CHARACTERIZATON OF CARBON FIBER IN MICRO- AND NANOSTRUCTURE USING ELECTRON MICROSCOPY IMAGE PROCESSING

TIAL CUAI MAN

A THESIS REPORT SUBMITTED IN PARTIAL FULFILLMENT
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THESIS TITLE	Characterization of Carbon Fiber Micro- and Nanostructure using Electron Microscopy Image Processing
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ABSTRACT

Nowadays, everything is changing to the better technology not only in the automotive field, but also in all vehicles, daily usage component especially in weight reduction for transportation. Composite is one of the effective material in many field due to its light weight, high stiffness and good chemical resistance. In current research, characterization and morphologies of carbon fiber and glass fiber are studied by using several equipment to investigate structure, morphology and characteristic of different fibers. Scanning electron microscopy (SEM), transmission electron microscopy (TEM), x-ray diffraction (XRD), Raman spectroscopy (RS) and Fourier transfer infrared (FTIR) are used in this research. The structure of fibers are examine by SEM, TEM, RS and XRD, and found that the structure of carbon fiber is the mixing of graphite and turbostratic while glass fiber give amorphous structure. Composites are fabricated using hand lay up process for all four types of carbon and glass fiber composite. The composite are tested in mechanical test for tensile properties, flexural properties and nanoindentation properties. Carbon fiber twill (CFT) and glass fiber woven (GFW) get the highest mechanical properties and the mechanical properties are increase with the increase of curing temperature. Tensile strength are increase 7% for CFT and 8% for GFW after the specimens were cured at 120ºC for 3hrs. Treatment on fiber surface are performed by using acetone solution to remove the sizing material from fiber surface and fabricate composite to investigate mechanical properties of treated fiber composite through tensile and flexural properties. Composite of treatment fiber give the improvement of mechanical properties for glass fiber while carbon fiber are not increase mechanical properties after treatment. The fracture mechanism of carbon fiber and glass fiber composite was investigated using scanning electron microscopy and found that the main fracture mechanism is debonding fiber from matrix for CFT and pull out fiber for GFW due to their bonding weakness between fiber and matrix. The fracture mechanism of glass fiber shows strong adhesive to the highest temperature cure at 120ºC.

Keywords: carbon fiber composite, glass fiber composite, morphology of fiber, mechanical properties, fracture mechanism

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CHAPTER 1
INTRODUCTION

1.1 Research Background

The role and the usage percentage of carbon fiber and glass fiber composite are growing rapidly due to their unique characters such as lightweight to the same weight ratio, high strength, high stiffness, high chemical resistance and etc. Nowadays, most of carbon fiber and glass fiber are used as a fiber to reinforce by matrix thermoset epoxy resin for better strength. Several composite were made not only in automotive parts, marine, aircraft, but also in sports, bicycles, tennis, golf club and furniture of glass fiber. Especially, composite of carbon fiber and glass fiber were used military aircraft, aerospace, automotive and transportation. Most of the automotive manufacturer is trying to replace conventional material steel with composite due to their stiffness and light weight [3]. Figure 1.1 shows 50% of material were composite material of carbon fiber and glass fiber.

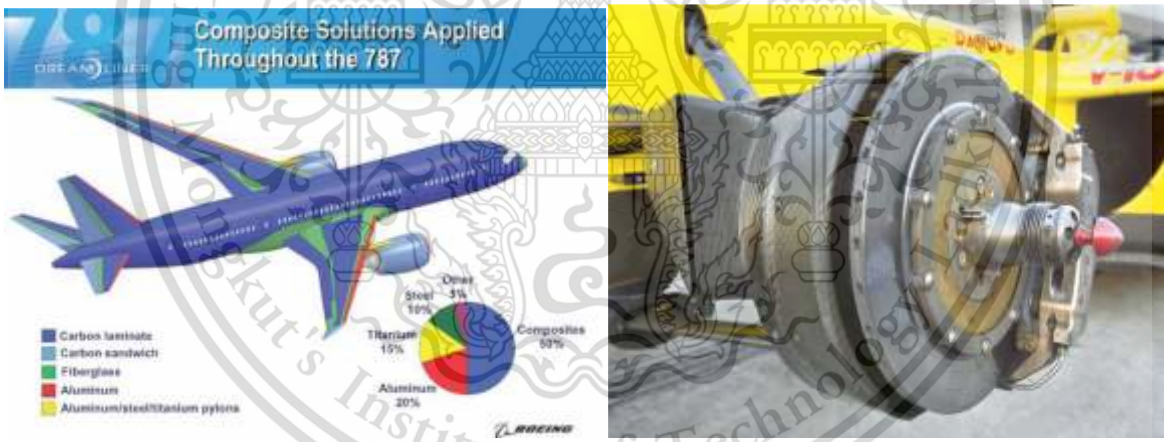


Figure 1.1 Boeing 787 Dreamliner commercial airplane and Formula1 brake disk made of carbon - carbon composites [3]

Several research of composite are conducted in various sections with several technical tools and equipment, and also still ongoing research due to its failure mechanism complexity. Failure mode investigation on a micro scale is the important part to analyze, but also the molecular level of the structural effect to the strength of the composite. Unlike alumina, the defects or dent on the composite cannot observe easily by human eyes, it need special tools to inspect defected area. The information of failure mode in carbon and glass fiber composite are still not sufficient to analyze in macro and micro scale. The molecular

level of carbon fiber is much depends on the manufacturing process. The previous research described the highly graphitic material give the highest strength in fibers itself and also in composite. Therefore, composition mechanical properties are much depend on nano structure of fiber. In this research, four types of carbon and glass fiber will examine in their micro and nano structure.

