.405
Tensile strength (MPa)	170	413- 1627	126.60
Lignin content (%)	-	5.0- 12.7	-
Cellulose content (%)	-	70- 82	-
Density (g/cm³)	1.526	-	1.07

2.2.2 Carbon Fiber

Carbon fiber is one of the build-up materials that have been widely used in order to make a composite material. Although polymers are always used a binder for the carbon fiber, non-polymer materials also can be used in order to act as a binder or matrix for the carbon fiber [6]. Carbon fiber is also known with their very light in weight and extremely strong material. Because of the fiber property that basically a very thin strands of carbon, it can be twisted and be woven together. This will help the carbon fiber act to be stronger than steel but lesser in weight [9]. Carbon fiber can be known as a material that have high-performance in mechanical properties that have so much functionality. Carbon fibers are also can be classified by the tensile modulus of fiber. This can be measure by referring to a certain size of the fiber

diameter with how much tensile force that being exerted to the fiber without breaking it. Thus, usually the diameter of carbon fiber is from 0.005 to 0.010 mm as it can be thinner than the human hair. The masterpiece of the carbon fiber are the mechanical properties which are consists of the very high tensile strength and elastic modulus, good electrical and heat conductor. Furthermore, it can withstand to harsh surroundings and have very light in weight make them a good material to produce many types of component such as for automotive industry, aerospace industry [6]. Figure 2.3 below show the example of carbon fiber.

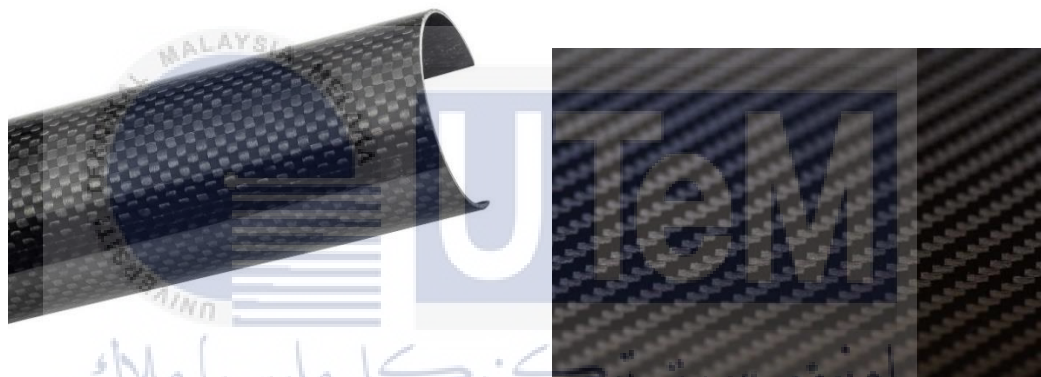


Figure 2.3: Carbon Fiber

2.2.3 Alkaline Treatment

Pineapple leaf fiber (PLF) is tough to extract due to the outstanding mechanical properties. The fiber in PLF is covered with lignin, thus PLF is always brittle compared with another natural fibers. Therefore, the extraction of the PLF reinforcement of composites materials should be support by a devised process [1]. An Alkaline treatment has a lot of advantages. Some of the advantages of the alkaline treatment is the moisture content in the fibers will be removed to increase the strength

of the PLF. After alkaline treatment has been done, PLF can be used as a filler, then rolling mill machine is used to cleave the first longitudinal of the raw PLF. Moreover, NaOH solution is used in this research. NaOH solution also had 1% degree of concentration. The orientation of the molecular will be stable and all the impurities which are adjoining the fiber material also will be clears filter after alkaline treatment are done [2].



Figure 2.4: PLF on alkaline treatment

From the Figure 2.4 above representing about (a) pineapple leaf, (b) extraction of fibre from pineapple leaf, (c) pineapple leaf fiber subjected to an alkaline treatment, (d) pineapple leaf fiber being dry after the treatment.

2.3 Binder/ Matrix

There are plenty types of polymer that can be used as a matrix to reinforce the fiber to make a composite material. The main purpose of a matrix/ binder is to transfer load acting to the fiber [4]. In this project, a natural polymer which is starch (SH) is used as the matrix in reinforcing the pineapple leaf fiber (PLF).

2.3.1 Starch (SH)

Starch is known as a biodegradable in a variety of environments and considered as a very good stuff to develop sustainable materials. Starch is one of the better natural polymers that can be found in a lot of sources mostly from corn, potato and other crops. Starch is a renewable polymer and fully biodegradable and can be obtained in very low in cost [11].

Besides that, the effect of the matrix on the properties of the reinforcement and fabrication method will be affected the selection of the matrix. The starch-based biodegradable polymers that have played a performance in medical field. Thus, with the excellent mechanical properties, the products are non-toxic, and good biocompatibility made this polymer can be used as bone plaster (cements) that provide support and can also be used as bone tissue engineering scaffold [28, 8]. Figure 2.5 shows the example of starch.



Figure 2.5: Example of Starch

2.4 Fiber Size

Based on previous study, the fiber size is used based on length of the fiber. The pineapple leaf fiber is cut into pieces with a measurement are 2 cm, 4 cm, 6 cm [1]. But in this study, the size of pineapple leaf fiber is fixed at several ratio at 70:30, 60:30, and 50:50 which chopped into small pieces which is 3 cm for each testing. We assume that, the smaller size has better PLF properties compared to the long fiber orientation.

2.5 Fiber Treatment

Before the reinforcement materials state on pineapple leaf fiber is accomplished, the fiber was gone through a surface treatment. A treatment that used to treat the fiber is an alkaline treatment because it has a good chemistry and it is easy to conduct. Besides, the other things to produce the PLF composites is used of chemical. As was I saying, the chemical that used for this treatment is Sodium hydroxide (NaOH) in a form of pallets. Based on the previous study, by conducting a surface treatment it influences the fiber matrix bond. Such as strength, improved the mechanical properties, flexural properties and fiber surface area. Moreover, each sample was treated with different absorption or combination of an alkaline which is 3%, 5%, and 10%. The PLF absorbed on the NaOH concentration solution for about 30 minutes. But in this study an alkaline treatment will be conducted with 4 hour and with same absorption of the PLF immersed only on the 5%. Thus, with 4 hours' time on each treatment and will got the six result or treatment on the fixed PLF/ SH composites ratio. After that, the fiber will be washed with distilled water. Figure 2.6 shows the sodium hydroxide pallet.



Figure 2.6: The sodium hydroxide pallet.

2.6 Fiber Testing

Based on a journal, before conducting a testing on a composite, the PLF and starch are mixed together before been pressed by using hot press machine at a 60 °C of temperature and under a pressure of 25 kg/cm² for about one hour. Before the sample is taken out from the mould, the sample is let to be rest/ cool at a room temperature. Then go through several mechanical tastings that are based on the American Standard Testing Method (ASTM) which is the tensile test (ASTM D 3039/D 3039M-00), shore hardness test, density measurement and microstructure analysis [1]. But on this study, tensile test will be replaced with another mechanical testing which is the flexural test (ASTM D790-03).

2.6.1 Tensile test

Tensile test is the test method use in order to identify the tensile properties for polymer matrix composite materials. The tensile test operates basically referred ASTM D3039 standard test. Based on the orientation of the materials test specimen will have a certain fixed sizing and it normally in rectangular shapes.

This testing method is carried out in order to achieve the value of the ultimate tensile strength, ultimate tensile strain, transition strain [ASTM D3039], tensile chord modulus of elasticity and Poisson's ratio. The specimen will monotonically increase load in tension until the specimen ability reach it failure. The ultimate strength can be received from the maximum load carried before it breaks, if the strain gauge is applied in the specimen the value of strain can be determined. Figure 2.7 shows the Instron Universal Testing Machines that used in this experiment. The requirements of the specimen shape, dimensions and tolerances have shown in Table 2.3 [7]. Table 2.3 show the Tensile Geometry Requirement [ASTM D3039].

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Figure 2.7: The Instron Universal Testing Machines

Table 2.3: Tensile Specimen Geometry Requirement [ASTM D3039]

Parameter	Requirement
Coupon Requirement:	Constant rectangular cross-section
Shape	Gripping +2 times width + gage length
Minimum length specimen width	As needed
Specimen thickness	$\pm 1\%$ of width
Specimen thickness tolerance	As needed
Specimen flatness	$\pm 4\%$ of thickness
	Flat with light finger pressure
Tab Requirements (if used):	As needed
Tab material	As needed
Fiber orientation (composite tabs)	As needed
Tab thickness	$\pm 1\%$ -tab thickness
Tab thickness variation between tabs	5 to 90°, inclusive
Tab bevel angle	Feathered without damaging specimen
Tab step at bevel to specimen	

2.6.2 Flexural test

The objectives of this test are to identify the material bending properties. In this test, the flexure will be tested as a simple beam support at two points and being loaded at the midpoint. This flexural test is based on the standard of ASTM D790-03. The still conduct with the same rectangular shape of the test specimen. The specimen will be clamped on a flexural testing machine with the cross section of sample on two support that given the force on the middle part of the sample. Figure 2.8 shows the diagram of the flexural test machine.

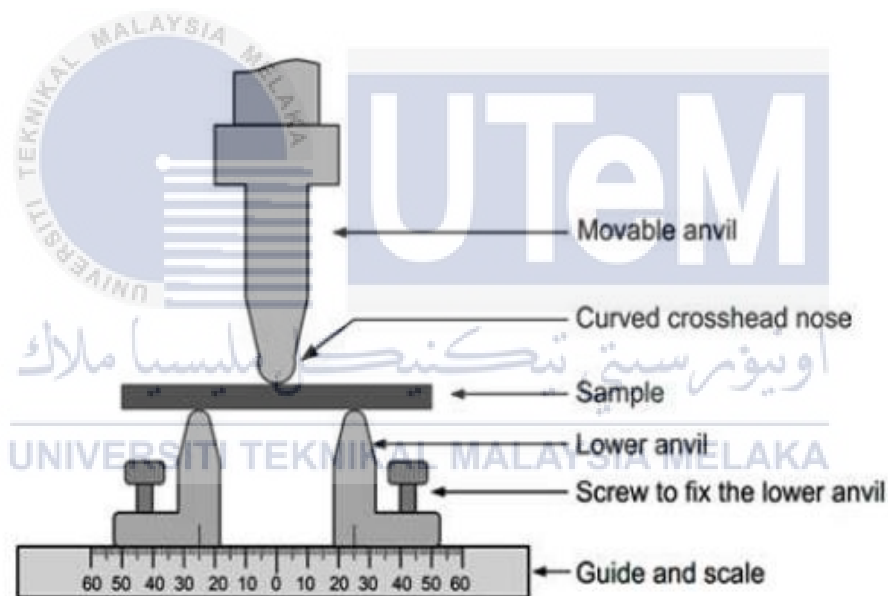


Figure 2.8: The diagram of the flexural test machine

The maximum stress that exist on the midpoint can be calculated by using this equation [ASTM D790-03].

$$\sigma = \frac{3FL}{2bd^2}$$

Where:

σ = stress in the outer fibers at midpoint, MPa

P = load at a given point on the load-deflection curve, N,

L = support span, mm,

b = width of beam tested, mm, and

d = depth of beam tested, mm

2.6.3 Hardness test

The hardness test of the PLF/SH composite, an Analogue Shore Scale “D” type Durometer is used for measuring the depth of indentation by indenter of the material based on the reading on the scale of the device. Figure 2.9 shows the shore hardness tester.



Figure 2.9: The shore hardness tester Analogue Shore Scale “D” type Durometer.

2.6.4 Density test

In this section, a Digital Electronic Densimeter (MD- 300S) as shown in Figure 2.10 is using to measure the density and specific gravity of the PLF/SH will be based on standard on the [ASTM D792]. Moreover, this standard can also be used for testing solid plastic in water and for testing solid plastics in liquid other than water [ASTM D792].



Figure 2.10: Digital Electronic Densimeter (MD- 300S)

2.6.5 Macrostructure Analysis

Based on the previous study, the macrostructure analysis is done by using the Environmental scanning electronic microscopy (ESEM) [2]. But in this experiment, to performed macrostructure analysis using Scanning Electron Microscope (SEM) to study the macrostructure composite. Figure 2.11 shows the Scanning Electron Microscope.



Figure 2.11: The Scanning Electron Microscope (SEM).

CHAPTER 3

METHODOLOGY

3.1 Experimental Overview

The target in this chapter is making and accomplish the objectives that going to achieved. A several procedures must be taken in order to reach all the objectives stated and make the project run smoothly and successful. Based on the flow chart below, the first step that must be consider is to determine what types of materials that must be used or in other words the selection of materials that want to be used. In this project, two major components are selected and consist of the reinforcement material and the matrix/binder. Second steps are preparation of all materials along with fiber and matrix/ binder or starch (SH) composite. Next, the process continues to chopped and sieving process to form the materials into a certain condition. From this aspect, to determine the particles size distribution of ta materials. The process separated from more course particles materials through several different sizes. After several suitable compositions are found, the process will proceed to the fabrication process and a few samples will be made before cutting process is done. Then, testing will be done to the several samples before collecting the data. The detail on this study will be overview in this chapter and Figure 3.1 below shows the flow chart of the process.

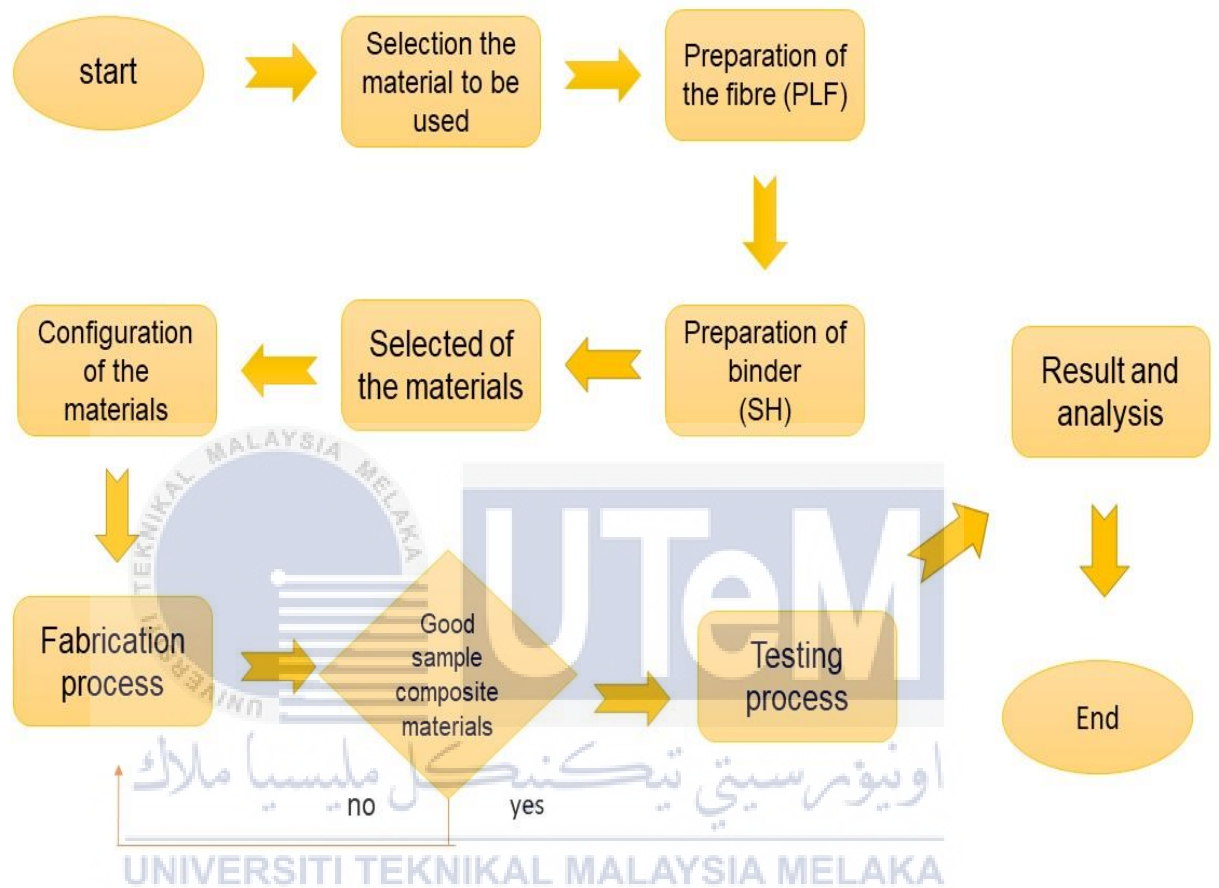


Figure 3.1: Flow chart of the PLF/ SH composite process.

3.2 Materials To Be Used

To figure out the concept in selecting the materials that will be used is very important in this experiment since the material selection is a very significant process in any Engineering application. As was I saying, this is because all the specification that want to be achieved are based from materials selection and the type of materials that want to be tested.

For instead, since in this project we used the composite of the material selection, the method that can be done for an example are on the composition of the selected materials to form them into a composite material. Besides, on this project composite materials are using combination of a natural fiber which is Pineapple Leaf Fiber and the starch composite. Hence the selection of those two parts are very significantly to conduct in this project.

Basically, there are many natural fibers that can be found such as pineapple leaf, kenaf, banana leaf, and other. In this project, will be focusing on the type of the natural fiber Pineapple Leaf Fiber (PLF) and the study will to understand principal of the selection natural fiber. The project will approach to the mechanical properties of PLF/ST composite and will determined used tensile test, flexure test, hardness test, density measurement and microstructure analysis.

A matrix also must consider as it acting as a major role to transfer the load or force acting on the fiber. This requires the selection of the matrix components should have a good adhesive property as it will help the fiber to retain the composite material shape or properties from their strong interface between the fiber and the matrix [1].

3.3 Preparation of Materials

In this project, the various of selection natural fiber was being study and pineapple leaf fiber was selected as the reinforcement materials and starch (SH) was used as the binder/ matrix. Pineapple leaf fiber have a good characteristic and it been chosen as it believed that is one of the fibers have substance with highest cellulose which produce the fiber very good in mechanical properties [3]. After that, materials preparation was being selected with a fixed ratio of composition in the PLF/ SH (70:30, 60:30, 50:50). The mould that be used is rectangular shape with dimension 140mm X 250mm. Then formulation process of PLF/ SH composite used hot press.

3.4 Processing Method

In this part, the first method will be start extraction method, the pineapple leaf fiber will be going through a several methods from the formation of the fiber which consist of the chemical treatment of the PLF with alkaline treatment based on each sample behaviour matrix/ binder preparation, the compression moulding and lastly testing process which is classify or analyse the effect of pineapple leaf fiber loading on the properties of PLF/ SH composite.

3.4.1 Appropriate Parameter

The next step has been taken to give appropriate parameter of hot compression moulding process for starch powder. In order to obtain a good sample, the first step must to know the melting point of the starch powder and then followed by the pressure of compression moulding. By using 250mm X 140mm mould the

PLF and starch are put into the mould then being heated by using the hot press machine.

Firstly, the sample have been on hot compressed with the temperature of 155 °C with pressure 100 psi for compression about 20 minutes and pre- heating time of 10 minutes. Secondly, the next trial is by increasing the range temperature is about 165 °C with the temperature is also increase by 300 psi. The third trial also same with the second, by increase the temperature and pressure but with different value which is 175 °C and 500 psi.

Three same composition with different parameter will be test on this experiment. Several trials have been conducted for an example the sample of this parameter will be discuss more further in next chapter for the result of PLF/ SH. Table 3.1 shows the parameters that have been used in order to find the best sample by using 70/30 composition of PLF/ SH as the sample.

Table 3.1: The sample parameter of 70/ 30 PLF/ SH composite PLF/ SH

Sample	Pressure (psi)	Temperature (°C)	Pre- Heat Time (minutes)	Compress Time (minutes)	Composition PLF/ SH (%)
1	100	155	10	20	70/ 30
2	300	165	20	30	70/ 30
3	500	175	30	60	70/ 30

3.4.2 Fiber Preparation

3.4.2.1 Alkaline Treatment

The PLF will have treated with alkaline treatment with chemical assistant which is by using sodium hydroxide (NaOH). Based on the previous study, the natural fiber PLF was immersed into the alkaline solution with 5% of concentration of (NaOH). The objective that should be done in this section is to removes al the impurities on the PLF and on the same time it also will helps to improves the sticking property among the fiber itself. This could be easier for cleaning process. Figure 3.2 and Figure 3.3 shows the sodium hydroxide (NaOH) and PLF before being operating.



Figure 3.2: The sodium hydroxide (NaOH)



Figure 3.3: The PLF before being operating.

Based on the journal, by operation with alkaline the fiber also helps to improve the mechanical properties [1]. In this current study, the PLF will be attempt in alkaline solution for about several hours which is (2, 12, 24 hours). Then PLF being neutralized by using distilled water. Besides, the process of washing is repeated for several time to make sure all the impurity had been removed. Figure 3.4 shows the PLF attempt with alkaline solution.



Figure 3.4: The PLF attempt with alkaline solution.

Next, drying process which is the PLF will dry in the oven at 60°C for 4 hours. Later, the fiber will be cooling down at room temperature until it totally dry. The excess cellulose fiber is removed manually, and it could be easy if the PLF is completely dry and do not have any moisture on it. Figure 3.5 shows the PLF after completely dry.



Figure 3.5: The PLF after treatment and be dry in room temperature.

3.4.3 Compression Moulding

Since the composition have been determined at the beginning of this project, it will be fixed. Thus, knowing the dimension of the mould that being used is 250mm X 140mm. Figure 3.7 shows the dimension of the mould used. PLF/ SH were mixed together and heated before formation process. After that, PLF/ SH with different size or ratio will be put into mould. Then, it will be attempt on hot press machine. Furthermore, 175°C with pressure 500 psi in this testing needed for this process in hot press machine. However, it needed to preheat is about 30 minutes and the compression time was 60 minutes. Figure 3.6 shows the hot press machine. Table 3.2

shows the composition of the two element which are PLF and SH powder in the form of percentage ratio before mixing.

Table 3.2: Composition of the PLF/ SH

Composition (%)	Total weight of the sample (g)	PLF (g)	SH (g)
50 /50	80	40	40
60/ 40	80	48	32
70/ 30	80	56	24



Figure 3.6: The hot press machine

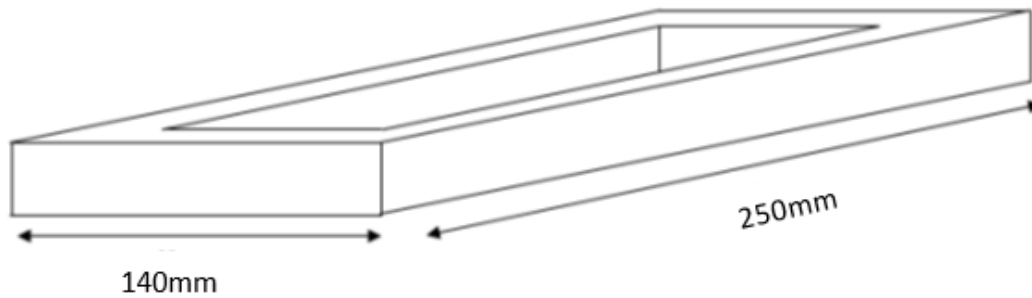


Figure 3.7: The dimensions of mould

After the compression hot press done, the mould straight gone to the cooling process for 30 minutes at the cooler machine before the sample take out from mould. This process is repeated to all composition of PLF/ SH composites.

3.4.4 Cutting Process

After the fabrication process, the next process is cutting process which is in this section the PLF/ SH composite sample will be cut into a specified dimension based on the cutting machine tool ASTM D 3039/d 3039M-00 before going through the mechanical testing. Figure 3.8 shows the Proxxon Table Saw being used to cut all the samples.



Figure 3.8: The Proxxon Table Saw

3.5 Mechanical Testing

To establish the result of the effect of the pineapple leaf fiber and starch loading that act as a particle, there are few testing methods that were used in this project. Remarkably of the methods will be assign to the American Standard Testing Methods (ASTM) which is an international standards organization that develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems, and services. Especially ASTM D790-03 for Flexural Test and other mechanical testing that will be enforced are the hardness test, density measurement and microstructure analysis.

3.5.1 Flexural Test

The flexural test method will measure the behaviour of a materials that has been impose to a beam loading. In this flexural test, there are two types, which is the 3-point flex and 4- point flex [1]. From this study is based on the ASTM standard for a flexural test. Figure 3.9 shows the example of a specimen that contact with the 3-point flexural test.



Figure 3.9: The example of a 3- point flexural test machine.



Figure 3.10: The ASRM D790 model

3.5.2 Hardness Test

In this test, the hardness of the composite materials of PLF/ SH are tested.

Generally, hardness test is the method to implies or measure the resistance to the total deformation of a materials [13]. The shore hardness tester is used to measure the hardness test of the specimen. Figure 3.11 shows The Shore Hardness tester devices. The analogue shore scale is pressed into the sample and the reading is taken by indicated the red scale on the Analogue Shore scale. The steps were repeated with the other samples of different formation.



Figure 3.11: The Analogue Shore Scale device

3.5.3 Density Measurement

In this section, the density was performed by using Electronic Densimeter (as shown in figure 3.12). The objective in this study is to measure the specific gravity or density of the materials which us the PLF/ SH composites [17]. The testers or PLF/ SH was put inside the container and the mass of the specimen is measured. Then, the specimen was put inside water. Furthermore, the specific gravity and volume of the specimen are automatically being measured. All the steps were repeated for each sample and the data being collected.



Figure 3.12: The example of Electronic Densimeter MD-300S

3.5.4 Macrostructure Analysis

The purpose of macrostructure analysis is to investigate the behaviour of the microstructure of the PLF/ SH composites. The examples of the macrostructure are likely finding the defect, discontinuity of PLF and SH and voids of the materials. The qualified tool to run the macrostructure analysis is been used which is the Scanning Electron Microscopy (SEM). Figure 3.13 shows the specialised tool of the macrostructure analysis.



Figure 3.13: The Scanning Electron Microscopy (SEM)



Figure 3.14: The PLF fiber after coating

There are few steps must be taken to complete this method. Firstly, connected the microscopy (SEM) to the computer/ laptop. Secondly, the specimen is put under the lens of the microscope and running the analysis. Besides, it will see the mechanical properties of the materials or specimen change or not change significantly under this microscope after been done in previous treatment. Thus, before it tests in SEM testing machine it must do a coating on the surface of fiber reinforce polymer figure 3.14 shown the fiber after doing coating. This is because, during the SEM test the fiber must be a conductive material. In addition, it also will give the result and the pattern of the PLF/ SH composite which is it will be the prove of the result during mechanical testing. Finally, the data of mechanical testing of materials can be collected and analyse the different between the different ratio of the PLF/ SH on the alkaline treatment and it will see the mixed of composite which is the bonding of both materials.

CHAPTER 4

RESULT AND DISCUSSION

In this chapter, shows all the experimental data and result for all mechanical testing that have done. All four mechanical testing that have been done are Tensile Test by referring (ASTM D 3039), Flexural Test (ASTM D 790-03), Scanning Electron Microscope (SEM), Hardness Test (ASTMD 2240 D).

4.1 Tensile Test

Instron 8872 machine was used to complete this tensile test. The Standard test method for tensile Properties of Polymer Matrix of Composite Materials (ASTM D 3039/ D 3039 M-0) has been related. The test was run, and the crosshead speed rate of the tensile test was 2.2mm/ min constant speed. In this study all the result of the samples was taken, and the data were collected. Below shows data of tensile test and two graph Tensile Stress σ (MPa) against Modulus Young (E) and Tensile Strain ε (mm/ mm) against Modulus young (E).

4.1.1 Result

Table 4.1 show data of the Tensile Stress σ (MPa), Modulus Young (E) and Tensile Strain ϵ (mm/ mm) for the composition 60%PLF and 40%SH with different time of concentration of alkaline treatment on PLF 2 hours until 16 hours. According to the table 4.1 from the result, the lowest tensile stress is 0.3322 MPa at 2 hours period time of PLF treatment. While, the highest value of tensile stress is 3.4740 MPa at 12 hours of PLF treatment.

Table 4.1: Data of Tensile Test

PLF Treatment (hours)	Tensile Stress σ (MPa)	Modulus young (E) (GPa)	Tensile Strain ϵ (mm/ mm)
2	0.3322	0.128	0.0026
4	0.3809	0.254	0.0015
6	0.7725	0.368	0.0021
8	1.3286	0.578	0.0023
10	1.1793	0.421	0.0028
12	3.474	2.316	0.0015
14	2.317	0.78	0.0035
16	2.7981	1.749	0.0016

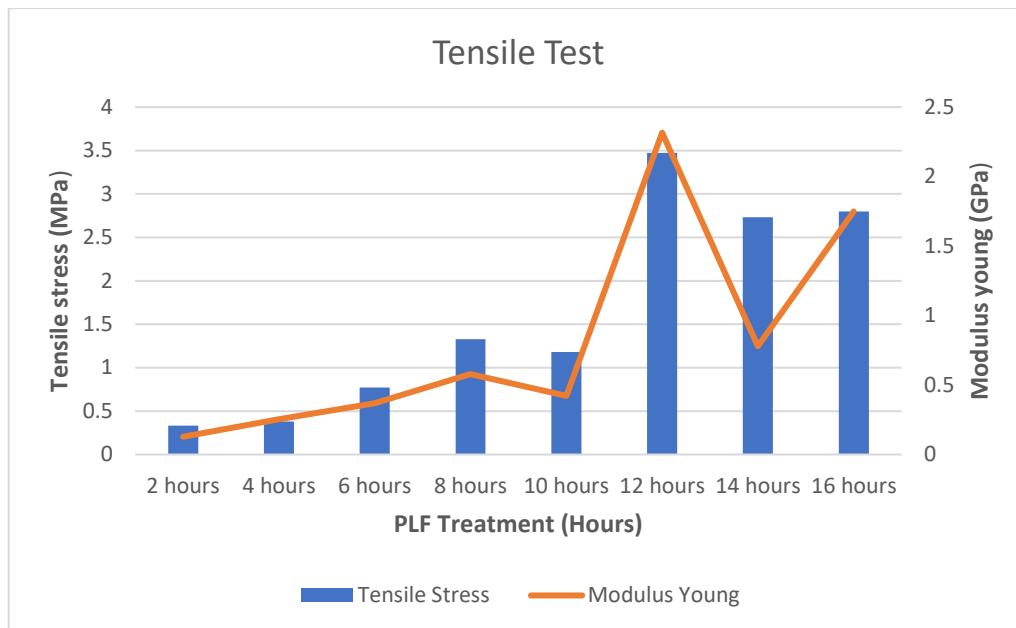


Figure 4.1: Graph of Tensile Stress (MPa) vs Modulus Young (GPa) on different concentration of PLF treatment (hours)

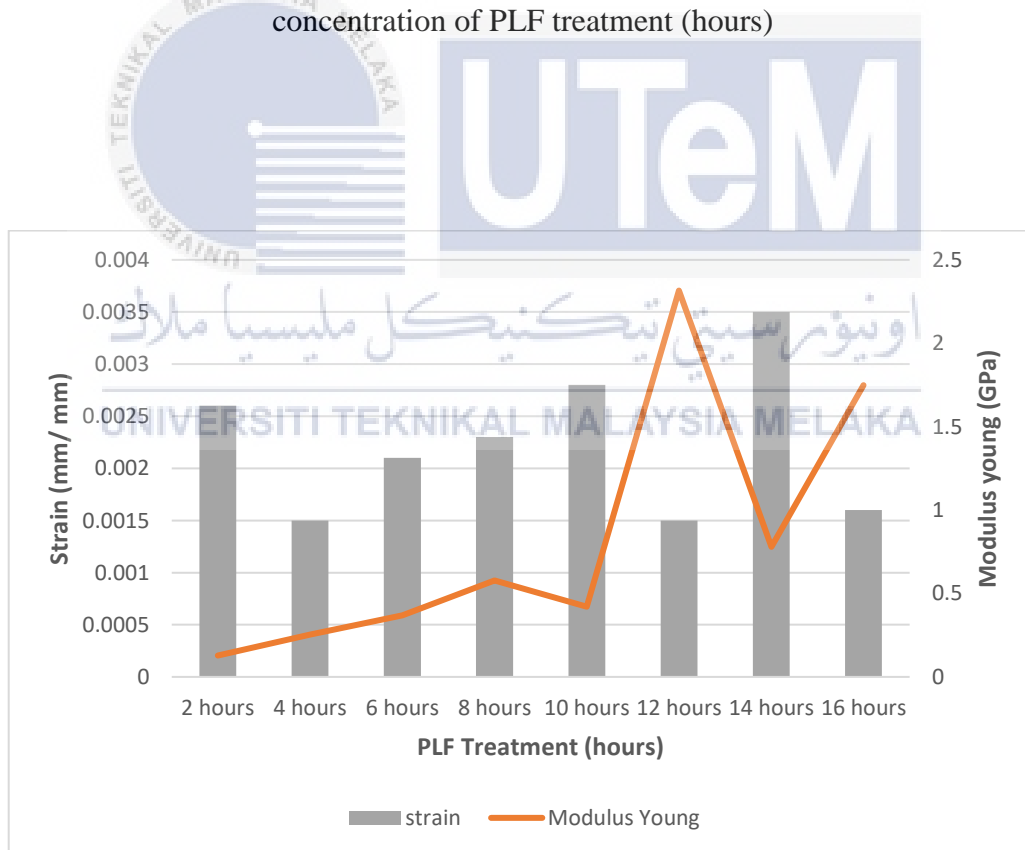


Figure 4.2: Graph of Tensile Strain (mm/mm) vs Modulus Young (GPa) on different concentration of PLF treatment (hours)

Figure 4.1 and figure 4.2 shows two graph of Tensile Stress (MPa) against Modulus Young (GPa) and Tensile Strain (mm/ mm) versus Modulus Young (GPa). The result of data shown in the graph, based on the graph obtained it shows that increasing the treatment on the PLF will give the result obviously increase the graph pattern give more strength of PLF.

4.2 Flexural Test

The flexural testing is to be measure the comportment of the sample that has been subjected to a simple support beam loading. By referring standard (ASTM D 790-03) testing machine was used to perform the flexural test. The crosshead speed rate is different with tensile test which is (2 mm/min) was constant and 3-point flex will be used. The samples were used to be made up of the same composition of 60PLF/40SH with different concentration treatment of PLF (hours). Eight samples that being test in this flexural test. Figure 4.3 shows the sample of PLF after being tested and figure 4.4 shows the flexural testing machine Instron 5585.



Figure 4.3: PLF samples



Figure 4.4: Flexural testing machine

4.2.1 Result

The result come by from this test are the modulus elasticity, flexural stress and flexural strain. Eight samples that has been test for the composition of 60PLF/40SH. Table 4.2 shows the data of PLF treatment. According to the data from table 4.2 the highest value of the flexural stress id 4.294 (MPa) at 16 hours of PLF treatment. While, the lowest value of flexural stress is 2.445 (MPa) at 2 hours PLF treatment.

Table 4.2: Data of Flexural test

PLF treatment (hours)	Flexural stress σ (MPa)	Modulus of (E) elasticity (Gpa)	Flexural strain ϵ (mm/ min)
2	2.445	0.19	0.0129
4	2.662	0.29	0.0091
6	2.761	0.54	0.00511
8	3.372	0.3	0.0109
10	3.247	0.49	0.0069
12	3.657	1.26	0.0029
14	3.871	0.31	0.0125
16	4.294	0.49	0.0069

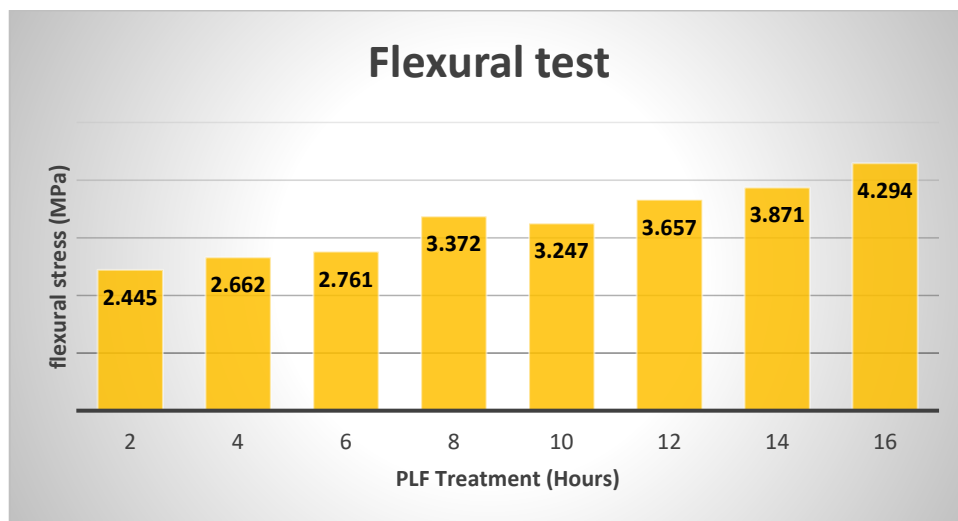


Figure 4.5: Graph of flexural stress (MPa) against PLF treatment (hours)

4.3 Hardness Test

In this section, the hardness test will be running for the eight tests of PLF samples which is all the samples have different period that treated with alkaline treatment. In this study, the 60PLF/40SH composition had been choose and to be investigate. Figure 4.5 and 4.6 shows The Shore hardness device is type D that be used to test the hardness of each samples. By referring standard of ASTM D2240.

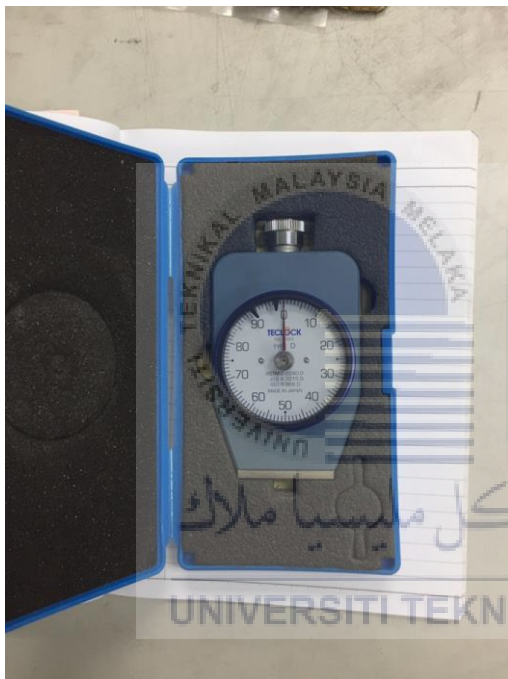


Figure 4.6: Shore hardness type D

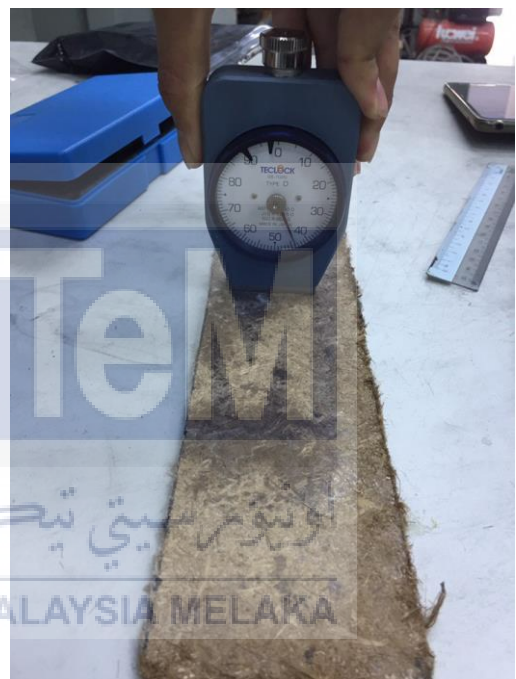


Figure 4.7: Hardness test at samples

4.3.1 Result

From the table 4.3 data of the hardness test on the eight test samples. Each sample has been done 5 test point to get the average values. Based on table 4.3, the hardness test result from eight different treatment (hours) being collected. It shows that the increment and decrement value of the hardness data. The increasing and decreasing value pattern of the data shown in graph (figure 4.7) below. The highest value of hardness test is 61.04 at 8 hours

of PLF treatment. While the lowest values of hardness test are 45.3 at 14 hours PLF treatment. Form this result it seen that the increasing values at shortest PLF treatment is much better that longer treatment. The estimation from this study the particulate size of SH will rise the hardness values of the samples of composite materials.