1.2 Research Objectives

1.1.1 Study about physical characteristic and chemical composition of carbon fiber and glass fiber in micro and nanostructure to the commercial materials

1.1.2 Investigate mechanical properties of different woven pattern of fabrics for both carbon fiber and glass fiber composite

1.1.3 Study the failure mechanism of carbon and glass fiber composite in micro structure

1.1.4 Observe the mechanical properties and failure mode of temperature treatment effect on the composite and treatment fiber composite.

1.3 Scope of Research

1.1.5 Explain Physicochemical Characteristics of carbon fiber and glass fiber in micro-and nanostructure both naked fiber and composite by using engineering and material science analysis tools such as Transmission Electron Microscopy (TEM), Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), Raman Spectroscopy (RS) and X-ray Diffraction (XRD).

1.1.6 Describe Mechanical properties of each fabric pattern and composite of carbon and glass fiber by using the mechanical properties analyzer such as tensile and flexural strength test.

1.1.7 Describe fracture mechanism of composite in microstructure by using image processing techniques through scanning electron microscope.

1.4 Research Methodology

1.1.8 Study physiochemical characteristic of carbon fiber and glass fiber both mechanical properties and physical analysis by using tensile machine, Raman Spectroscopy, X-Ray Diffraction (XRD), Fourier-Transform Infrared spectroscopy (FT-IR), field emission scanning electron microscopy with energy dispersive spectroscopy (FE-SEM/ EDS) and transmission electron microscope (TEM).

1.1.9 Fabricate composite by hand lay up process to investigate mechanical properties of carbon and glass fiber composite by using tensile and flexural mechanical testing machine. The failure mode of the composite are investigate by using image processing method electron microscopy.

1.1.10 Fabricated composite with different temperature post cured at room temperature (RT means 30ºC) until it is dry, and 80ºC for 6hrs after curing at room temperature and 120ºC for 3hrs after cured at room temperature, to investigate the effects of different temperature curing in mechanical properties and fracture mechanism. Thermo gravimetric analysis (TGA) was used to analyze the temperature effect on the composite and their weight reduction steps.

1.1.11 Fiber manufacturer use sizing agent to protect fiber surface from environment and for better handling purpose during manufacturing. This sizing agent layer from fiber surface is the main weakness to adhere fiber and matric. Therefore, treatment on fiber surface are perform by immersing fiber in acetone to release out the sizing material. Composite was fabricated by treated fiber with the previous hand lay up process after treatment fiber surface. Composite are test to observe the mechanical properties of treatment fiber composite and their fracture mechanism were investigated by electron microscopy.

1.1.12 Summary in this thesis

Chapter 1. Introduction, Objectives, Scope of research and Methodology.

Chapter 2. Discuss the previous research, theory, the basic of carbon fiber and glass fiber, composite's strength at different temperature curing.

Chapter 3. Introduce the materials and producing method of composite, the using tools to reveal the characteristic and the morphologies of fibers.

Chapter 4. Result and Discussion of fiber characteristic, mechanical properties, fracture mechanism of composite, treatment fiber, treatment fiber composite and their effect base on temperature curing effect.

Chapter 5. Conclusion and suggest

1.5 Expected Out

The fracture mechanism of carbon fiber and glass fiber composite will help to study about the effect of increment temperature curing and fiber treatment method for composite. The current research result will distribute in international academic conference such as The 9th International Conference on Mechanical Engineering (TSME-ICoME2018) and 20th Asia Pacific Automotive engineering conference (APAC-20).

CHAPTER 2
LITERATURE REVIEW

2.1 Carbon Fiber

Carbon fiber is the most useful fiber in composite which fiber is reinforce by matrix all epoxy especially for automotive field, vehicles and other component. The inner structure is also one of the most important character for composite properties because structure of fiber are the source of composite's mechanical properties. Carbon fiber is also call as graphite carbon.

2.1.1 Formation of Carbon Fiber and its Structure

Carbon fiber is one of the main important material in composite world not only in automotive field but also in marine, aerospace, aircraft, military vehicles, sport and etc. There are several type of carbon fiber such as pitch, aramid, PAN and mesopitch carbon fiber. Among them, precursor polyacrylonitrile (PAN) based carbon fiber is one of the most useful materials due to its high strength and young modulus compare to other materials. PAN formula is CH_2=CHCN and it was made from propylene and ammonia [1]. The structure of PAN carbon fiber is zig-zag form before the stabilization stage. Most of fibers are produced by wet or dry spinning method as shown in figure 2.1. There are several steps to form carbon fiber after spinning fiber filament such as stabilization, oxidation, carbonization and graphitization for high graphitic crystal plane in order to achieve high tensile modulus. After carbonization stage of the precursor, different crystal are form based on the manufacturing process, environment and temperature.

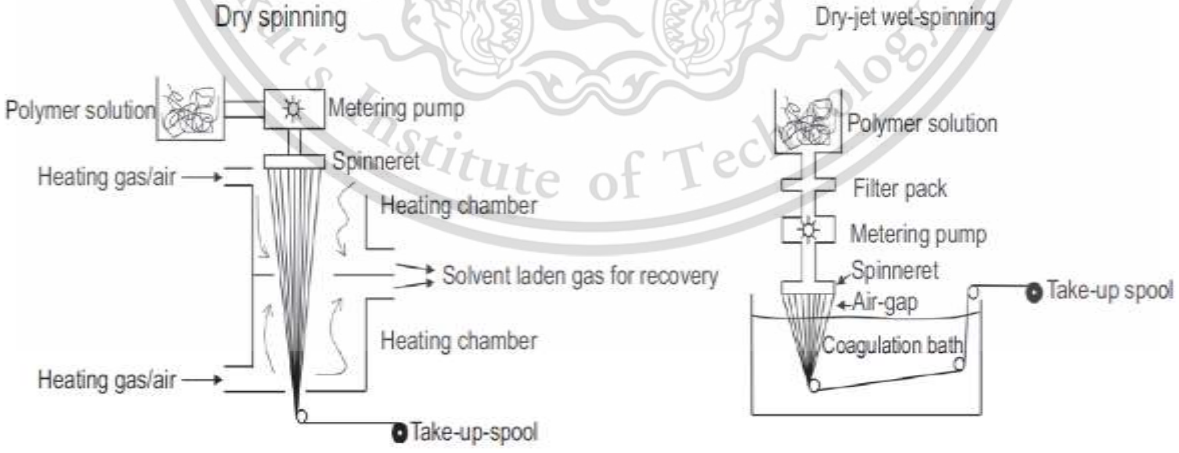


Figure 2.1 Schematic of the dry spinning and dry-jet wet-spinning methods [2]