Table 4.3: Data of Hardness test

PLF Treatment (Hours)	Hardness (Shore -D)					
	Test 1	Test2	Test 3	Test 4	Test 5	Average
2	46	43	59	59.3	51	51.66
4	63	41	58	45	49	51.2
6	60	44	45	55	50	50.8
8	66.2	71	56	62	50	61.04
10	39.3	57.2	64	63	51.2	54.9
12	55	52.5	48.5	42	47	49
14	42	40	40.5	41	63	45.3
16	52.3	59	66	67	58	60.5

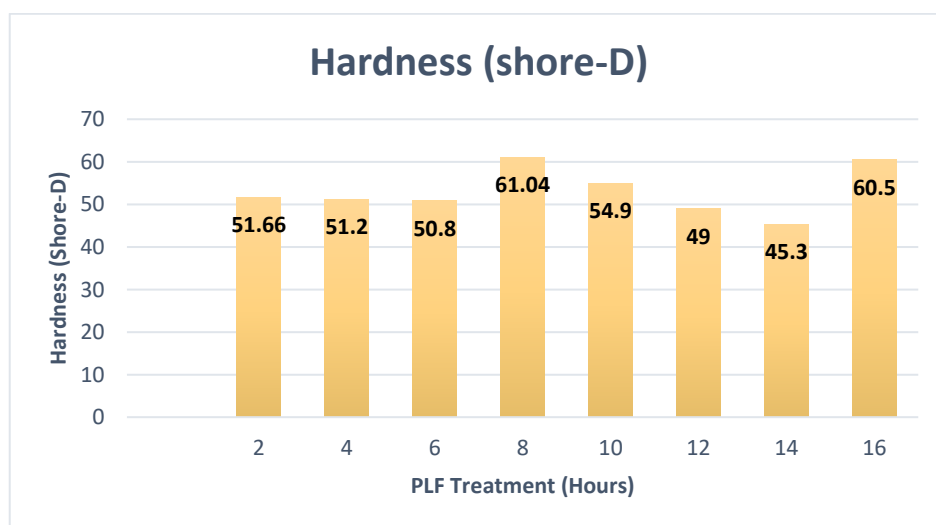
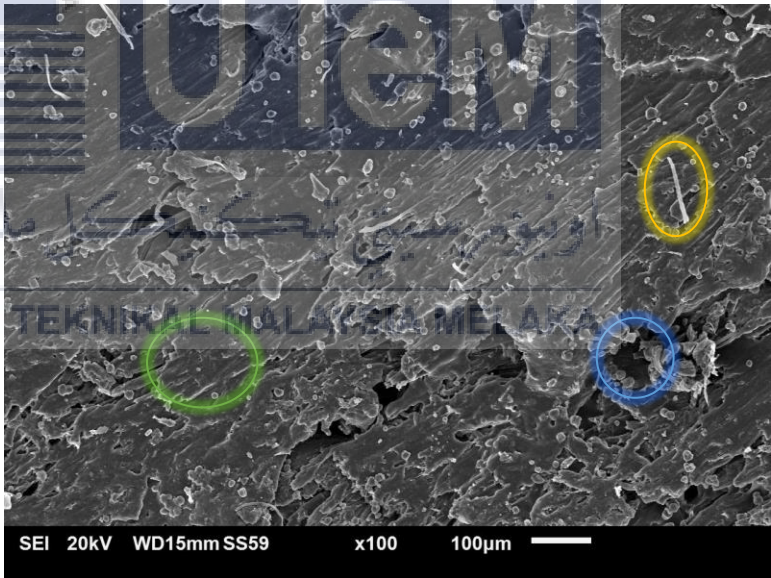


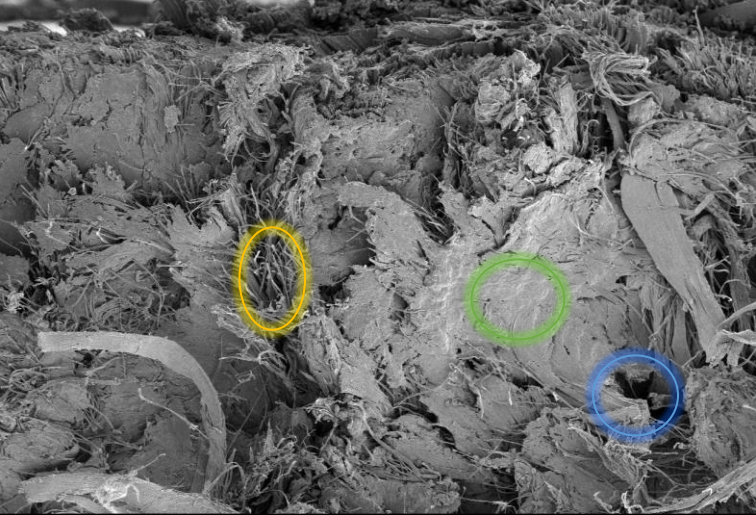


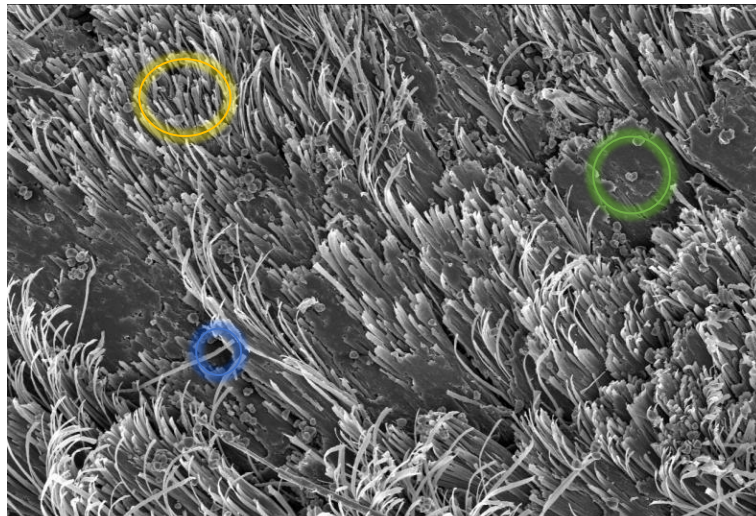
Figure 4.8: Graph of Hardness against PLF treatment (hours)

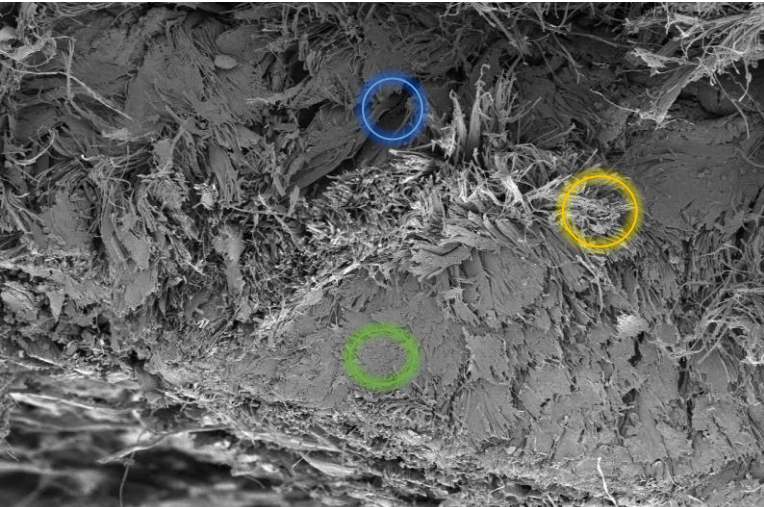

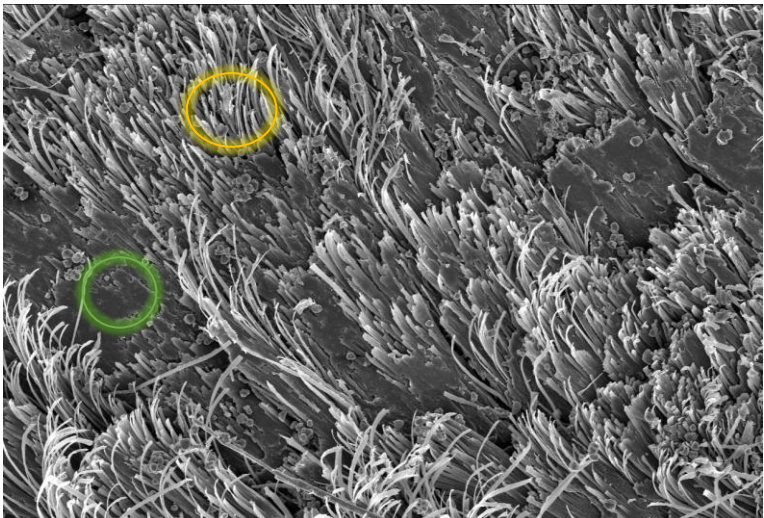
4.4 Scanning Electron Microscopy (Sem) Analysis.

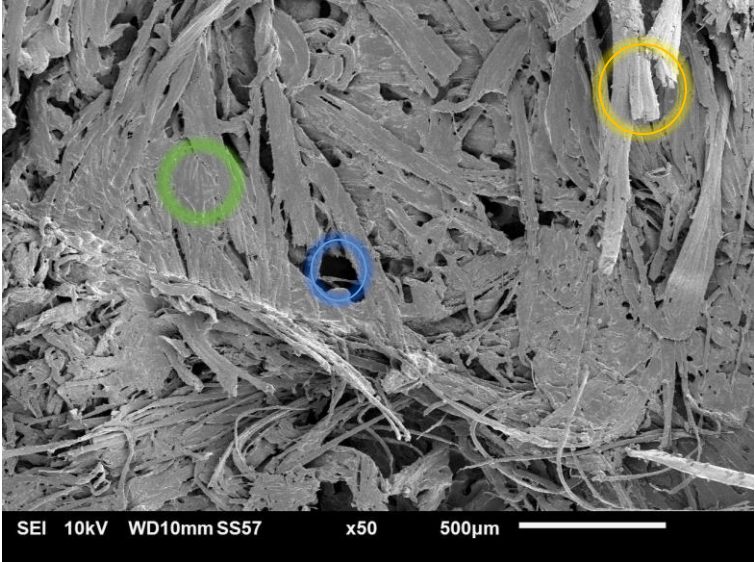
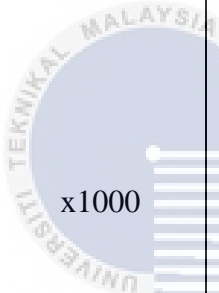
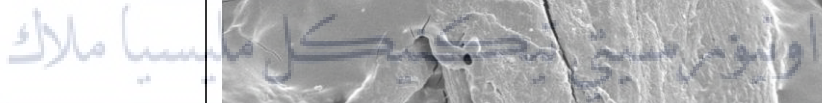

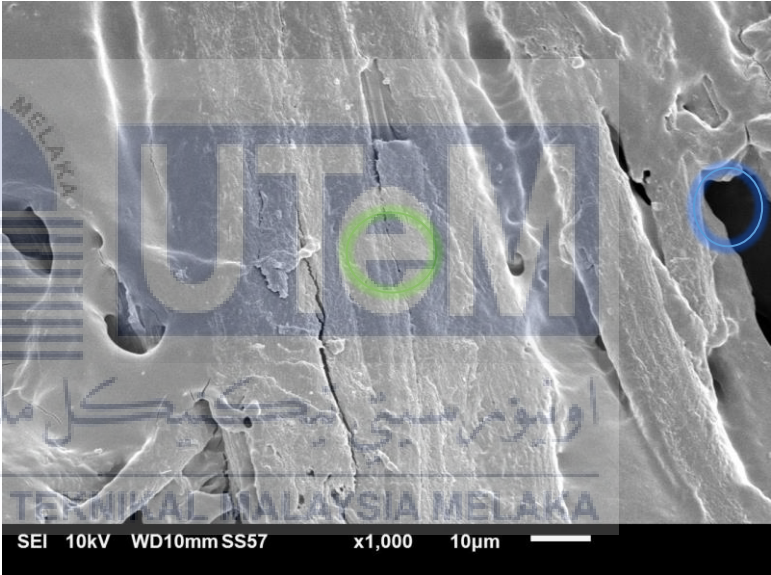
In this part, Scanning Electron Microscopy (SEM) analysis will be carry out in order to view the microstructure of the sample. Therefore, it can achieve the resolution better that 1 nanometer. The result shown are the arrangement and relations between the parts or element of PLF and SH. Besides, it will know does it has porosity, crack or void on the samples based on the composition and fiber treatments. Table 4.4 shows the SEM analysis.

Table 4.4: Result view under Scanning Electron Microscopy (SEM)

PLF Treatment (Hours)	Magnification (X)	SEM view
2	x100	

4	x50	 <p>SEI 10kV WD10mm SS57 x50 500µm</p>
	 <p>x1000</p>	 <p>SEI 10kV WD10mm SS57 x1,000 10µm</p>
6	x100	 <p>SEI 20kV WD15mm SS59 x100 100µm</p>

10`	x50	 <p>SEI 10kV WD10mm SS57 x50 500µm</p>
10	x1000	 <p>SEI 10kV WD10mm SS57 x1,000 10µm</p>
12	x100	 <p>SEI 20kV WD15mm SS59 x100 100µm</p>

14	50	
14	  	



= PLF



= PLF/SH mixed well



= Void

Based on the table 4.4 the behaviour of PLF and SH can be seen clearly as the SH seem to be stockpile among the PLF with the composition of SH. Next, The SH spotted to be well mixed with the PLF until it homogenous. This will make it easier to melt properly during heat treatments. Furthermore, at the 2 hours treatment seem the void on the sample which is this result can estimate that the sample do not homogenous during mixture. This result will prove that lowest value on the mechanical testing is 2 hours PLF treatment.

4.5 DISCUSSION AND ANALYSIS

From all the four mechanical testing of the samples which has been perform, the result will discuss in this section.

4.5.1 Effect of short pineapple leaf fiber treatment on the properties of pineapple leaf fiber- starch composites on Tensile test.

Figure 4.8 and 4.9 shows the result of tensile stress σ (MPa), modulus young E (GPa) and tensile strain ϵ (mm/mm). From the two of tensile stress (σ) against modulus young (E) and tensile strain (ϵ) against modulus young (E). The composition is 60PLF/40SH with different time of treatment on the PLF. The highest value of tensile stress is 3.474 (MPa) also the highest values of modulus young 2.316 (GPa) at 12 hours concentration of PLF treatment. While the lowest value of tensile stress is 0.3322 (MPa) and the lowest values of modulus young is 0.128 (GPa) at 2 hours concentration of PLF treatment. Next, the tensile strain (mm/mm) over modulus young (GPa) the value is increase significantly form 4 hours to 10 hours of PLF treatment. However, 12 hours and 16 hours are decrease but the modulus young increase. From this result the reason for this distinct value because the different period of PLF treatment that conduct will give different strength on the PLF.

Moreover, 12 hours PLF concentration due to alkaline treatment is longer than 2 hours. Therefore, the PLF totally or longer thoroughly seep into the fiber. Besides, the strength of the composites will be enhancing when the fiber treatment is comprehensive due to longer period of time [1]. Furthermore, according to the graph PLF treatment at 14 hours and 16 hours period of time, the decreasing value of tensile stress 2.7317 (MPa) and 2.7981 (MPa) because of the matrix. This result is quite stable value since the composition between PLF and SH are balance. Hence, SH cannot hold the PLF firmly. In addition, the fiber length has big significant on the properties of the composites. The longer the PLF, the higher tensile stress (σ) [31]. The longer the fiber length, the less ends. Similarly, it means, the fiber performance as stress concentration points which cause failure consistently to occur. This possibly cause the devaluation of tensile strength [32]. The high fiber to reciprocal action of fiber, which is SH was not totally secured with PLF [1].

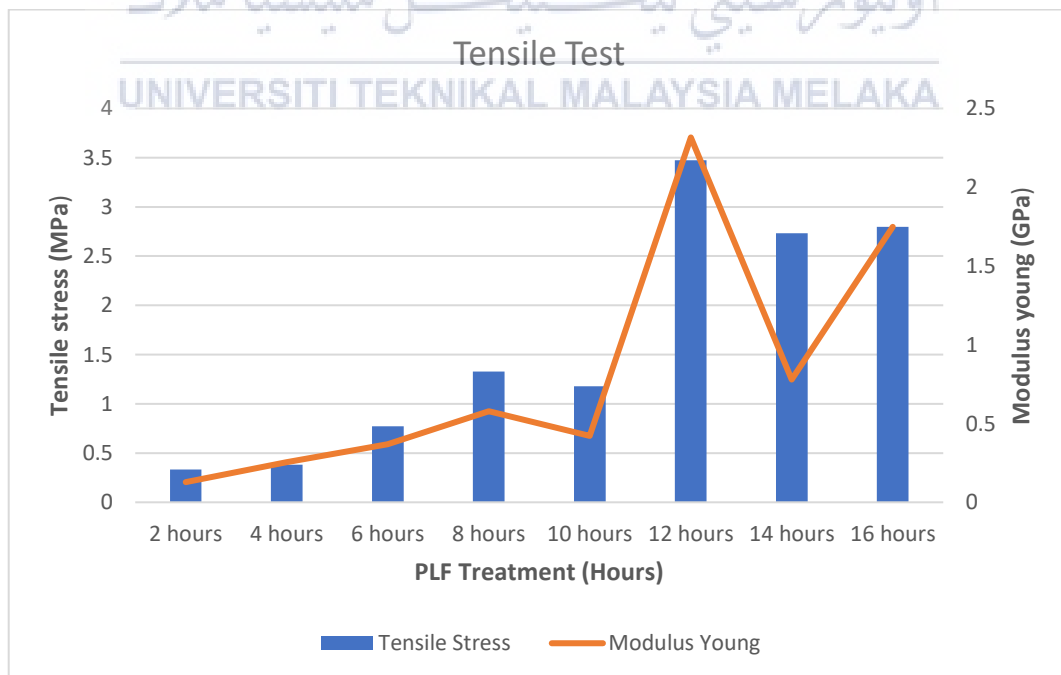


Figure 4.9: Graph of Tensile Stress (MPa) vs Modulus Young (GPa) on different concentration of PLF treatment (hours)

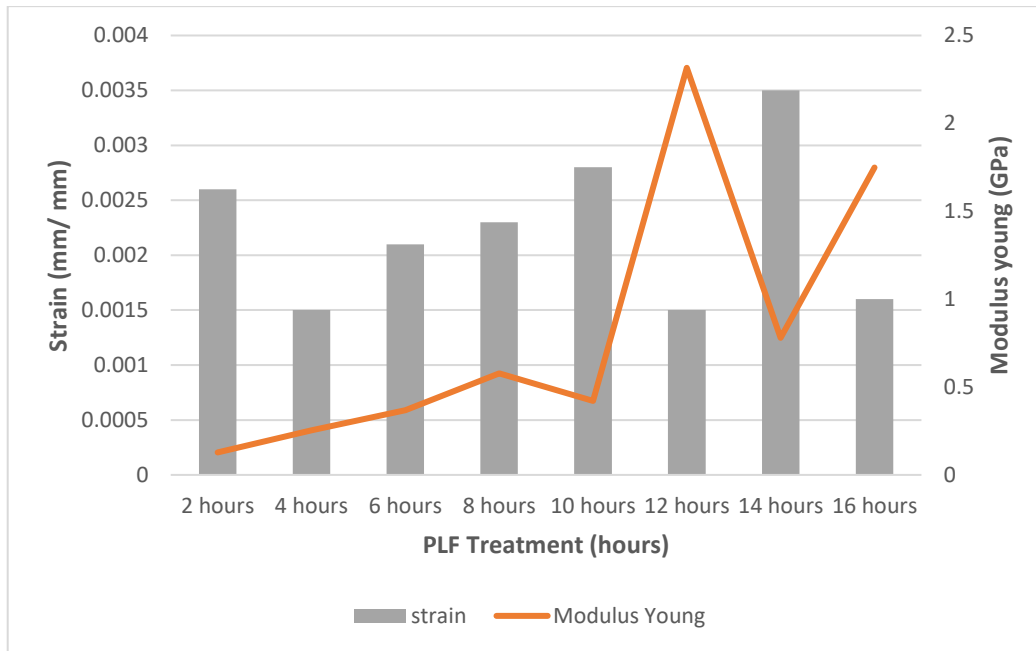


Figure 4.10: Graph of Tensile Strain (mm/mm) vs Modulus Young (GPa) on different concentration of PLF treatment (hours)

4.5.2 Effect of short pineapple leaf fiber treatment on the properties of pineapple leaf fiber- starch composites on Flexural test.

The result flexural test of PLF treatment of the composite materials are shown in figure 4.10. The result shows based on the same composition from 60PLF/40SH with different period concentration time of PLF treatment. Besides, the outcome discovery finds that the different time of PLF treatment will produce a different significant result. From the previous study, they find that the different size of particulate matrix will show the higher the fiber content in composite materials, the toughest fiber in mechanical properties [2].

Figure 4.10 shows the result testing for eight samples of PLF composite materials. The orientation from this test was found that the flexural stresses are constantly increase as the PLF treatment hours also increased. The composition PLF/ SH of 60PLF/40SH with 2 hours PLF treatment, the value is 2.445 (MPa). Thus, the result increase from 2.445 (MPa)

at 4 hours PLF treatment. With the result that, the flexural stress also increases every 2 hours. However, in this study it limits to 16 hours of PLF treatment. The highest result of flexural stress is 4.294 (MPa). In fact, the result on that data shows the longer the PLF treated will constantly increase the value of mechanical properties.

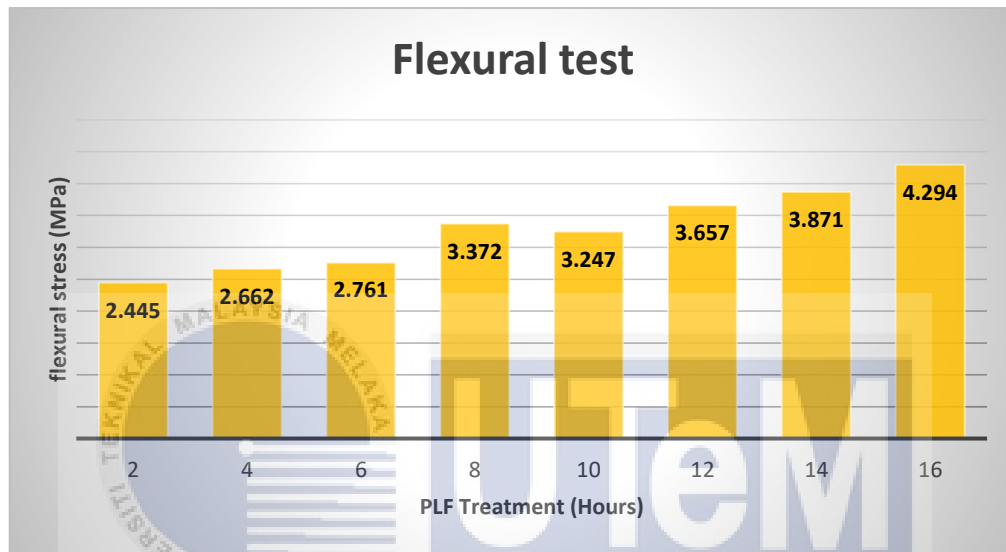


Figure 4.11: Graph of flexural stress (MPa) against PLF treatment (hours)

Furthermore, the study respecting of the composition of the fiber and matrix. In this analysis also focus point about the different size of the matrix in composite materials even if it will give a remarkable result on the mechanical properties or not. Figure 4.11 graph of flexural stress (MPa) against the PLF treatment (hours), surely, it shows that the longer hours penetration of PLF on alkaline treatment will give result the higher value in or strength in mechanical properties of composite materials. Besides, this due to the ability of the fiber react with alkaline and absorbability very excellence until it sips to fiber completely. For instance, when it reacts and melted with heat transfer as it being pressed make it grip and attachment to the fiber securely and strong. This can be seen on the Scanning Electron

Microscope (SEM) of the sample PLF/ SH as shown in figure 4.12 below. Thus, it makes the mechanical properties went stronger, but it might have some discontinuities during process. It shows standard error bar in the graph.

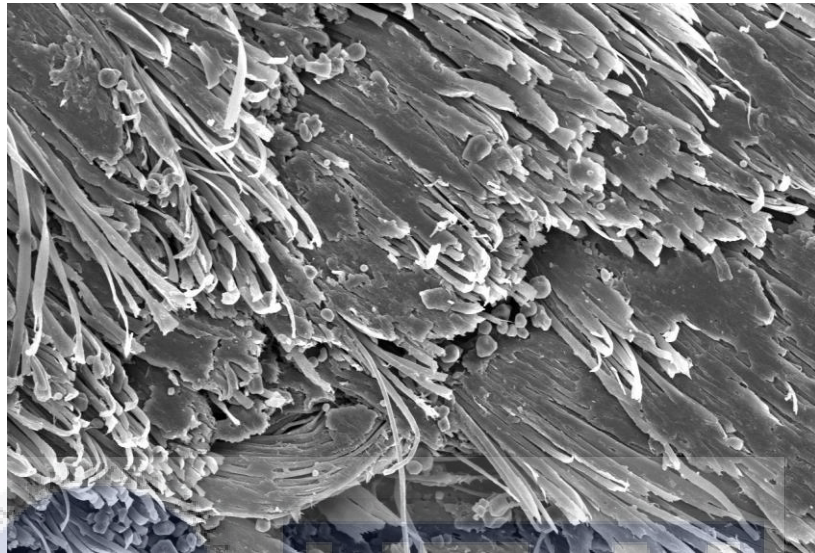


Figure 4.12: The SEM result on PLF/SH composite.

Another than that, based on the previous research, finding resources from Siregar (n.d) stated that the longer size of the fiber penetrated with alkaline and the longer period of time it will increase the tensile properties. This due to larger fiber size that dominance to more even diffusion in matrix. Thus, some samples are not tally with the result which is higher the treatment period of time cause the PLF more comprehensive through the fiber. The result must give the high flexural stress. However, it might not show it. This is because during fabrication process they maybe not perfectly bounded well together [1]. So that, the result of flexural stress value decrease in 10 hours PLF treatment compare to 8 hours concentration on alkaline treatment. Hence, the failure to get on the mark parameter to make perfect samples. For this reason, the accurate parameter cannot be determining in a short time and more study on the PLF treatment.

4.5.3 Effect of short pineapple leaf fiber treatment on the properties of pineapple leaf fiber- starch composites on Hardness test.

Based on figure 4.12, the graph shows the result of hardness (Shore-D) the result of with the various of concentration of PLF treatment respectively. With the result that, from the graph shows that the hardness of the samples increases with enlargement of the concentration time of fiber due to an alkaline treatment for all samples. Five point that had been repeated and the average had been selected to get more accurate result.

As a result, figure 4.12 shows graph hardness against PLF treatment (hours). By referring to graph, the highest result is 61.04 at 8 hours concentration time of alkaline treatment on PLF. While, the lowest hardness reading which is at concentration time treatment on PLF at 14 hours is 45.3. The result shows the unexpected trend which have increment and decrement at graph result. Thus, for the sample at fiber treatment 12 hours shown at graph is lower than 10 hours treatment. On this situation shows that on the 10 hours reading of the graph is more hardness compare to 12 hours treatment. While it may be true, the fiber and starch at 10 hours treatment is perfectly wrapped and cause the samples more homogeneous and perfectly bonded together during heat treatment (hot press).

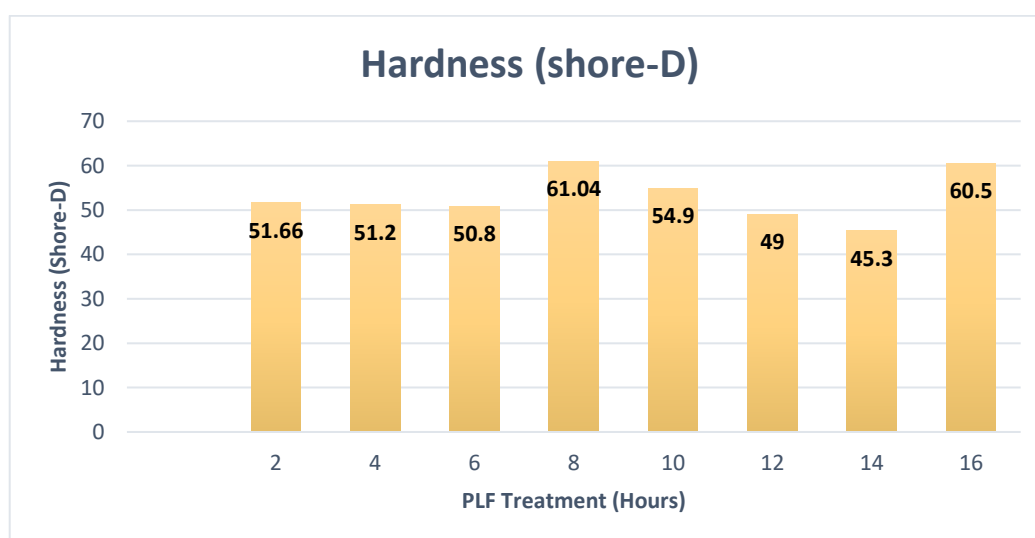


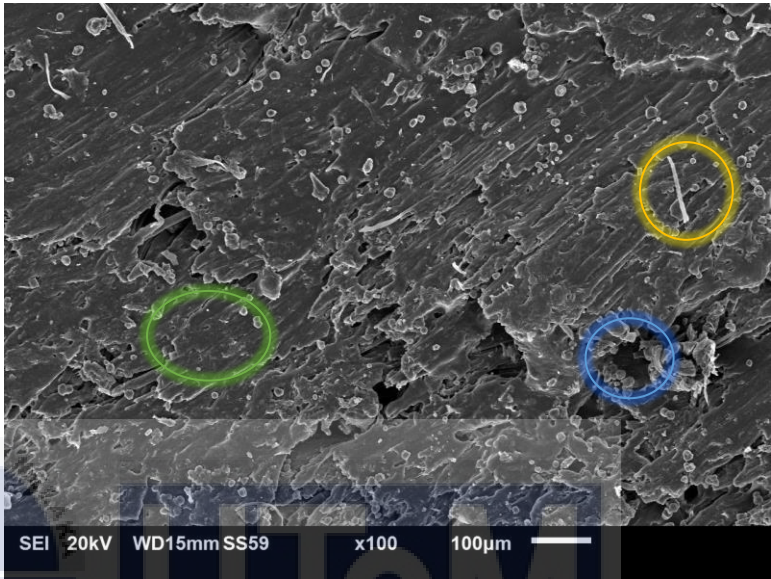
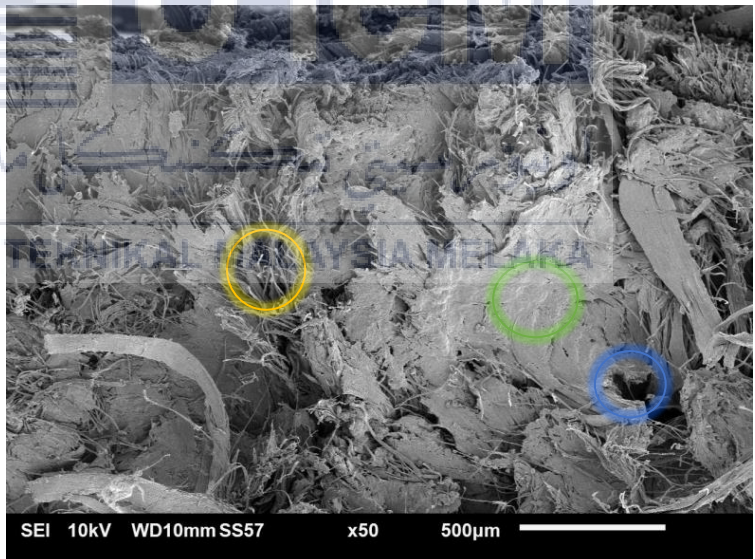
Figure 4.13: Graph of Hardness against PLF treatment (hours)

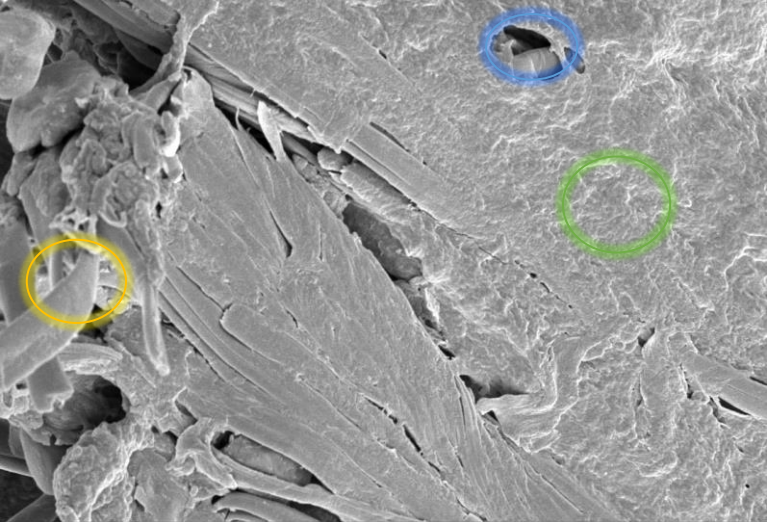
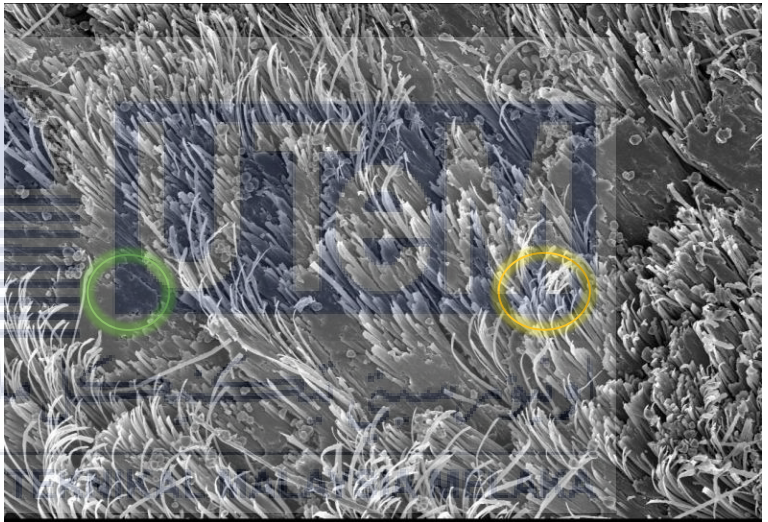
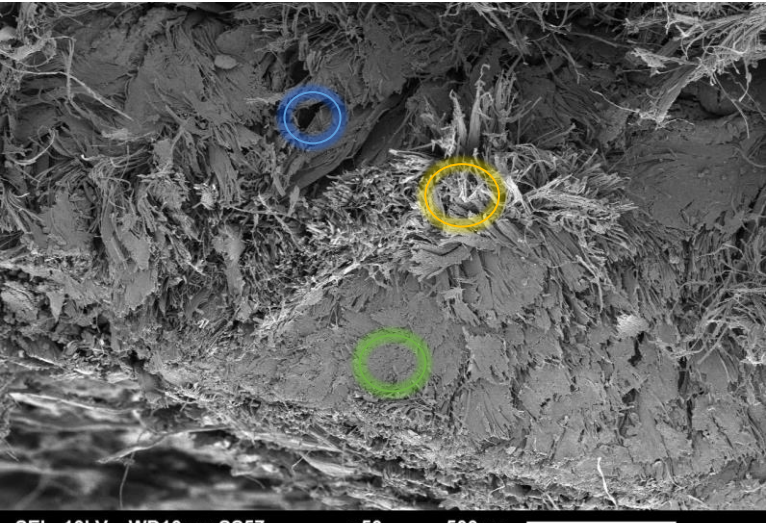
Form the previous finding, hardness of the composites is affected the fiber content which is PLF treatment is increasing corresponding to the hardness. Moreover, the error bars showed on the graph which is when fiber is more concentration with an alkaline for more longer time it will more hardness samples can be produce. Conversely, the decrement data of the result may cause due to unsmooth surface on the matrix. That why, repeated test for a several times is needed to get a good result. In research by Salamat and Mashiach (2015) PLF and SH are used for the composite. The result shows, the highest of PLF content will certainly extent increase the result of hardness test.

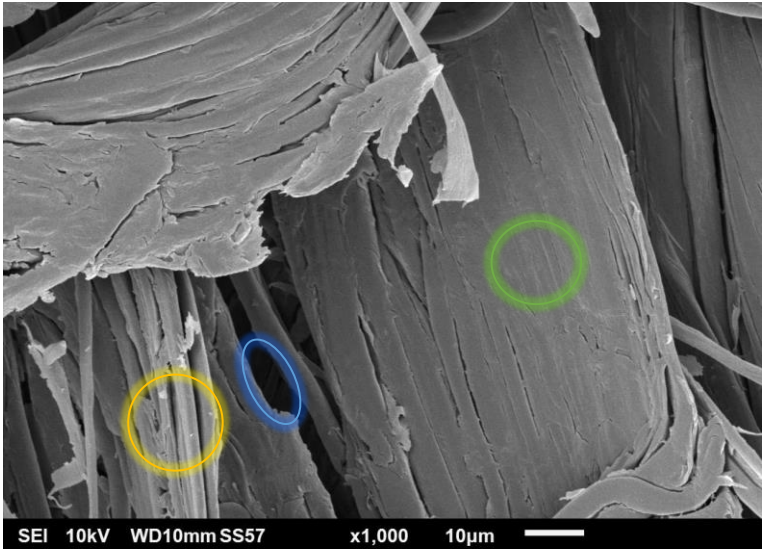

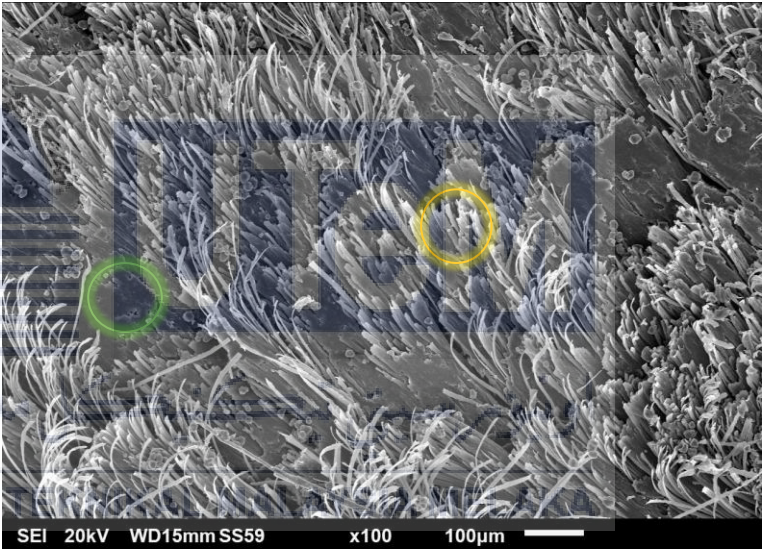
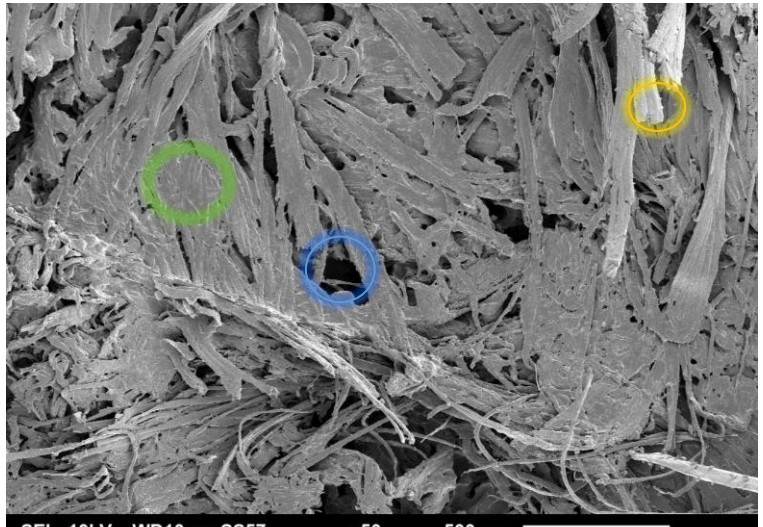
4.5.4 Effect of short pineapple leaf fiber treatment on the properties of pineapple leaf fiber- starch composites on SEM analysis.