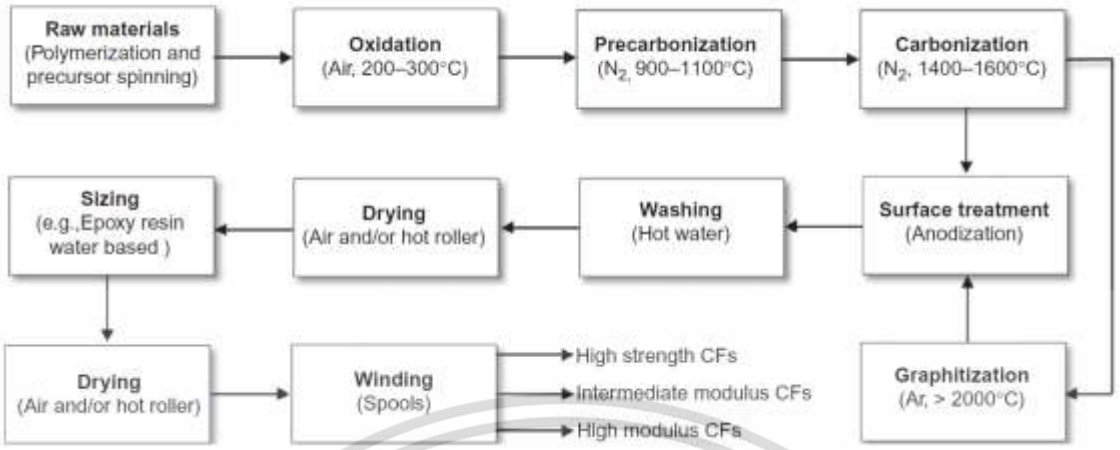


Figure 2.2 Diagram of CF manufacturing process from PAN-based precursors [5]

Polymerization of PAN homopolymer/monopolymer are used to make carbon fiber by spinning method and continues to make stabilization and carbonization by different environment and temperature. Carbon fiber contains over 92% of carbon in commercial carbon fiber. Traditional stabilization is oxidation fiber at 200-300ºC temperature to the fiber filament with air condition. The filament are carbonize at the pre stage with temperature 900ºC to 1100ºC in nitrogen. Carbonization process was performed at 1400-1600ºC in nitrogen flow as shown in figure 2.2. Increase heat treatment temperature on carbonization stage brings better graphite crystal structure. Commercial PAN based carbon fiber is carbonize at 1400-1600ºC temperature in nitrogen flow [2]. In order to get high modulus in mechanical properties of carbon fiber composite, the graphite carbon crystal structure is very important in fiber. The highly oriented pyrolytic graphite (HOPG) fiber structure gives the highest strength. In graphitization stage, higher temperature make the higher graphitic structure of carbon fiber filament. The final structure of carbon fiber becomes the graphite structure of crystal studied by R. Moreton [3]. Carbon to carbon hexagonal bonding are formed to be a graphene sheet and several graphene sheets were stacking parallel into the graphite structure. The non-equivalent carbon bonding structure and graphite crystal are mixed together along the fiber axis with various orientations. Carbon-carbon bonding distance is 0.1422nm for a graphite structure which can vary depends on the manufacturing process. Crystal size (L_a) and crystal stacking height (L_c) are large enough in highly graphitic structure which can give higher modulus in mechanical properties. Graphitization of fiber at 2500-3000 ºC make reducing in crystal growth [4]. Figure 2.3 shows the basic perfect graphite structure form of a carbonized filament of carbon fiber. The perfect graphitic structure are the crystallite with several graphene of carbon to carbon covalent hexagonal bond were stacking parallel. In carbon fiber, several graphitic crystallite are randomly oriented and the whole structure are not graphitic crystallite. It was the mixture of graphitic crystallite and defected atom which can take place in the edge of graphite crystallite as a turbostratic structure as shown in figure 2.4.

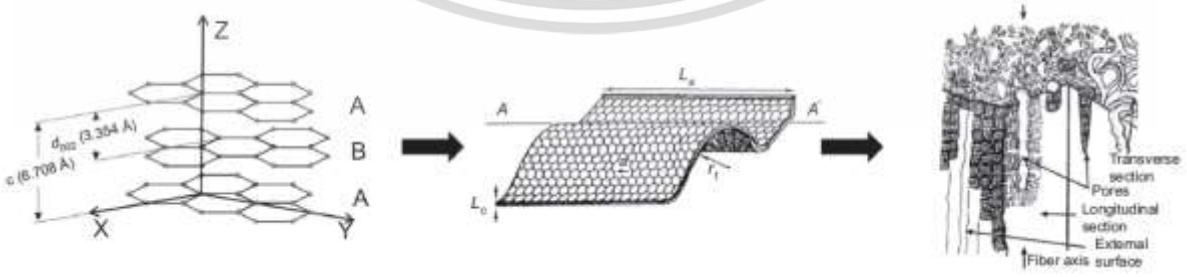


Figure 2.3 Carbon fiber basic structural unit as adapted from a perfect graphitic structure and its relation to the carbonized filament