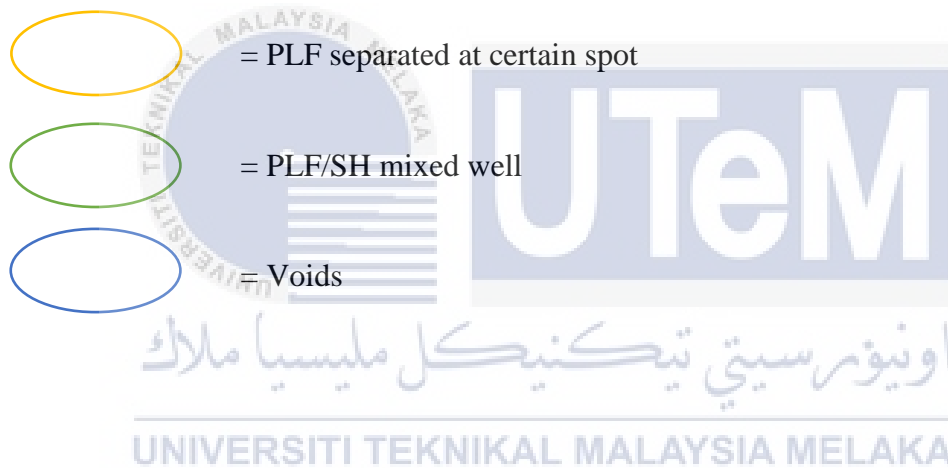
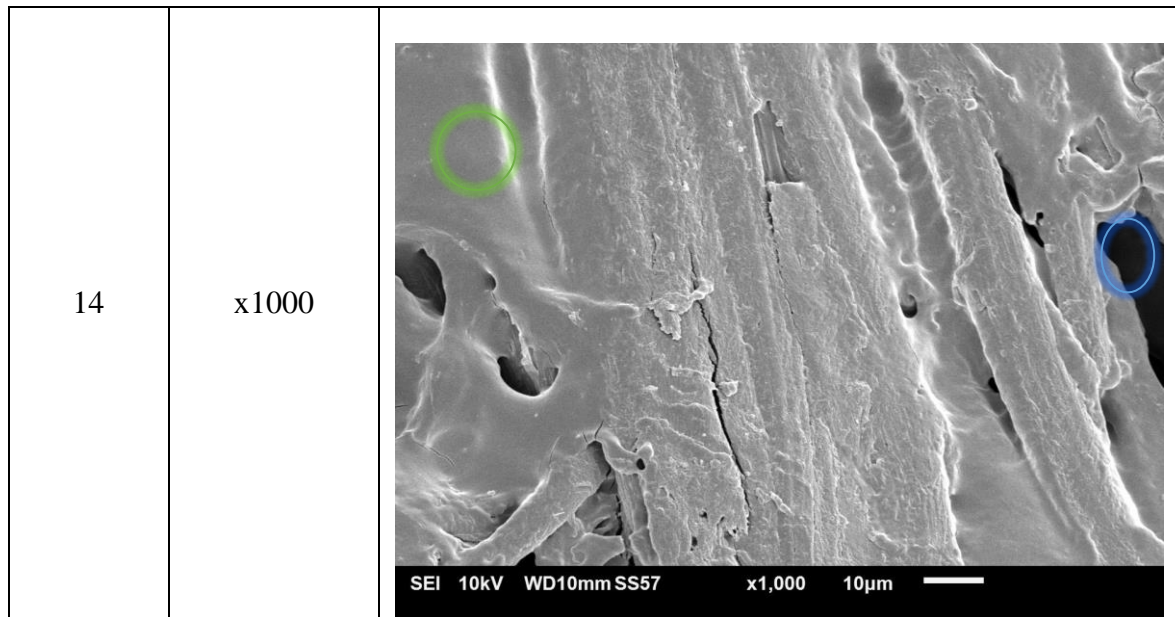
The result shown at table 4 .5 that the shortest PLF treatment which is 2 hours will have more void compare to longer PLF treatment. Next, from this result it can seem the great adhesion joint between fiber and matrix is at 12 hours of PLF treatment. This result of SEM, it can conclude that the strong the bonding to each other and homogeneous texture will be affecting the mechanical properties. Therefore, the longer the treatment will give more longer NaOH solution and alkaline can penetrated at the fiber. The fiber can easily to merge together with SH. In short, the void in samples it can cause the incomplete wettability in dispersion through the starch and PLF matrix [33].

Table 4.5: Result view under Scanning Electron Microscopy (SEM)

PLF Treatment (Hours)	Magnification (X)	SEM view
2	x100	
4	x50	

	x1000	 <p>SEI 10kV WD10mm SS57 x1,000 10µm</p>
6	x100	 <p>SEI 20kV WD15mm SS59 x100 100µm</p>
10`	x50	 <p>SEI 10kV WD10mm SS57 x50 500µm</p>

10	x1000	
12	 x100	
14	50	



With the result that on SEM it seems that the homogeneous texture of combination PLF/SH provided much improved surface compare to the non-smooth texture of PLF/SH. The samples that produced good combination is at 14 hours treatment shows good homogenous bonding between PLF/SH. Table 4.5 above shown 14 hours treatment at x 50 x1000 magnification result. This is because no voids founded on that sample. Therefore, for this reason it shows that the finest both of PLF/SH the easier to be mix and when it processes in hot press it will be melt perfectly. Hence, making an excellent bonding between fiber and matrix materials.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This project is essentially to study and focus on how producing a Bio-degradable of composite materials which is from combination of pineapple leaf fiber and natural polymer (starch). This innovation and idea from green composite materials is very suitable to be used in plastic industries. In this study pineapple leaf fiber is used as the reinforce materials while starch is used as a matrix. The key objective of this research is to determine the effect pineapple leaf fiber (PLF) treatment on the properties of starch (SH) composite and to study the effect of PLF loading on the properties of PLF/SH composites.

The expected result from this finding is the longer PLF treated in alkaline treatment and NaOH solution may have a greater value in mechanical properties. For this reason, the PLF after treated may find that it become more finest and homogeneous compare PLF not being treated. So that, from this result that achieve from this study is the finest of composition PLF/SH may have a greater stronger bonding to each other. From this, it can come out with a greater strength in mechanical properties.

Lastly, for the conclusion that can make the longer PLF treated and the finest the particulate matrix will give a superior ability of the fiber. Besides, during fabrication process the finest and homogeneous mixture will give a perfect bonding. Thus, this finding will give an information and good potential producing high performance of composite materials.

5.2 Recommendation

For further research in this study, there are a few recommendations important that should be highlighted to be improve that result of mechanical properties of PLF/SH.

Recommendation need to consider in this study:

I. Sample Preparation

Pineapple leaf fiber must be a very fine texture to mix well with matrix such as SH. The preparation of PLF from it fiber process until it become powder form. Therefore, it can mixture very well with SH. Hence during the fabrication process the sample that produce is melt together. Moreover, to obtain the PLF fiber is very dry before it processes become powder. Further study tries to overcome with vacuum over to make sure the PLF is really dry before cutting process [31]. For this purpose, it will make bonding with this two material become stronger.

II. Fiber Size

Achieving a good fiber orientation to determine fiber size such as particulate form of fiber will affect the mechanical properties. The distribution of particles in the composite matrix is random. Therefore, strength and other properties of the composite material are usually isotropic. If this can be achieved, the strengthening mechanism depends on particle size.

III. Fiber Length

For further study, it is recommended to increase the strength of composite materials. Besides, effect of the strength of composite materials. Besides, effect of length of fiber reinforced is important. So, as fibers get longer and thinner, the overall properties of the composite are improved. Hence, the stress along a fiber will see when the maximum fiber load is achieved at the centre of fiber length. Another than that, the maximum fiber load is carried by most of the fiber. These are considered to be continuous fibers.

IV. Composition of The Composite Materials

Pineapple leaf fiber treatment on properties of SH composition 60PLF/40SH was choose on this research. For the further study, try to change the composition of PLF conversely. This can be giving a different result that can be investigate. Besides, try to change the percentage of PLF and SH. For example, 60SH/ 40PLF, 70SH/ 30 PLF. Which is SH being the more percentage that PLF. By referring this composition, it might give good composite materials can be produced.

V. Testing Method

Mechanical testing had been done to determine the effect of PLF result for this study. In addition, to improve this research, result adding more testing such as water absorption test, impact tested and degradation analysis in future study. For this purpose, other effect mechanical properties testing can be study to improve the research of PLF/ SH composite materials. The investigation effect of PLF with this test method will know the behaviour strength of this PLF/ SH composites.



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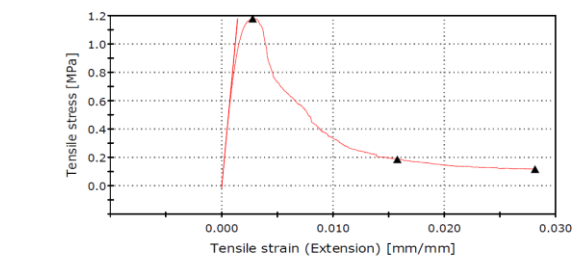
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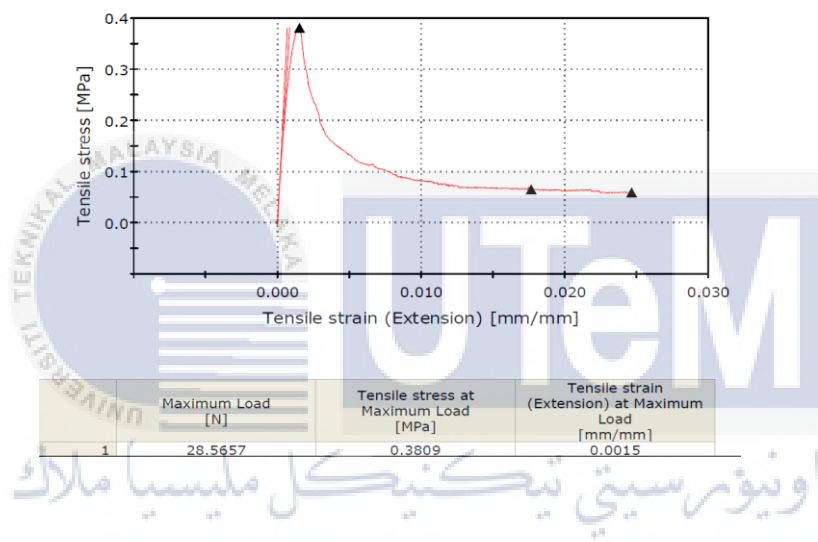
Tensile Test for PLF

2 Hour PLF Treatment



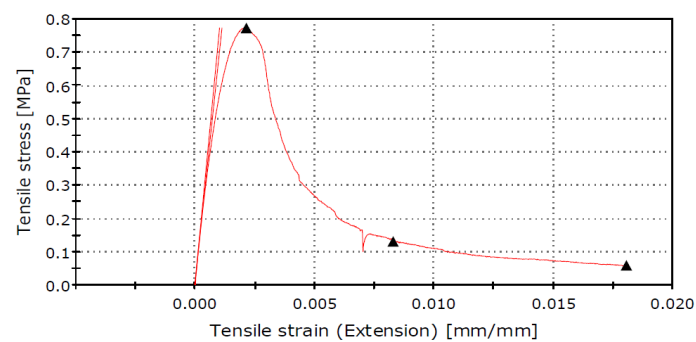
	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	88.4457	1.1793	0.0028

4 Hour PLF Treatment



	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	28.5657	0.3809	0.0015

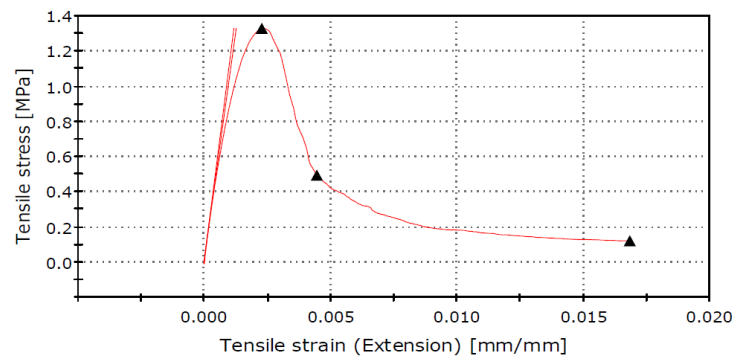
6 Hour PLF Treatment



	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	57.9375	0.7725	0.0021

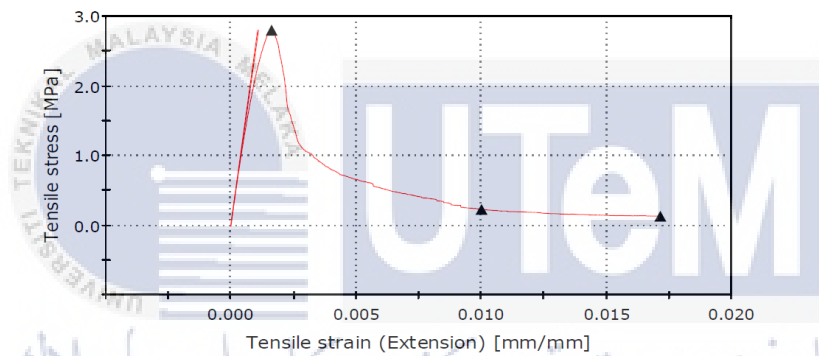
Tensile Test for 2, 4, 6 hours PLF treatment

8 Hour PLF Treatment



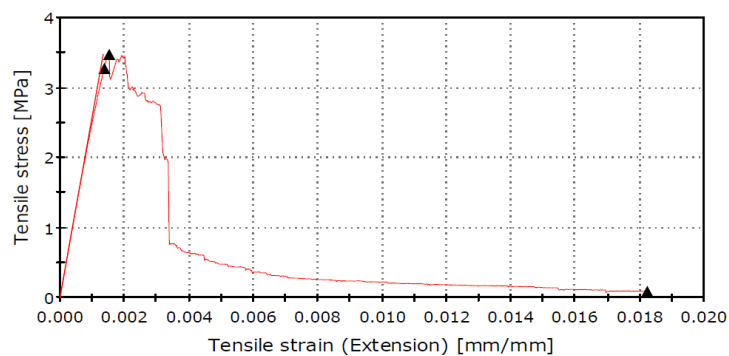
	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	99.6454	1.3286	0.0023

10 Hour PLF Treatment



	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	209.8558	2.7981	0.0016

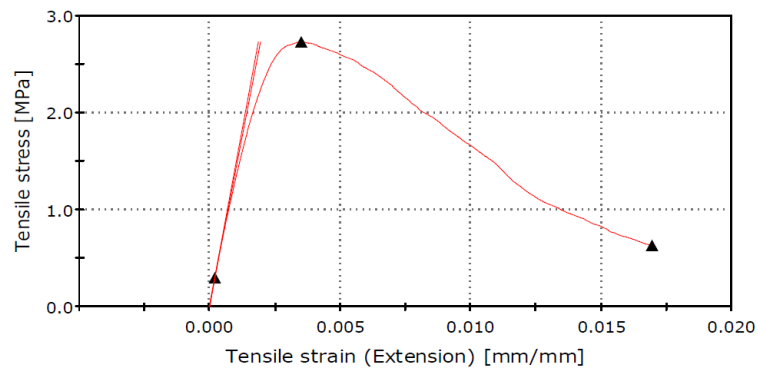
12 Hour PLF Treatment



	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	260.5463	3.4740	0.0015

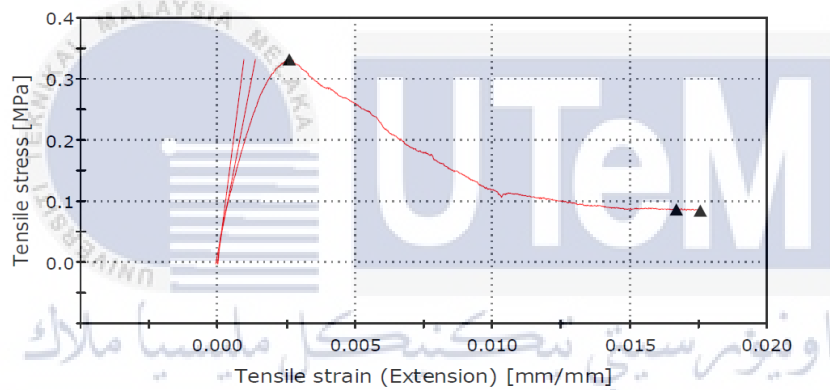
Tensile Test for 8, 10, hours 12 PLF treatment

14 Hour PLF Treatment



	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	204.8770	2.7317	0.0035

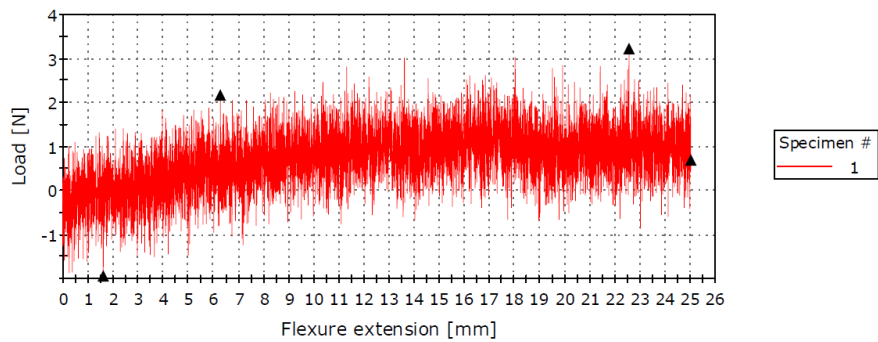
16 Hour PLF Treatment



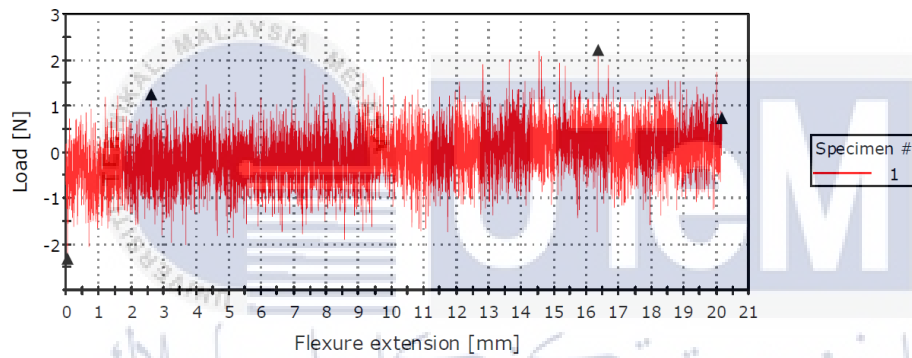
	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]
1	24.9119	0.3322	0.0026

Tensile test for 14, and 16 Hours PLF Treatment

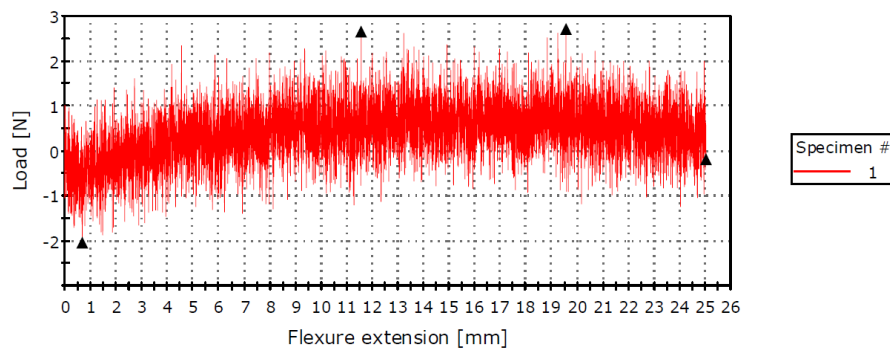
PLF Treatment 2 hours



PLF Treatment 4 hours

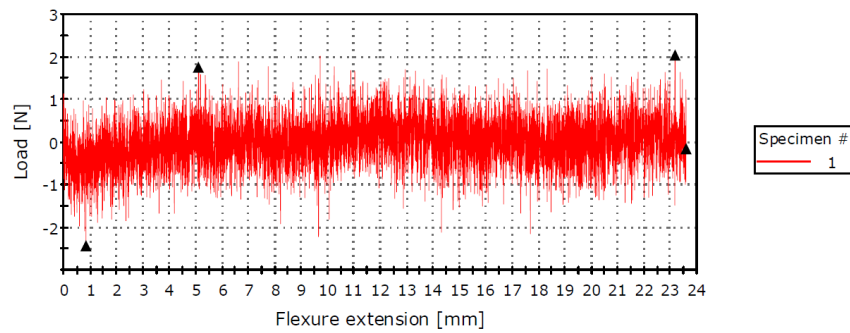


PLF Treatment 6 hours

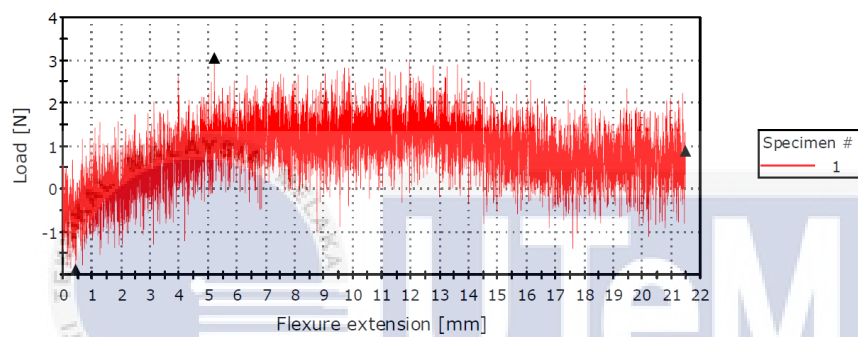


Flexural test for 2, 4, and 6 Hours PLF Treatment

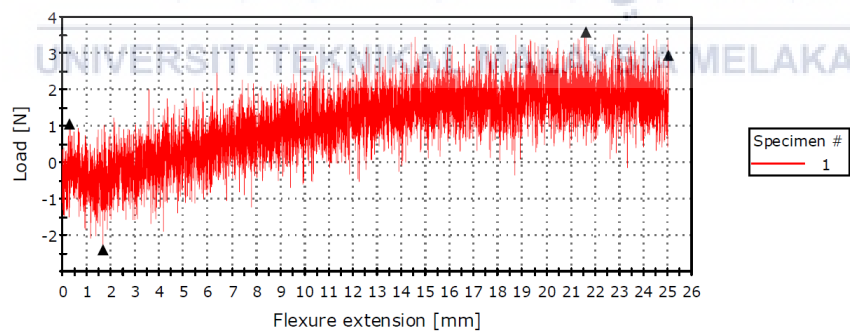
PLF Treatment 8 hours



PLF Treatment 10 hours

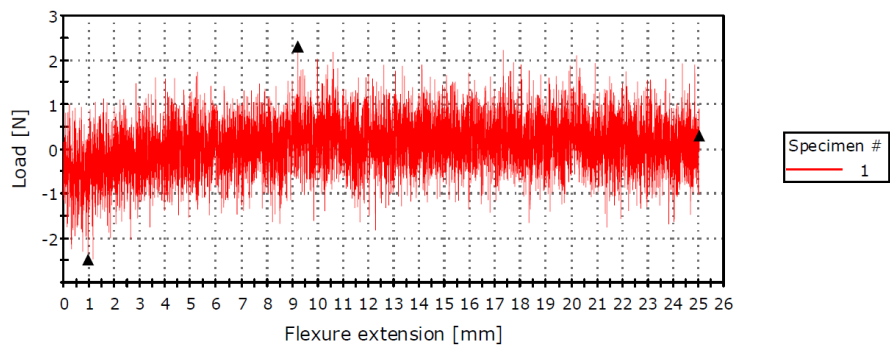


PLF Treatment 12 hours

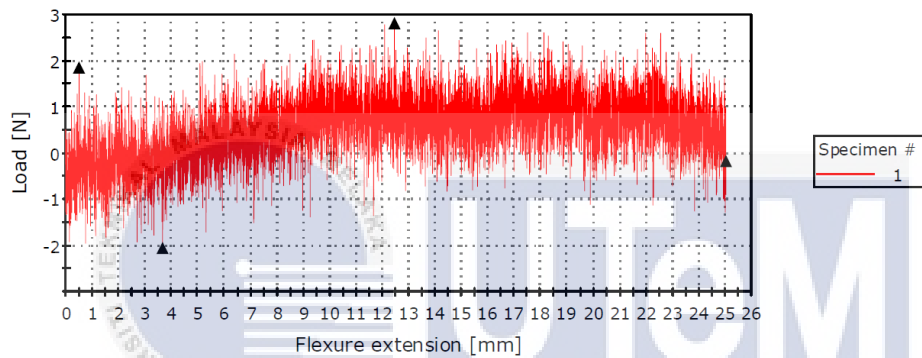


Flexural test for 8, 10, and 12 Hours PLF Treatment

PLF Treatment 14 hours



PLF Treatment 16 hours



اونیورسیتی تکنیکل مالایسیا ملاک
Flexural test for 14, and 16 Hours PLF Treatment

UNIVERSITI TEKNIKAL MALAYSIA MELAKA



Designation: D 3039/D 3039M – 00^{e1}

Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials¹

This standard is issued under the fixed designation D 3039/D 3039M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

^{e1} NOTE—Eq 5 was revised editorially in December 2002.

1. Scope

1.1 This test method determines the in-plane tensile properties of polymer matrix composite materials reinforced by high-modulus fibers. The composite material forms are limited to continuous fiber or discontinuous fiber-reinforced composites in which the laminate is balanced and symmetric with respect to the test direction.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement²
- D 883 Terminology Relating to Plastics²
- D 2584 Test Method for Ignition Loss of Cured Reinforced Resins³
- D 2734 Test Method for Void Content of Reinforced Plastics³
- D 3171 Test Methods for Constituent Content of Composites Materials⁴
- D 3878 Terminology for Composite Materials⁴
- D 5229/D 5229M Test Method for Moisture Absorption

Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials⁴

- E 4 Practices for Force Verification of Testing Machines⁵
- E 6 Terminology Relating to Methods of Mechanical Testing⁵
- E 83 Practice for Verification and Classification of Extensometers⁵
- E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus⁵
- E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process⁶
- E 132 Test Method for Poisson's Ratio at Room Temperature⁵
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁶
- E 251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages⁵
- E 456 Terminology Relating to Quality and Statistics⁶
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁶
- E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading⁵
- E 1237 Guide for Installing Bonded Resistance Strain Gages⁵

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. Terminology E 6 defines terms relating to mechanical testing. Terminology E 456 and Practice E 177 define terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other standards.

3.2 Definitions of Terms Specific to This Standard:

NOTE—If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental

¹ This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

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² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 15.03.

⁵ Annual Book of ASTM Standards, Vol 03.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

dimensions, shown within square brackets: $[M]$ for mass, $[L]$ for length, $[T]$ for time, $[\Theta]$ for thermodynamic temperature, and $[nd]$ for nondimensional quantities. Use of these symbols is restricted to analytical dimensions when used with square brackets, as the symbols may have other definitions when used without the brackets.

3.2.1 *nominal value*, n —a value, existing in name only, assigned to a measurable property for the purpose of convenient designation. Tolerances may be applied to a nominal value to define an acceptable range for the property.

3.2.2 *transition region*, n —a strain region of a stress-strain or strain-strain curve over which a significant change in the slope of the curve occurs within a small strain range.

3.2.3 *transition strain*, $\epsilon_{transition}^{transition} [nd]$, n —the strain value at the mid range of the transition region between the two essentially linear portions of a bilinear stress-strain or strain-strain curve.

3.2.3.1 *Discussion*—Many filamentary composite materials show essentially bilinear behavior during loading, such as seen in plots of either longitudinal stress versus longitudinal strain or transverse strain versus long longitudinal strain. There are varying physical reasons for the existence of a transition region. Common examples include: matrix cracking under tensile loading and ply delamination.

3.3 *Symbols*:

3.3.1 A —minimum cross-sectional area of a coupon.

3.3.2 B_y —percent bending for a uniaxial coupon of rectangular cross section about y axis of the specimen (about the narrow direction).

3.3.3 B_z —percent bending for a uniaxial coupon of rectangular cross section about z axis of the specimen (about the wide direction).

3.3.4 CV —coefficient of variation statistic of a sample population for a given property (in percent).

3.3.5 E —modulus of elasticity in the test direction.

3.3.6 F^u —ultimate tensile strength in the test direction.

3.3.7 F^u —ultimate shear strength in the test direction.

3.3.8 h —coupon thickness.

3.3.9 L_g —extensometer gage length.

3.3.10 L_{min} —minimum required bonded tab length.

3.3.11 n —number of coupons per sample population.

3.3.12 P —load carried by test coupon.

3.3.13 P' —load carried by test coupon at failure.

3.3.14 P^{max} —maximum load carried by test coupon before failure.

3.3.15 s_{n-1} —standard deviation statistic of a sample population for a given property.

3.3.16 w —coupon width.

3.3.17 x_i —test result for an individual coupon from the sample population for a given property.

3.3.18 \bar{x} —mean or average (estimate of mean) of a sample population for a given property.

3.3.19 δ —extensional displacement.

3.3.20 ϵ —general symbol for strain, whether normal strain or shear strain.

3.3.21 ϵ —indicated normal strain from strain transducer or extensometer.

3.3.22 σ —normal stress.

3.3.23 ν —Poisson's ratio.

4. Summary of Test Method

4.1 A thin flat strip of material having a constant rectangular cross section is mounted in the grips of a mechanical testing machine and monotonically loaded in tension while recording load. The ultimate strength of the material can be determined from the maximum load carried before failure. If the coupon strain is monitored with strain or displacement transducers then the stress-strain response of the material can be determined, from which the ultimate tensile strain, tensile modulus of elasticity, Poisson's ratio, and transition strain can be derived.

5. Significance and Use

5.1 This test method is designed to produce tensile property data for material specifications, research and development, quality assurance, and structural design and analysis. Factors that influence the tensile response and should therefore be reported include the following: material, methods of material preparation and lay-up, specimen stacking sequence, specimen preparation, specimen conditioning, environment of testing, specimen alignment and gripping, speed of testing, time at temperature, void content, and volume percent reinforcement. Properties, in the test direction, which may be obtained from this test method include the following:

5.1.1 Ultimate tensile strength,

5.1.2 Ultimate tensile strain,

5.1.3 Tensile chord modulus of elasticity,

5.1.4 Poisson's ratio, and

5.1.5 Transition strain.

6. Interferences

6.1 *Material and Specimen Preparation*—Poor material fabrication practices, lack of control of fiber alignment, and damage induced by improper coupon machining are known causes of high material data scatter in composites.

6.2 *Gripping*—A high percentage of grip-induced failures, especially when combined with high material data scatter, is an indicator of specimen gripping problems. Specimen gripping methods are discussed further in 7.2.4, 8.2, and 11.5.

6.3 *System Alignment*—Excessive bending will cause premature failure, as well as highly inaccurate modulus of elasticity determination. Every effort should be made to eliminate excess bending from the test system. Bending may occur as a result of misaligned grips or from specimens themselves if improperly installed in the grips or out-of-tolerance caused by poor specimen preparation. If there is any doubt as to the alignment inherent in a given test machine, then the alignment should be checked as discussed in 7.2.5.

6.4 *Edge Effects in Angle Ply Laminates*—Premature failure and lower stiffnesses are observed as a result of edge softening in laminates containing off-axis plies. Because of this, the strength and modulus for angle ply laminates can be drastically underestimated. For quasi-isotropic laminates containing significant 0° plies, the effect is not as significant.

7. Apparatus

7.1 *Micrometers*—A micrometer with a 4- to 5-mm [0.16- to 0.20-in] nominal diameter double-ball interface shall be

used to measure the thickness of the specimen. A micrometer with a flat anvil interface shall be used to measure the width of the specimen. The accuracy of the instruments shall be suitable for reading to within 1 % of the sample width and thickness. For typical specimen geometries, an instrument with an accuracy of $\pm 2.5 \mu\text{m}$ [$\pm 0.0001 \text{ in.}$] is adequate for thickness measurement, while an instrument with an accuracy of $\pm 25 \mu\text{m}$ [$\pm 0.001 \text{ in.}$] is adequate for width measurement.

7.2 Testing Machine—The testing machine shall be in conformance with Practices E 4 and shall satisfy the following requirements:

7.2.1 Testing Machine Heads—The testing machine shall have both an essentially stationary head and a movable head.

7.2.2 Drive Mechanism—The testing machine drive mechanism shall be capable of imparting to the movable head a controlled velocity with respect to the stationary head. The velocity of the movable head shall be capable of being regulated as specified in 11.3.

7.2.3 Load Indicator—The testing machine load-sensing device shall be capable of indicating the total load being carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the load with an accuracy over the load range(s) of interest of within $\pm 1 \%$ of the indicated value. The load range(s) of interest may be fairly low for modulus evaluation, much higher for strength evaluation, or both, as required.

NOTE 1—Obtaining precision load data over a large range of interest in the same test, such as when both elastic modulus and ultimate load are being determined, place extreme requirements on the load cell and its calibration. For some equipment, a special calibration may be required. For some combinations of material and load cell, simultaneous precision measurement of both elastic modulus and ultimate strength may not be possible and measurement of modulus and strength may have to be performed in separate tests using a different load cell range for each test.

7.2.4 Grips—Each head of the testing machine shall carry one grip for holding the test specimen so that the direction of load applied to the specimen is coincident with the longitudinal axis of the specimen. The grips shall apply sufficient lateral pressure to prevent slippage between the grip face and the coupon. If tabs are used the grips should be long enough that they overhang the beveled portion of the tab by approximately 10 to 15 mm [0.5 in.]. It is highly desirable to use grips that are rotationally self-aligning to minimize bending stresses in the coupon.

NOTE 2—Grip surfaces that are lightly serrated, approximately 1 serration/mm [25 serrations/in.], have been found satisfactory for use in wedge-action grips when kept clean and sharp; coarse serrations may produce grip-induced failures in untabbed coupons. Smooth gripping surfaces have been used successfully with either hydraulic grips or an emery cloth interface, or both.

7.2.5 System Alignment—Poor system alignment can be a major contributor to premature failure, to elastic property data scatter, or both. Practice E 1012 describes bending evaluation guidelines and describes potential sources of misalignment during tensile testing. In addition to Practice E 1012, the degree of bending in a tensile system can also be evaluated using the following related procedure. Specimen bending is considered separately in 11.6.1.

7.2.5.1 A rectangular alignment coupon, preferably similar in size and stiffness to the test specimen of interest, is instrumented with a minimum of three longitudinal strain gages of similar type, two on the front face across the width and one on the back face of the specimen, as shown in Fig. 1. Any difference in indicated strain between these gages during loading provides a measure of the amount of bending in the thickness plane (B_x) and width plane (B_y) of the coupon. The strain gage location should normally be located in the middle of the coupon gage section (if modulus determination is a concern), near a grip (if premature grip failures are a problem), or any combination of these areas.

7.2.5.2 When evaluating system alignment, it is advisable to perform the alignment check with the same coupon inserted in each of the four possible installation permutations (described relative to the initial position): initial (top-front facing observer), rotated back to front only (top back facing observer), rotated end for end only (bottom front facing observer), and rotated both front to back and end to end (bottom back facing observer). These four data sets provide an indication of whether the bending is due to the system itself or to tolerance in the alignment check coupon or gaging.

7.2.5.3 The zero strain point may be taken either before gripping or after gripping. The strain response of the alignment coupon is subsequently monitored during the gripping process, the tensile loading process, or both. Eq 1-3 use these indicated strains to calculate the ratio of the percentage of bending strain to average extensional strain for each bending plane of the alignment coupon and the total percent bending, B_{total} . Plotting percent bending versus axial average strain is useful in understanding trends in the bending behavior of the system.

7.2.5.4 Problems with failures during gripping would be reason to examine bending strains during the gripping process in the location near the grip. Concern over modulus data scatter would be reason to evaluate bending strains over the modulus evaluation load range for the typical transducer location. Excessive failures near the grips would be reason to evaluate bending strains near the grip at high loading levels. While the

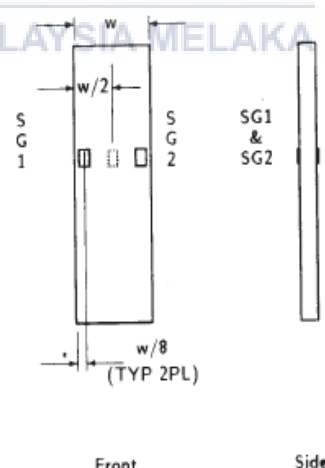


FIG. 1 Gage Locations for System Alignment Check Coupon

maximum advisable amount of system misalignment is material and location dependent, good testing practice is generally able to limit percent bending to a range of 3 to 5 % at moderate strain levels ($>1000 \mu\epsilon$). A system showing excessive bending for the given application should be readjusted or modified.

$$B_y = \frac{\epsilon_{ave} - \epsilon_3}{\epsilon_{ave}} \times 100 \quad (1)$$

$$B_z = \frac{4/3 (\epsilon_2 - \epsilon_1)}{\epsilon_{ave}} \times 100 \quad (2)$$

where:

B_y = percent bending about system y axis (about the narrow plane), as calculated by Eq 1, %;

B_z = percent bending about system z axis (about the wide plane), as calculated by Eq 2, %;

ϵ_1 , ϵ_2 , and ϵ_3 = indicated longitudinal strains displayed by Gages 1, 2, and 3, respectively, of Fig. 1, $\mu\epsilon$; and

ϵ_{ave} = $(\epsilon_1 + \epsilon_2)/2 + \epsilon_3/2$.

The total bending component is:

$$B_{total} = |B_y| + |B_z| \quad (3)$$

7.3 Strain-Indicating Device—Load-strain data, if required, shall be determined by means of either a strain transducer or an extensometer. Attachment of the strain-indicating device to the coupon shall not cause damage to the specimen surface. If Poisson's ratio is to be determined, the specimen shall be instrumented to measure strain in both longitudinal and lateral directions. If the modulus of elasticity is to be determined, the longitudinal strain should be simultaneously measured on opposite faces of the specimen to allow for a correction as a result of any bending of the specimen (see 11.6 for further guidance).

7.3.1 Bonded Resistance Strain Gage Selection—Strain gage selection is a compromise based on the type of material. An active gage length of 6 mm [0.25 in.] is recommended for most materials. Active gage lengths should not be less than 3 mm [0.125 in.].⁷ Gage calibration certification shall comply with Test Methods E 251. When testing woven fabric laminates, gage selection should consider the use of an active gage length that is at least as great as the characteristic repeating unit of the weave. Some guidelines on the use of strain gages on composites follow. A general reference on the subject is Tuttle and Brinson.⁸

7.3.1.1 Surface preparation of fiber-reinforced composites in accordance with Practice E 1237 can penetrate the matrix material and cause damage to the reinforcing fibers resulting in improper coupon failures. Reinforcing fibers should not be exposed or damaged during the surface preparation process.