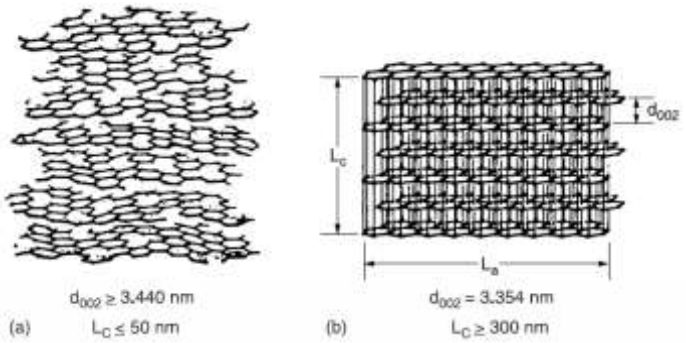


Figure 2.4 crystallite of a carbon material: (a) carbon turbostratic structure with (b) 3-D graphite lattice

2.1.2 Characteristic of Carbon Fiber

There are several tools that can reveal the structure of carbon fiber and morphology of carbon fiber such as x-ray diffraction, Raman spectroscopy and transmission electron microscopy in nano scale. XRD indicate the oriented plane of graphite from fiber and represent in the graph through intensity and 2θ angle. The carbonization fiber will give different peak results in XRD especially when it has exceeded temperature 2000ºC. Figure 2.5 shows XRD spectra of carbon nano fiber and carbon fiber intensity peak at 2800ºC [6]. From XRD result, crystal size, fringe length and interlayer spacing can be calculated by Scherrer's equation and Bragg's law. Mechanical properties of composite are highly effected by crystalline size and length [7]. Raman spectroscopy is one of the important tools that can indicate the structure of fiber such as amorphous by disorder (D) band which indicate the disorder atom from the edge of the crystalline lattice and crystalline by graphite (G) band which is from the high oriented graphite and. Most of carbon fiber is constructed with many of graphite structure, crystalline and amorphous structure of disorder atom. Figure 2.6 explains details about the morphology of fiber structure and different intensity peak. The common graphite (G) peak show at 1590cm^{-1} Raman shift and disorder (D) peak at 1380cm^{-1} in most of the previous research. The structure of the graphite crystalline and the disorder band was shown in figure 2.4. G band indicated well order of hexagonal bonding of carbon with graphite crystalline structure while D band associate with poorly graphite edge and the intensity is lower than the graphite intensity peak [27]

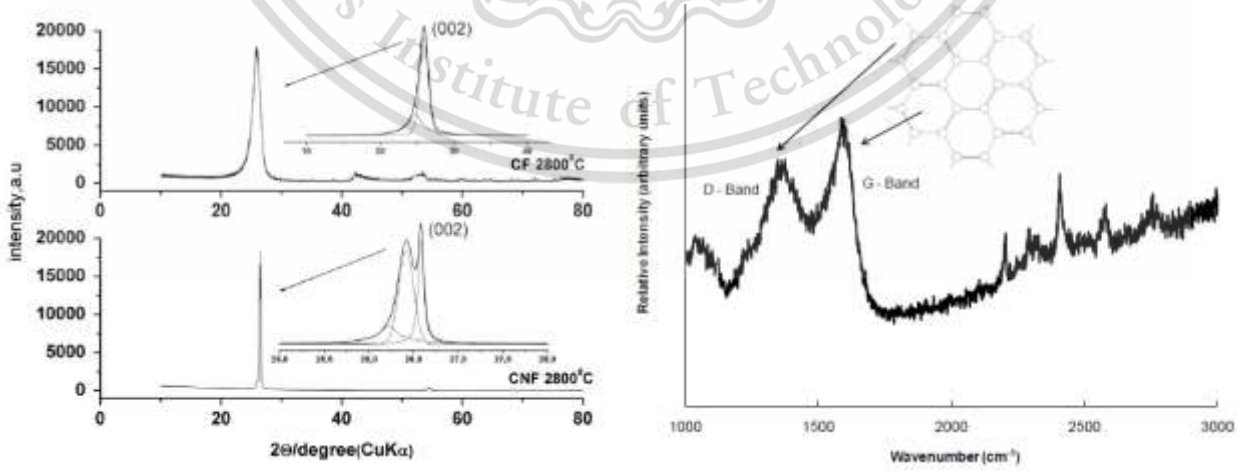


Figure 2.5 X-ray diffraction spectra of CNF and CF obtained at 2800 °C [6] and Raman Shift of carbon fiber crystallite [27]

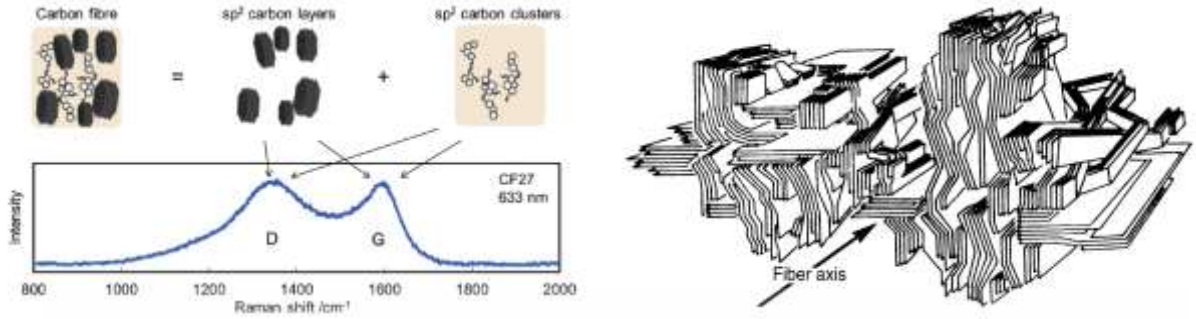


Figure 2.6 Peak assignments of the Raman spectra based on the plausible nanostructure of PAN-based carbon fibers [8] and three dimensional structure of the HM carbon fiber depicting interlinked crystallinity

The whole fiber cross section depiction was shown in figure 2.6 right side, several fringe are laid randomly with several orientations in fiber filament. It can not consider every fiber were the same, it may vary based on the manufacturer, the properties and the morphology as per customer needed. Heat treatment on the fiber filament bundle will give different structure and properties as shown in figure 2.7. Low temperature heat treatment gives low crystallinity, and high temperature heat treatment gives high crystallinity as show in the figure 2.7. Several graphite crystal are oriented with the same direction on the surface of fiber parallel to the fiber axis, it can make the weakness of interfacial bonding between fiber and matrix in composite. The whole fiber appearance was shown in figure 2.7, it was the structure of commercial fiber.

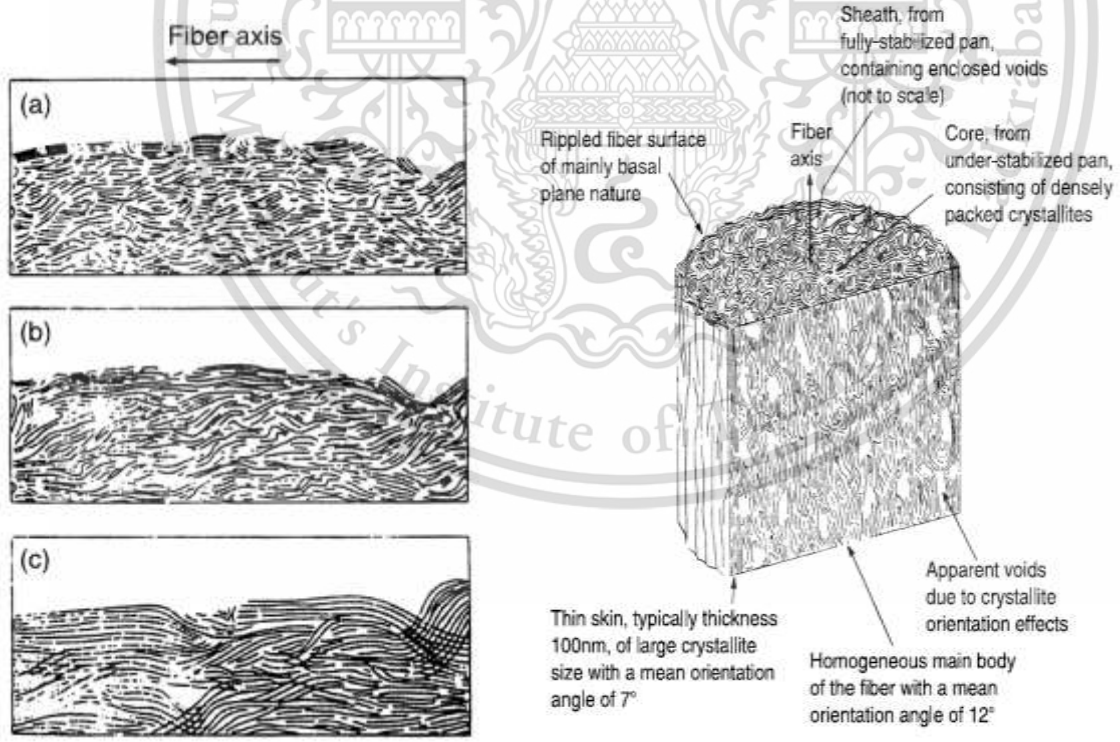


Figure 2.7 Schematic representation of the development of a layer-plane structure of TEM studies. (a) 1000ºC; (b) 1500ºC and (c) 2500ºC and a proposed three dimensional structure of a PAN based HM carbon fiber

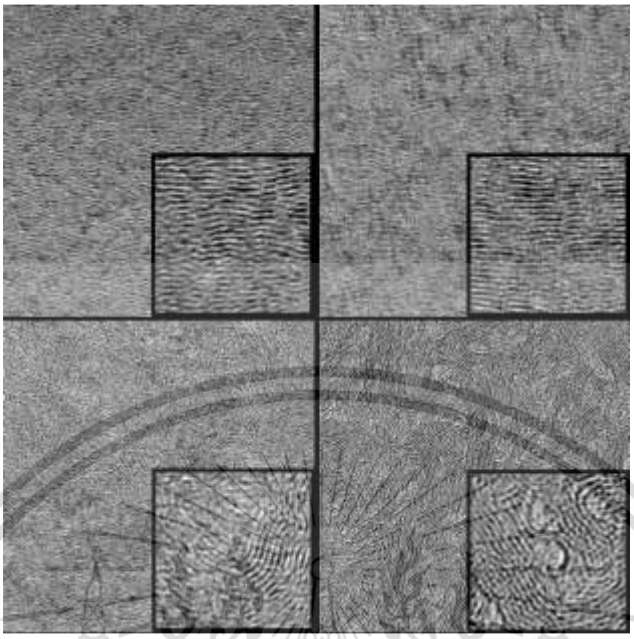


Figure 2.8 HRTEM images of various raw carbons [46]

TEM image of carbon fiber are shown in figure 2.8. Most of the commercial fiber available in the market are the carbonization carbon fiber at temperature 1000-1500ºC which was constructed in the mixing of turbostratic structure and well order graphite structure. Several bundle with different filament quantity are produced with many type of woven pattern such as 0/90, +/-45, twill, plain and etc. Different filament quantity of bundle are also available in the market based on the needed of vendor. After the fibers were treated and sizing, the various of pattern like twill weave, tow or plain weave pattern were woven as customer desire which are available in the market as shown in figure 2.9.