The strain gage manufacturer should be consulted regarding surface preparation guidelines and recommended bonding agents for composites pending the development of a set of standard practices for strain gage installation surface preparation of fiber-reinforced composite materials.

7.3.1.2 Consideration should be given to the selection of gages having larger resistances to reduce heating effects on low-conductivity materials. Resistances of 350 Ω or higher are preferred. Additional consideration should be given to the use of the minimum possible gage excitation voltage consistent with the desired accuracy (1 to 2 V is recommended) to reduce further the power consumed by the gage. Heating of the coupon by the gage may affect the performance of the material directly, or it may affect the indicated strain as a result of a difference between the gage temperature compensation factor and the coefficient of thermal expansion of the coupon material.

7.3.1.3 Consideration of some form of temperature compensation is recommended, even when testing at standard laboratory atmosphere. Temperature compensation is required when testing in nonambient temperature environments.

7.3.1.4 Consideration should be given to the transverse sensitivity of the selected strain gage. The strain gage manufacturer should be consulted for recommendations on transverse sensitivity corrections and effects on composites. This is particularly important for a transversely mounted gage used to determine Poisson's ratio, as discussed in Note 11.

7.3.2 Extensometers—For most purposes, the extensometer gage length should be in the range of 10 to 50 mm [0.5 to 2.0 in.]. Extensometers shall satisfy, at a minimum, Practice E 83, Class B-1 requirements for the strain range of interest and shall be calibrated over that strain range in accordance with Practice E 83. For extremely stiff materials, or for measurement of transverse strains, the fixed error allowed by Class B-1 extensometers may be significant, in which case Class A extensometers should be considered. The extensometer shall be essentially free of inertia lag at the specified speed of testing, and the weight of the extensometer should not induce bending strains greater than those allowed in 6.3.

NOTE 3—It is generally less difficult to perform strain calibration on extensometers of longer gage length as less precision in displacement is required of the extensometer calibration device.

7.4 Conditioning Chamber—When conditioning materials at nonlaboratory environments, a temperature/vaporlevel-controlled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to within $\pm 3^\circ\text{C}$ [$\pm 5^\circ\text{F}$] and the required relative vapor level to within $\pm 3\%$. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

7.5 Environmental Test Chamber—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the gage section of the test specimen at the required test environment during the mechanical test.

⁷ A typical gage would have a 0.25-in. active gage length, 350- Ω resistance, a strain rating of 3 % or better, and the appropriate environmental resistance and thermal coefficient.

⁸ Tuttle, M. E. and Brinson, H. F., "Resistance-Foil Strain-Gage Technology as Applied to Composite Materials," *Experimental Mechanics*, Vol 24, No. 1, March 1984; pp. 54-65; errata noted in Vol 26, No. 2, June 1986, pp. 153-154.

8. Sampling and Test Specimens

8.1 *Sampling*—Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E 122 should be consulted. Report the method of sampling.

NOTE 4—If specimens are to undergo environmental conditioning to equilibrium, and are of such type or geometry that the weight change of the material cannot be properly measured by weighing the specimen itself (such as a tabbed mechanical coupon), then use another traveler coupon of the same nominal thickness and appropriate size (but without tabs) to determine when equilibrium has been reached for the specimens being conditioned.

8.2 *Geometry*—Design of mechanical test coupons, especially those using end tabs, remains to a large extent an art rather than a science, with no industry consensus on how to approach the engineering of the gripping interface. Each major composite testing laboratory has developed gripping methods for the specific material systems and environments commonly encountered within that laboratory. Comparison of these methods shows them to differ widely, making it extremely difficult to recommend a universally useful approach or set of approaches. Because of this difficulty, definition of the geometry of the test coupon is broken down into the following three levels, which are discussed further in each appropriate section:

Purpose	Degree of Geometry Definition
8.2.1 <i>General Requirements</i>	Mandatory Shape and Tolerances
8.2.2 <i>Specific Recommendations</i>	Nonmandatory Suggested Dimensions
8.2.3 <i>Detailed Examples</i>	Nonmandatory Typical Practices

8.2.1 General Requirements:

8.2.1.1 *Shape, Dimensions, and Tolerances*—The complete list of requirements for specimen shape, dimensions, and tolerances is shown in Table 1.

8.2.1.2 *Use of Tabs*—Tabs are not required. The key factor in the selection of specimen tolerances and gripping methods is the successful introduction of load into the specimen and the prevention of premature failure as a result of a significant discontinuity. Therefore, determine the need to use tabs, and specification of the major tab design parameters, by the end

result: acceptable failure mode and location. If acceptable failure modes occur with reasonable frequency, then there is no reason to change a given gripping method (see 11.10).

8.2.2 Specific Recommendations:

8.2.2.1 *Width, Thickness, and Length*—Select the specimen width and thickness to promote failure in the gage section and assure that the specimen contains a sufficient number of fibers in the cross section to be statistically representative of the bulk material. The specimen length should normally be substantially longer than the minimum requirement to minimize bending stresses caused by minor grip eccentricities. Keep the gage section as far from the grips as reasonably possible and provide a significant amount of material under stress and therefore produce a more statistically significant result. The minimum requirements for specimen design shown in Table 1 are by themselves insufficient to create a properly dimensioned and toleranced coupon drawing. Therefore, recommendations on other important dimensions are provided for typical material configurations in Table 2. These geometries have been found by a number of testing laboratories to produce acceptable failure modes on a wide variety of material systems, but use of them does not guarantee success for every existing or future material system.

8.2.2.2 *Gripping/Use of Tabs*—There are many material configurations, such as multidirectional laminates, fabric-based materials, or randomly reinforced sheet-molding compounds, which can be successfully tested without tabs. However, tabs are strongly recommended when testing unidirectional materials (or strongly unidirectionally dominated laminates) to failure in the fiber direction. Tabs may also be required when testing unidirectional materials in the matrix direction to prevent gripping damage.

8.2.2.3 *Tab Geometry*—Recommendations on important dimensions are provided for typical material configurations in Table 2. These dimensions have been found by a number of testing laboratories to produce acceptable failure modes on a wide variety of material systems, but use of them does not guarantee success for every existing or future material system. The selection of a tab configuration that can successfully produce a gage section tensile failure is dependent upon the coupon material, coupon ply orientation, and the type of grips being used. When pressure-operated nonwedge grips are used with care, squared-off 90° tabs have been used successfully. Wedge-operated grips have been used most successfully with tabs having low bevel angles (7 to 10°) and a feathered smooth transition into the coupon. For alignment purposes, it is essential that the tabs be of matched thickness.

8.2.2.4 *Friction Tabs*—Tabs need not always be bonded to the material under test to be effective in introducing the load into the specimen. Friction tabs, essentially nonbonded tabs held in place by the pressure of the grip, and often used with emery cloth or some other light abrasive between the tab and the coupon, have been successfully used in some applications. In specific cases, lightly serrated wedge grips (see Note 2) have been successfully used with only emery cloth as the interface between the grip and the coupon. However, the abrasive used

TABLE 1 Tensile Specimen Geometry Requirements

Parameter	Requirement
Coupon Requirements:	
shape	constant rectangular cross-section
minimum length	gripping + 2 times width + gage length
specimen width	as needed ⁴
specimen width tolerance	±1 % of width
specimen thickness	as needed
specimen thickness tolerance	±4 % of thickness
specimen flatness	flat with light finger pressure
Tab Requirements (if used):	
tab material	as needed
fiber orientation (composite tabs)	as needed
tab thickness	as needed
tab thickness variation between	±1 % tab thickness
tabs	
tab bevel angle	5 to 90°, inclusive
tab step at bevel to specimen	feathered without damaging specimen

⁴ See 8.2.2 or Table 2 for recommendations.

TABLE 2 Tensile Specimen Geometry Recommendations⁴

Fiber Orientation	Width, mm [in.]	Overall Length, mm [in.]	Thickness, mm [in.]	Tab Length, mm [in.]	Tab Thickness, mm [in.]	Tab Bevel Angle, °
0° unidirectional	15 [0.5]	250 [10.0]	1.0 [0.040]	56 [2.25]	1.5 [0.062]	7 or 90
90° unidirectional	25 [1.0]	175 [7.0]	2.0 [0.080]	25 [1.0]	1.5 [0.062]	90
balanced and symmetric	25 [1.0]	250 [10.0]	2.5 [0.100]	emery cloth	—	—
random-discontinuous	25 [1.0]	250 [10.0]	2.5 [0.100]	emery cloth	—	—

⁴ Dimensions in this table and the tolerances of Fig. 2 or Fig. 3 are recommendations only and may be varied so long as the requirements of Table 1 are met.

must be able to withstand significant compressive loads. Some types of emery cloth have been found ineffective in this application because of disintegration of the abrasive.⁹

8.2.2.5 Tab Material—The most consistently used bonded tab material has been continuous E-glass fiber-reinforced polymer matrix materials (woven or unwoven) in a [0/90]_ns laminate configuration. The tab material is commonly applied at 45° to the loading direction to provide a soft interface. Other configurations that have reportedly been successfully used have incorporated steel tabs or tabs made of the same material as is being tested.

8.2.2.6 Bonded Tab Length—When using bonded tabs, estimate the minimum suggested tab length for bonded tabs by the following simple equation. As this equation does not account for the peaking stresses that are known to exist at the ends of bonded joints. The tab length calculated by this equation should normally be increased by some factor to reduce the chances of joint failure:

$$L_{\min} = F_{\min}^a h / 2F_{\min}^b \quad (4)$$

where:

L_{\min} = minimum required bonded tab length, mm [in.];
 F_{\min}^a = ultimate tensile strength of coupon material, MPa [psi];
 h = coupon thickness, mm [in.]; and
 F_{\min}^b = ultimate shear strength of adhesive, coupon material, or tab material (whichever is lowest), MPa [psi].

8.2.2.7 Bonded Tab Adhesive—Any high-elongation (tough) adhesive system that meets the environmental requirements may be used when bonding tabs to the material under test. A uniform bondline of minimum thickness is desirable to reduce undesirable stresses in the assembly.

8.2.3 Detailed Examples—The minimum requirements for specimen design discussed in 8.2.1 are by themselves insufficient to create a properly dimensioned and toleranced coupon drawing. Dimensionally toleranced specimen drawings for both tabbed and untabbed forms are shown as examples in Fig. 2 (SI) and Fig. 3 (inch-pound). The tolerances on these drawings are fixed, but satisfy the requirements of Table 1 for all of the recommended configurations of Table 2. For a specific configuration, the tolerances on Fig. 2 and Fig. 3 might be able to be relaxed.

8.3 Specimen Preparation:

8.3.1 Panel Fabrication—Control of fiber alignment is critical. Improper fiber alignment will reduce the measured

properties. Erratic fiber alignment will also increase the coefficient of variation. The specimen preparation method shall be reported.

8.3.2 Machining Methods—Specimen preparation is extremely important for this specimen. Mold the specimens individually to avoid edge and cutting effects or cut from them plates. If they are cut from plates, take precautions to avoid notches, undercuts, rough or uneven surfaces, or delaminations caused by inappropriate machining methods. Obtain final dimensions by water-lubricated precision sawing, milling, or grinding. The use of diamond tooling has been found to be extremely effective for many material systems. Edges should be flat and parallel within the specified tolerances.

8.3.3 Labeling—Label the coupons so that they will be distinct from each other and traceable back to the raw material and in a manner that will both be unaffected by the test and not influence the test.

9. Calibration

9.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

10. Conditioning

10.1 Standard Conditioning Procedure—Unless a different environment is specified as part of the experiment, condition the test specimens in accordance with Procedure C of Test Method D 5229/D 5229M and store and test at standard laboratory atmosphere (23 ± 3°C [73 ± 5°F] and 50 ± 10 % relative humidity).

11. Procedure

11.1 Parameters To Be Specified Before Test:

11.1.1 The tension specimen sampling method, coupon type and geometry, and conditioning travelers (if required).

11.1.2 The tensile properties and data reporting format desired.

NOTE 5—Determine specific material property, accuracy, and data reporting requirements before test for proper selection of instrumentation and data-recording equipment. Estimate operating stress and strain levels to aid in transducer selection, calibration of equipment, and determination of equipment settings.

11.1.3 The environmental conditioning test parameters.

11.1.4 If performed, the sampling method, coupon geometry, and test parameters used to determine density and reinforcement volume.

11.2 General Instructions:

11.2.1 Report any deviations from this test method, whether intentional or inadvertent.

⁹ E-Z Flex Metalite K224 cloth, Grit 120-J, available from Norton Company, Troy, NY 12181, has been found satisfactory in this application. Other equivalent types of emery cloth should also be suitable.

DRAWING NOTES:

1. INTERPRET DRAWING IN ACCORDANCE WITH ANSI Y14.5M-1982, SUBJECT TO THE FOLLOWING:
2. ALL DIMENSIONS IN MILLIMETRES WITH DECIMAL TOLERANCES AS FOLLOWS:

NO DECIMAL	X	XX
± 3	± 1	± .3
3. ALL ANGLES HAVE TOLERANCE OF $\pm .5^\circ$.
4. PLY ORIENTATION DIRECTION TOLERANCE RELATIVE TO $[-A-]$ WITHIN $\pm .5^\circ$.
5. FINISH ON MACHINED EDGES NOT TO EXCEED $1.6\sqrt{}$ (SYMBOLOLOGY IN ACCORDANCE WITH ASA B46.1, WITH ROUGHNESS HEIGHT IN MICROMETRES.)
6. VALUES TO BE PROVIDED FOR THE FOLLOWING, SUBJECT TO ANY RANGES SHOWN ON THE FIELD OF DRAWING: MATERIAL, LAY-UP, PLY ORIENTATION REFERENCE RELATIVE TO $[-A-]$ OVERALL LENGTH, GAGE LENGTH, COUPON THICKNESS, TAB MATERIAL, TAB THICKNESS, TAB LENGTH, TAB BEVEL ANGLE, TAB ADHESIVE.
7. NO ADHESIVE BUILDUP ALLOWED IN THIS AREA.

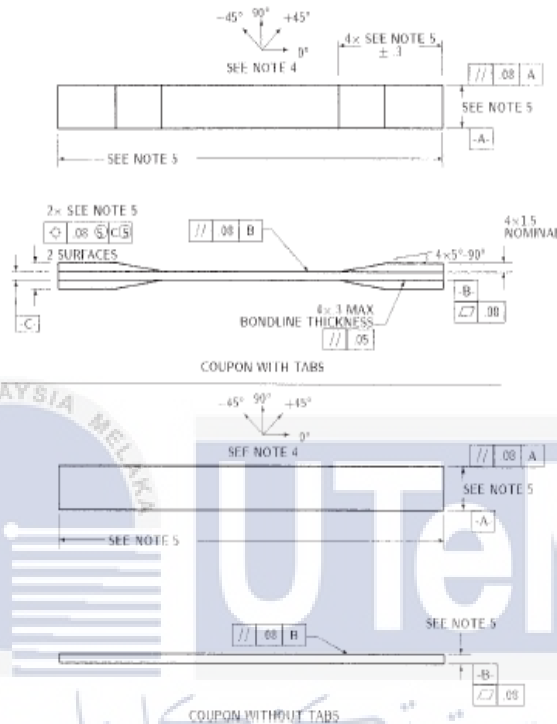


FIG. 2 Tension Test Specimen Drawing (SI)

11.2.2 If specific gravity, density, reinforcement volume, or void volume are to be reported, then obtain these samples from the same panels being tension tested. Specific gravity and density may be evaluated by means of Test Methods D 792. Volume percent of the constituents may be evaluated by one of the matrix digestion procedures of Test Method D 3171, or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method D 2584. The void content equations of Test Methods D 2734 are applicable to both Test Method D 2584 and the matrix digestion procedures.

11.2.3 Following final specimen machining and any conditioning, but before the tension testing, determine the specimen area as $A = w \times h$, at three places in the gage section, and report the area as the average of these three determinations to the accuracy in 7.1. Record the average area in units of mm^2 (in^2).

11.3 *Speed of Testing*—Set the speed of testing to effect a nearly constant strain rate in the gage section. If strain control is not available on the testing machine, this may be approximated by repeated monitoring and adjusting of the rate of load application to maintain a nearly constant strain rate, as mea-

sured by strain transducer response versus time. The strain rate should be selected so as to produce failure within 1 to 10 min. If the ultimate strain of the material cannot be reasonably estimated, initial trials should be conducted using standard speeds until the ultimate strain of the material and the compliance of the system are known, and the strain rate can be adjusted. The suggested standard speeds are:

11.3.1 *Strain-Controlled Tests*—A standard strain rate of 0.01 min^{-1} .

11.3.2 *Constant Head-Speed Tests*—A standard head displacement rate of 2 mm/min [0.05 in./min].

NOTE 6—Use of a fixed head speed in testing machine systems with a high compliance may result in a strain rate that is much lower than required. Use of wedge grips can cause extreme compliance in the system, especially when using compliant tab materials. In some such cases, actual strain rates 10 to 50 times lower than estimated by head speeds have been observed.

11.4 *Test Environment*—Condition the specimen to the desired moisture profile and, if possible, test under the same conditioning fluid exposure level. However, cases such as

DRAWING NOTES:

1. INTERPRET DRAWING IN ACCORDANCE WITH ANSI Y14.5M-1982, SUBJECT TO THE FOLLOWING:
2. ALL DIMENSIONS IN INCHES WITH DECIMAL TOLERANCES AS FOLLOWS:

.X	.XX	.XXX
±.1	±.03	±.01
3. ALL ANGLES HAVE TOLERANCE OF ±5°.
4. PLY ORIENTATION DIRECTION TOLERANCE RELATIVE TO [A] WITHIN ±5°.
5. FINISH ON MACHINED EDGES NOT TO EXCEED 64√ (SYMBOLGY IN ACCORDANCE WITH ASA B46.1, WITH ROUGHNESS HEIGHT IN MICROINCHES.)
6. VALUES TO BE PROVIDED FOR THE FOLLOWING, SUBJECT TO ANY RANGES SHOWN ON THE FIELD OF DRAWING: MATERIAL, LAY-UP, PLY ORIENTATION REFERENCE RELATIVE TO [A], OVERALL LENGTH, GAGE LENGTH, COUPON THICKNESS, TAB MATERIAL, TAB THICKNESS, TAB LENGTH, TAB BEVEL ANGLE, TAB ADHESIVE.
7. NO ADHESIVE BUILDUP ALLOWED IN THIS AREA.

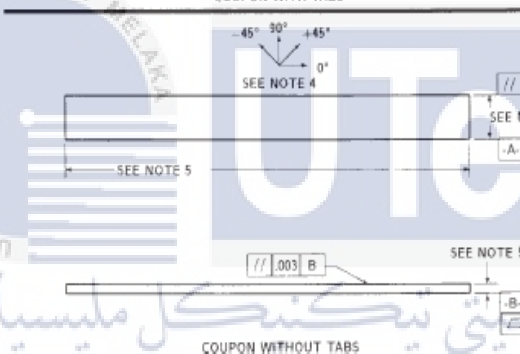
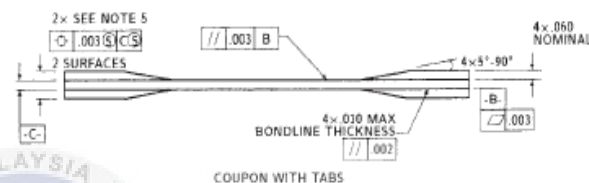
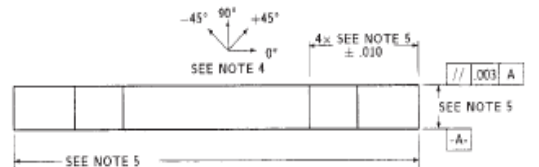


FIG. 3 Tension Test Specimen Drawing (inch-pound)

elevated temperature testing of a moist specimen place unrealistic requirements on the capabilities of common testing machine environmental chambers. In such cases, the mechanical test environment may need to be modified, for example, by testing at elevated temperature with no fluid exposure control, but with a specified limit on time to failure from withdrawal from the conditioning chamber. Modifications to the test environment shall be recorded.