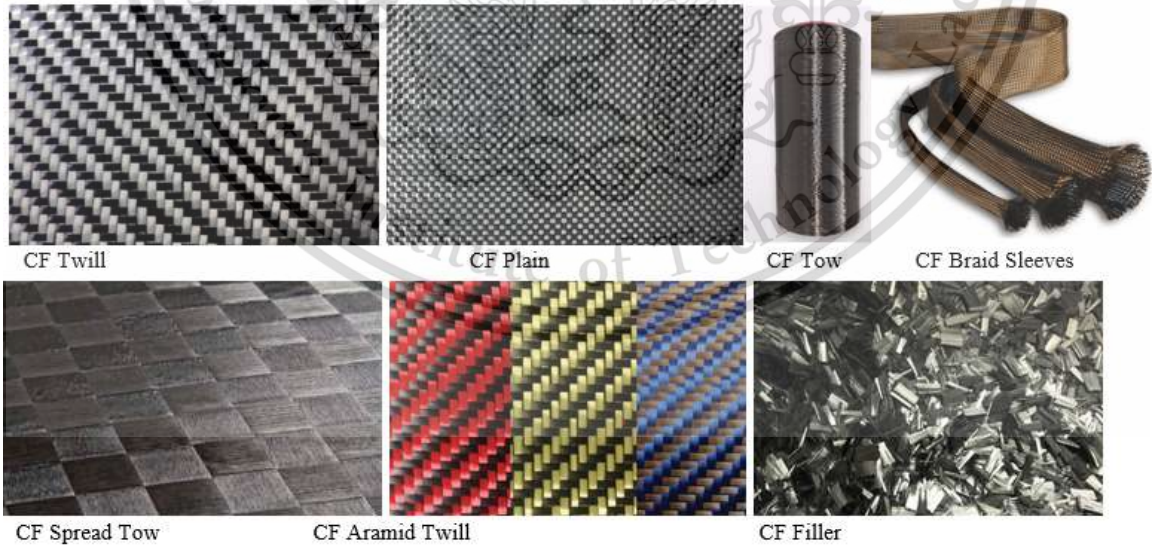


Figure 2.9 Various woven pattern of carbon fiber

2.2 Glass Fiber

Glass fiber is one of the most versatile material that can use in various fields such as automotive field, sporty, furniture, heat exchanger, boat null /seat and fishing rod. Nowadays, glass fiber and carbon fiber composite are very useful in several fields. Most of glass fiber are based on silica (SiO_2) and it contain around 50-60%. Additionally, it was combine with another chemical such as oxides of calcium, boron, sodium, aluminum, iron and etc. Glass is mainly come from sand and it is the source of silica [12] and it need a good care to get free of impurities material to process. The structure of silica glass are shown in figure 2.10, two types of crystalline structure with long range and silica is absent of long range bonding. Particularly, long range bonding with crystalline are close to that of a liquid structure. Silica glass structure is common structure and the bonding range between Si-O and O-O are about 0.162 and 0.265nm respectively [10].

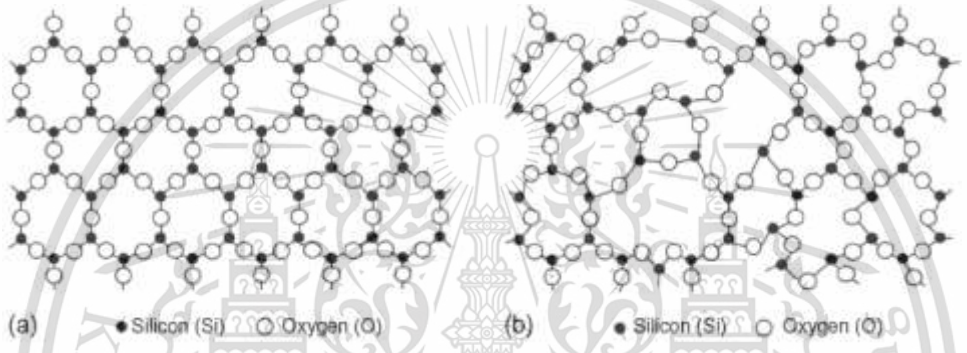


Figure 2.10 Schematic two-dimensional representation of (a) the structure of crystalline silica (quartz) and (b) the structure of silica glass. [11]

There are many types glass fiber to use in different respective field such as E glass, S glass, C glass and so on as shown in figure 2.11. E glass stands for electric insulation beside good strength and low density comparison to other, S stands for high silica content to withstand higher temperatures and C stands for corrosion as it have a better chemical resistance. Among them, E glass fiber is the most useful material as a commercial with continuous glass fiber [11]. There are many methods to fabricate the glass fiber, such as melt-quenching, sol-gel, chemical vapor deposition and sputtering. Glass fiber is noncrystalline but it is an amorphous solid structure and it was the mixture of oxides. Therefore, it can not burn easily in oxygen or air, but they can be soft or melt at 850-900ºC high temperature [12].

Oxide	*A-glass (%)*	*C-glass (%)*	*D-glass (%)*	*E-glass (%)*	*ECR-glass (%)*	*AR-glass (%)*	*R-glass (%)*	*S-2-glass (%)*	*No boron E-glass (%)*
SiO_2	63–72	64–68	72–75	52–56	54–62	55–75	55–65	64–66	52–62
Al_2O_3	0–6	3–5	0–1	12–16	9–15	0–5	15–30	24–25	12–16
B_2O_3	0–6	4–6	21–24	5–10		0–8			
CaO	6–10	11–15	0–1	16–25	17–25	1–10	9–25	0–0.1	16–25
MgO	0–4	2–4		0–5	0–4		3–8	9.5–10	0–5
ZnO					2–5				
BaO		0–1							
Li_2O						0–1.5			
$Na_2O + K_2O$	14–16	7–10	0–4	0–2	0–2	11–21	0–1	0–0.2	0–2
TiO_2	0–06			0–1.5	0–4	0–12			0–1.5
ZrO_2						1–18			
Fe_2O_3	0–0.5	0–0.8	0–0.3	0–0.8	0–0.8	0–5		0–0.1	0–0.8
F_2	0–0.4			0–1		0–5	0–0.3		0–1.0

Figure 2.11 Composition ranges for the oxides used in specific commercial glass fibers [11]

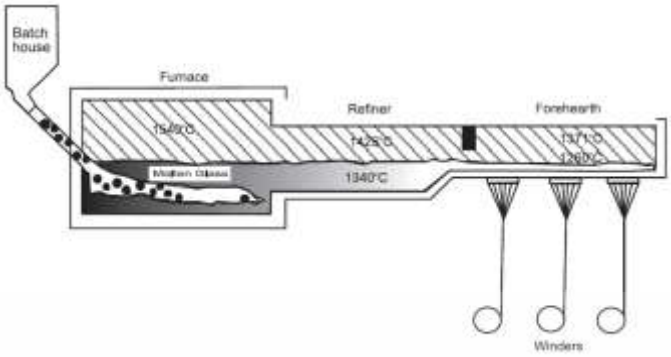


Figure 2.12 Simple schematic diagram of a larch glass fiber melting and drawing operation [12]

After the glass fiber filaments are produced, sizing process on the fiber surface is follow to protect the fiber surface or to prevent handling problems of glass fiber surface. Glass fiber bundle was pass through coupling agent or sizing solution for sizing the surface and the thickness of sizing is varying from 10nm to 1μm in each fiber filament with around 0.2-2% by weight as shown in figure 2.13. The main fiber are continuous fiber and also available filler or short fiber for injection. The fiber pattern are design in different woven, pattern and different fiber orientation as needed. Some fiber was made a filament winding as the vendor desire especially for tube or hollow shaft. Generally, there are several woven fabrics in the market for continuous glass fiber with different filament quantity in one bundle.

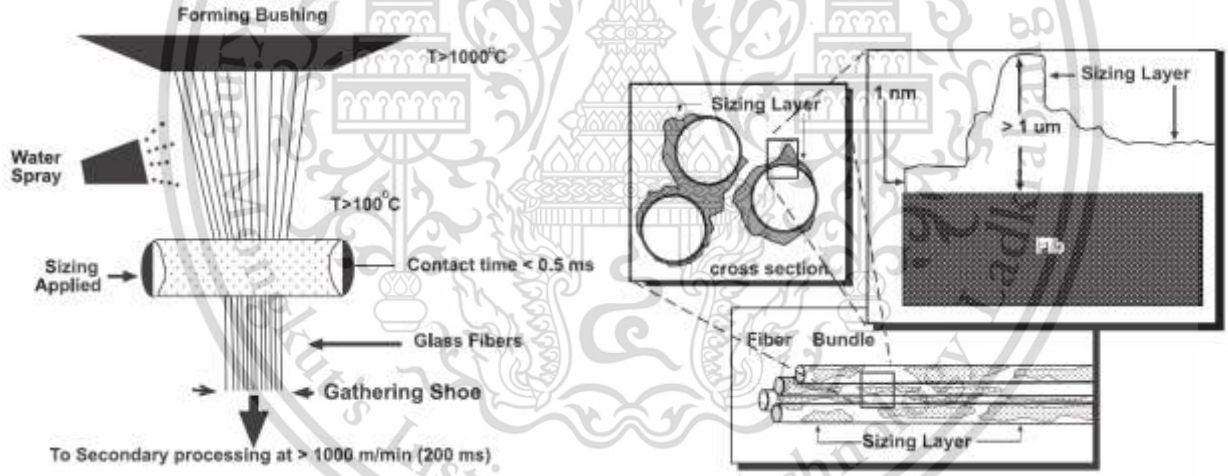


Figure 2.13 schematic of sizing on fiber and sizing layers of commercial glass fiber [12]

2.3 Matrix Epoxy

For matrix of composite, there are several matrix epoxy which are used not only in the composite field, but also in structural and construction field. In composite, there are two types of matrix such as thermoplastic and thermosetting epoxy resin. In the case of composite of carbon and glass fiber, thermoset resin is one of the most used matric to fabricate the composite in various method. Epoxies are made from epichlorhydrin (ECH) and diphenylolpropane (DPP), also known as Bisphenol-A (BPA) and the chemical formula are as shown in figure 2.14. Most of epoxide are used in bonding, adhesive, electric field and composite to enhance mechanical properties as reinforce properties.

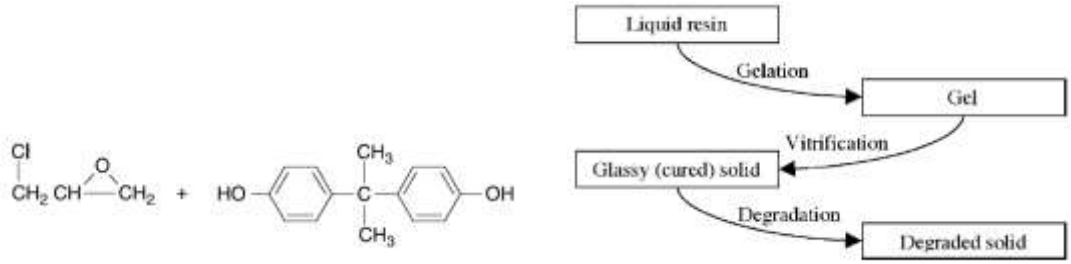


Figure 2.14 chemical formula of Bisphenol-A epoxy and the cured process of epoxy

There are many types of epoxy that can cure at room temperature and high temperature. The moment of mixing epoxy resin with hardener, epoxy becomes Crosslink at room temperature and gel condition with the limited time. Full cured is at solid stage and the epoxy is brittle when it turn to solid state. The highest mechanical properties, chemical reaction are achieved at full cured solid stage when the needed temperature and environment are obtained. Figure 2.14 shows the curing process of epoxy with the first stage from liquid to gel stage until it becomes solid glassy when it is fully cured. The material can degrade when there is an extra temperature of curing more than the needed temperature. For composite of carbon and glass fiber, there are several cured temperature and gel time depends on the hardener. For this current research, EPOTEC YD 582 Bisphenol-A type is use as epoxy and EPOTEC TH 7257 from concrete composite.