11.4.1 Store the specimen in the conditioned environment until test time, if the testing area environment is different than the conditioning environment.

11.5 *Specimen Insertion*—Place the specimen in the grips of the testing machine, taking care to align the long axis of the gripped specimen with the test direction. Tighten the grips, recording the pressure used on pressure controllable (hydraulic or pneumatic) grips.

NOTE 7—The ends of the grip jaws on wedge-type grips should be even with each other following insertion to avoid inducing a bending moment that results in premature failure of the specimen at the grip. When using

untabbed specimens, a folded strip of medium grade (80 to 150 grit) emery cloth between the specimen faces and the grip jaws (grit-side toward specimen) provides a nonslip grip on the specimen without jaw serration damage to the surface of the specimen. When using tabbed specimens, insert the coupon so that the grip jaws extend approximately 10 to 15 mm [0.5 in.] past the beginning of the tapered portion of the tab. Coupons having tabs that extend beyond the grips are prone to failure at the tab ends because of excessive interlaminar stresses.

11.6 *Transducer Installation*—If strain response is to be determined attach the strain-indication transducer(s) to the specimen, symmetrically about the mid-span, mid-width location. Attach the strain-recording instrumentation to the transducers on the specimen.

11.6.1 When determining modulus of elasticity, it is recommended that at least one specimen per like sample be evaluated with back-to-back axial transducers to evaluate the percent bending, using Eq 5, at the average axial strain checkpoint value (the mid range of the appropriate chord modulus strain range) shown in Table 3. A single transducer can be used if the

TABLE 3 Specimen Alignment and Chord Modulus Calculation Strain Ranges

Tensile Chord Modulus Calculation Longitudinal Strain Range		Longitudinal Strain Checkpoint for Bending
Start Point	End Point	
$\mu\epsilon^A$	$\mu\epsilon$	$\mu\epsilon$
1000 ^B	3000	2000

^A 1000 $\mu\epsilon$ = 0.001 absolute strain.

^B This strain range is to be contained in the lower half of the stress/strain curve. For materials that fail below 6000 $\mu\epsilon$, a strain range of 25 to 50 % of ultimate is recommended.

percent bending is no more than 3 %. When bending is greater than 3 % averaged strains from back-to-back transducers of like kind are recommended.

$$B_y = \frac{|\epsilon_f - \epsilon_b|}{|\epsilon_f + \epsilon_b|} \quad (5)$$

where:

ϵ_f = indicated strain from front transducer, $\mu\epsilon$;

ϵ_b = indicated strain from back transducer, $\mu\epsilon$; and

B_y = percent bending in specimen.

11.7 Loading—Apply the load to the specimen at the specified rate until failure, while recording data.

11.8 Data Recording—Record load versus strain (or transducer displacement) continuously or at frequent regular intervals. If a transition region or initial ply failures are noted, record the load, strain, and mode of damage at such points. If the specimen is to be failed, record the maximum load, the failure load, and the strain (or transducer displacement) at, or as near as possible to, the moment of rupture.

NOTE 8—Other valuable data that can be useful in understanding testing anomalies and gripping or specimen slipping problems includes load versus head displacement data and load versus time data.

11.9 Failure Mode—Record the mode and location of failure of the specimen. Choose, if possible, a standard description using the three-part failure mode code that is shown in Fig. 4.

11.10 Grip/Tab Failures—Reexamine the means of load introduction into the material if a significant fraction of failures in a sample population occur within one specimen width of the tab or grip. Factors considered should include the tab alignment, tab material, tab angle, tab adhesive, grip type, grip pressure, and grip alignment.

12. Calculation

12.1 Tensile Stress/Tensile Strength—Calculate the ultimate tensile strength using Eq 6 and report the results to three significant figures. If the tensile modulus is to be calculated, determine the tensile stress at each required data point using Eq 7.

$$F^u = P^{\max}/A \quad (6)$$

$$\sigma_i = P_i/A \quad (7)$$

where:

F^u = ultimate tensile strength, MPa [psi];

P^{\max} = maximum load before failure, N [lbf];

σ_i = tensile stress at i th data point, MPa [psi];

P_i = load at i th data point, N [lbf]; and

A = average cross-sectional area from 11.2.3, mm² [in.²].

12.2 Tensile Strain/Ultimate Tensile Strain—If tensile modulus or ultimate tensile strain is to be calculated, and material response is being determined by an extensometer, determine the tensile strain from the indicated displacement at each required data point using Eq 8 and report the results to three significant figures.

$$\epsilon_i = \delta_i/L_g \quad (8)$$

where:

ϵ_i = tensile strain at i th data point, $\mu\epsilon$;

δ_i = extensometer displacement at i th data point, mm [in.]; and

L_g = extensometer gage length, mm [in.].

12.3 Tensile Modulus of Elasticity:

NOTE 9—To minimize potential effects of bending it is recommended that the strain data used for modulus of elasticity determination be the average of the indicated strains from each side of the specimen, as discussed in 7.3 and 11.6.

12.3.1 Tensile Chord Modulus of Elasticity—Select the appropriate chord modulus strain range from Table 3. Calculate the tensile chord modulus of elasticity from the stress-strain data using Eq 9. If data is not available at the exact strain range end points (as often occurs with digital data), use the closest available data point. Report the tensile chord modulus of elasticity to three significant figures. Also report the strain range used in the calculation. A graphical example of chord modulus is shown in Fig. 5.

12.3.1.1 The tabulated strain ranges should only be used for materials that do not exhibit a transition region (a significant change in the slope of the stress-strain curve) within the given strain range. If a transition region occurs within the recommended strain range, then a more suitable strain range shall be used and reported.

$$E^{\text{chord}} = \Delta\sigma/\Delta\epsilon \quad (9)$$

where:

E^{chord} = tensile chord modulus of elasticity, GPa [psi];

$\Delta\sigma$ = difference in applied tensile stress between the two strain points of Table 3, MPa [psi]; and

$\Delta\epsilon$ = difference between the two strain points of Table 3 (nominally 0.002).

12.3.2 Tensile Modulus of Elasticity (Other Definitions)—Other definitions of elastic modulus may be evaluated and reported at the user's discretion. If such data is generated and reported, report also the definition used, the strain range used,

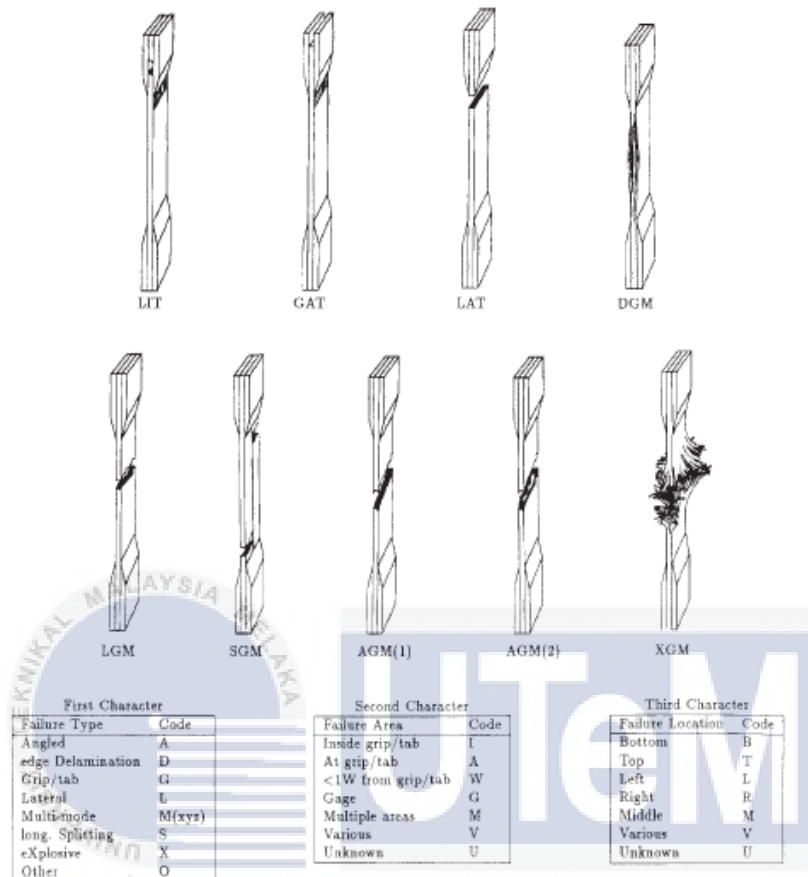


FIG. 4 Tensile Test Failure Codes/Typical Modes

and the results to three significant figures. Test Method E 111 provides additional guidance in the determination of modulus of elasticity.

NOTE 10—An example of another modulus definition is the secondary chord modulus of elasticity for materials that exhibit essentially bilinear stress-strain behavior. An example of secondary chord modulus is shown in Fig. 5.

12.4 Poisson's Ratio:

NOTE 11—If bonded resistance strain gages are being used, the error produced by the transverse sensitivity effect on the transverse gage will generally be much larger for composites than for metals. An accurate measurement of Poisson's ratio requires correction for this effect. The strain gage manufacturer should be contacted for information on the use of correction factors for transverse sensitivity.

12.4.1 Poisson's Ratio By Chord Method—Select the appropriate chord modulus longitudinal strain range from Table 3. Determine (by plotting or otherwise) the transverse strain (measured perpendicular to the applied load), ϵ_t , at each of the two longitudinal strains (measured parallel to the applied load), ϵ_l , strain range end points. If data is not available at the exact strain range end points (as often occurs with digital data), use

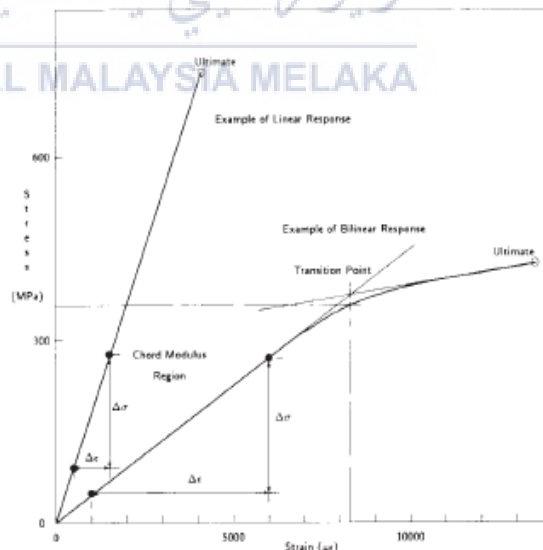


FIG. 5 Typical Tensile Stress-Strain Curves

the closest available data point. Calculate Poisson's ratio by Eq 10 and report to three significant figures. Also report the strain range used.

$$\nu = -\Delta\epsilon_t/\Delta\epsilon_l \quad (10)$$

where:

ν = Poisson's ratio;

$\Delta\epsilon_t$ = difference in lateral strain between the two longitudinal strain points of Table 3, $\mu\epsilon$; and

$\Delta\epsilon_l$ = difference between the two longitudinal strain points of Table 3 (nominally either 0.001, 0.002, or 0.005).

12.4.2 Tensile Poisson's Ratio (Other Definitions)—Other definitions of Poisson's ratio may be evaluated and reported at the user's direction. If such data is generated and reported, report also the definition used, the strain range used, and the results to three significant figures. Test Method E 132 provides additional guidance in the determination of Poisson's ratio.

12.5 Transition Strain—Where applicable, determine the transition strain from either the bilinear longitudinal stress versus longitudinal strain curve or the bilinear transverse strain versus longitudinal strain curve. Create a best linear fit or chord line for each of the two linear regions and extend the lines until they intersect. Determine to three significant digits the longitudinal strain that corresponds to the intersection point and record this value as the transition strain. Report also the method of linear fit (if used) and the strain ranges over which the linear fit or chord lines were determined. A graphical example of transition strain is shown in Fig. 5.

12.6 Statistics—For each series of tests calculate the average value, standard deviation and coefficient of variation (in percent) for each property determined:

$$\bar{x} = (\sum_{i=1}^n x_i)/n \quad (11)$$

$$s_{n-1} = \sqrt{(\sum_{i=1}^n x_i^2 - n\bar{x}^2)/(n-1)} \quad (12)$$

$$CV = 100 \times s_{n-1}/\bar{x} \quad (13)$$

where:

\bar{x} = sample mean (average);

s_{n-1} = sample standard deviation;

CV = sample coefficient of variation, in percent;

n = number of specimens; and

x_i = measured or derived property.

13. Report

13.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable (reporting of items beyond the control of a given testing laboratory, such as might occur with material details or panel fabrication parameters, shall be the responsibility of the requestor):

13.1.1 The revision level or date of issue of this test method.

13.1.2 The date(s) and location(s) of the test.

13.1.3 The name(s) of the test operator(s).

13.1.4 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.

13.1.5 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch number, source (if not from manufacturer), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, prepreg matrix content, and prepreg volatiles content.

13.1.6 Description of the fabrication steps used to prepare the laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used.

13.1.7 Ply orientation stacking sequence of the laminate.

13.1.8 If requested, report density, volume percent reinforcement, and void content test methods, specimen sampling method and geometries, test parameters, and test results.

13.1.9 Average ply thickness of the material.

13.1.10 Results of any nondestructive evaluation tests.

13.1.11 Method of preparing the test specimen, including specimen labeling scheme and method, specimen geometry, sampling method, coupon cutting method, identification of tab geometry, tab material, and tab adhesive used.

13.1.12 Calibration dates and methods for all measurement and test equipment.

13.1.13 Type of test machine, grips, jaws, grip pressure, alignment results, and data acquisition sampling rate and equipment type.

13.1.14 Results of system alignment evaluations, if any such were done.

13.1.15 Dimensions of each test specimen.

13.1.16 Conditioning parameters and results, use of travelers and traveler geometry, and the procedure used if other than that specified in the test method.

13.1.17 Relative humidity and temperature of the testing laboratory.

13.1.18 Environment of the test machine environmental chamber (if used) and soak time at environment.

13.1.19 Number of specimens tested.

13.1.20 Speed of testing.

13.1.21 Transducer placement on the specimen and transducer type for each transducer used.

13.1.22 If strain gages were used, the type, resistance, size, gage factor, temperature compensation method, transverse sensitivity, lead-wire resistance, and any correction factors used.

13.1.23 Stress-strain curves and tabulated data of stress versus strain for each specimen.

13.1.24 Percent bending results for each specimen so evaluated.

13.1.25 Individual strengths and average value, standard deviation, and coefficient of variation (in percent) for the population. Note if the failure load was less than the maximum load before failure.

13.1.26 Individual strains at failure and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.27 Strain range used for chord modulus and Poisson's ratio determination.

13.1.28 If another definition of modulus of elasticity is used in addition to chord modulus, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

13.1.29 Individual values of modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.30 If another definition of Poisson's ratio is used in addition to the chordwise definition, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

13.1.31 Individual values of Poisson's ratio, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.32 If transition strain is determined, the method of linear fit (if used) and the strain ranges over which the linear fit or chord lines were determined.

13.1.33 Individual values of transition strain (if applicable), and the average value, standard deviation, and coefficient of variation (in percent) for the population.

13.1.34 Failure mode and location of failure for each specimen.

14. Precision and Bias

14.1 Precision:

14.1.1 The precision and bias of tension test strength and modulus measurements depend on strict adherence to the Test Method D 3039/D 3039M and are influenced by mechanical and material factors, specimen preparation, and measurement errors.

14.1.2 Mechanical factors that can affect the test results include: the physical characteristics of the testing machine (stiffness, damping, and mass), accuracy of loading and displacement/strain measurement, speed of loading, alignment of test specimen with applied load, parallelism of the grips, grip pressure, and type of load control (displacement, strain, or load).

14.1.3 Material factors that can affect test results include: material quality and representativeness, sampling scheme, and specimen preparation (dimensional accuracy, tab material, tab taper, tab adhesive, and so forth).

14.1.4 The mean tensile strength for a strain rate sensitive, glass/epoxy tape composite testing in the fiber direction was found to increase by approximately two standard deviations with decreasing time to failure tested at the limits of the recommended time to failure prescribed in Test Method D 3039/D 3039M. This result suggest that caution must be used when comparing test data obtained for strain rate sensitive composite materials tested in accordance with this standard.

14.1.5 Measurement errors arise from the use of specialized measuring instruments such as load cells, extensometers and strain gages, micrometers, data acquisition devices, and so forth.

14.1.6 Data obtained from specimens that fracture outside the gage area should be used with caution as this data may not be representative of the material. Failure in the grip region indicates the stress concentration at the tab is greater than the

natural strength variation of the material in the gage section. A tapered tab, bonded with a ductile low-modulus adhesive has a relatively low-stress concentration and should result in the lowest frequency of grip failures. Low-strength bias increases with the frequency of grip failures by an amount proportional to the stress concentration at the tab.

14.1.7 An interlaboratory test program was conducted where an average of five specimens each, of six different materials and lay-up configurations, were tested by nine different laboratories.¹⁰ Table 4 presents the precision statistics generated from this study as defined in Practice E 691 for tensile strength, modulus, and failure strain. All data except that for Material B (90° lay-up) was normalized with respect to an average thickness. The materials listed in Table 15 are defined as:

- A IM-6/3501-6 uni-tape (0)n
- B IM-6/3501-6 uni-tape (90)n
- C IM-6/3501-6 uni-tape (90/0)n
- F Glass/epoxy fabric (7781 glass/Ciba R 7376 Epoxy)-warp aligned
- G Carbon/epoxy fabric (66108 carbon/Ciba R 6376

TABLE 4 Precision Statistics

Material	\bar{x}	$s \bar{x}$	S_r	S_R	$S_r/\bar{x}, \%$	$S_R/\bar{x}, \%$
Strength, ksi						
A	342.69	8.49	10.68	12.78	3.12	3.73
B	8.52	0.52	0.85	0.82	9.84	10.84
C	156.37	3.84	10.85	10.85	6.94	6.94
F	66.18	3.20	1.52	3.48	2.30	5.26
G	121.52	1.59	3.92	3.92	3.23	3.23
Modulus, Msi						
A	23.57	0.65	0.63	0.86	2.69	3.66
B	1.30	0.05	0.04	0.06	3.12	4.57
C	12.38	0.29	0.37	0.44	2.98	3.54
F	3.95	0.08	0.04	0.09	1.01	2.28
G	9.47	0.16	0.12	0.20	1.29	2.06
Failure Strain, %						
A	1.36	0.08	0.07	0.08	4.65	6.15
B	0.66	0.04	0.08	0.09	12.47	13.02
C	1.22	0.03	0.06	0.06	5.25	5.27
F	2.04	0.15	0.07	0.16	3.19	8.03
G	1.27	0.03	0.05	0.05	3.83	4.13

14.1.8 The averages of the coefficients of variation are in Table 5. The values of S_r/\bar{x} and S_R/\bar{x} represent the repeatability and the reproducibility coefficients of variation, respectively. These averages permit a relative comparison of the repeatability (within laboratory precision) and reproducibility (between laboratory precision) of the tension test parameters. Overall, this indicates that the failure strain measurements exhibit the least repeatability and reproducibility of all the parameters measured while modulus was found to provide the highest repeatability and reproducibility of the parameters measured.

¹⁰ International Harmonization of Composite Materials—Phase 1: Harmonization of ASTM D 3039/D 3039M and ISO 527-5, Final Report, ASTM Institute for Standards Research, April 1997.

TABLE 5 Averages of the Coefficients of Variation

Parameter	Average of S_y/X , %	Average of S_R/X , %
Strength	5.11	6.00
Modulus	2.22	3.22
Failure strain	5.94	7.32

14.1.9 The consistency of agreement for repeated tests of the same material is dependent on lay-up configuration, mate-

rial and specimen preparation techniques, test conditions, and measurements of the tension test parameters.

14.2 *Bias*—Bias cannot be determined for this test method as no acceptable reference standard exists.

15. Keywords

15.1 composite materials; modulus of elasticity; Poisson's ratio; tensile properties; tensile strength

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