2.4 Treatment Fiber

Treatment fiber is one of the process to make pitted valley surface from smooth surfaces after the spinning fiber in manufacturing process. Coupling agent or sizing solution were used to make sizing around 0.5-6% of carbon fiber and 0.2-2% for glass fiber by weight on the surface of fiber to prevent the fiber surface while handling of the bundle of fiber. But it can block out all of the treated surface by sizing material that could make the weakness in the bonding of fiber and epoxy matrix. The treated surface have several pores and high roughness on surface that can interlock easily with matrix. But treated fiber might be one of the obstacle for handling in manufacturing process. Therefore, the manufacturer always used sizing on the surface to prevent the surface damage and to handle easily. Some of researcher also does desizing or the removal of sizing material from the fiber surface, it may reveal the original treated surface. Different solution treatment is used in different research give a different result from composite. The changes of sizing material and unsized material properties can measure by Atomic Force Microscope (AFM) through small spindle of the AFM's nose. Acetone was used to extract the sizing material from the fiber surface at 80ºC for 24hrs. The removal of sizing prove that there are effects on the surface of fiber and the sizing content was changed with heat treatment [14]. The surface morphology are change to enhance interaction of fiber and epoxy matrix as shown in figure 2.15 based on AFM analysis. The removal sizing carbon fiber shows better wetting with epoxy resin than can enhance mechanical properties in composite. The weakness or strong adhesive in fiber and matrix are much depends on the sizing agent and process of sizing. Different sizing agent alters the fiber surface morphology different as shown in table 1. HNO3 treated is used to treat several carbon fiber with different timing give different surface morphology as shown in figure 2.15. The roughness of surface is one on the important mechanism for composite and surface treatment of carbon fiber is essential needed to improve the adhesive of fibers and matrices. It also influence fiber chemical structure to enhance interfacial bonding of fiber and matrix [16]. Some of chemical group also observes by FTIR, those groups are responsible for chemical reaction with a matrix and wettability of fiber.

Table 1. Surface roughness of unsized and sized carbon fibers

	Unsized	Ultem® sized	PTPO sized
Root mean square roughness, RMS	9.1 nm	11.0 nm	18.0 nm
Mean roughness, Ra	7.5 nm	9.0 nm	15.0 nm
Maximum height, R_{max}	49.0 nm	58.0 nm	86.0 nm
Dimensions	1 μm×1 μm	1 μm×1 μm	1 μm×1 μm

The desizing or etching to sizing fiber surface layer is one of the effective way for composite especially in flexural strength. Flexural strength of composite increase 20% by etching the sizing layer of fiber and increase 7% by desizing respectively because fiber and matrix interfacial bonding are stronger after desizing or etching. The flexural strength result was shown in figure 2.16. Three types of sizing material were used in fiber and produce composite to observe mechanical properties through flexural test and toughness impact test. As the result, all types of sizing fiber show reducing the mechanical impact toughness and not significant improvement on the composite compare to unsized fiber composite [35]. Therefore, unsized fiber show the better mechanical properties somehow in composite.

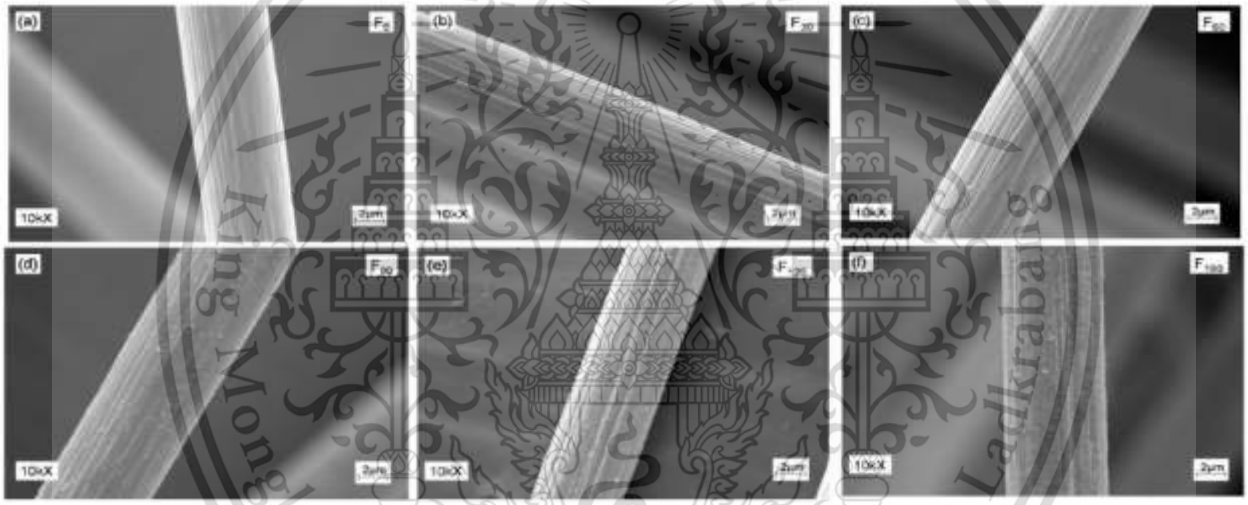


Figure 2.15 SEM micrograph of untreated and HNO3 treated CF; (a) untreated (b) 30 (c) 60 (d) 90 (e) 120 (f) 180 mints treated CF's

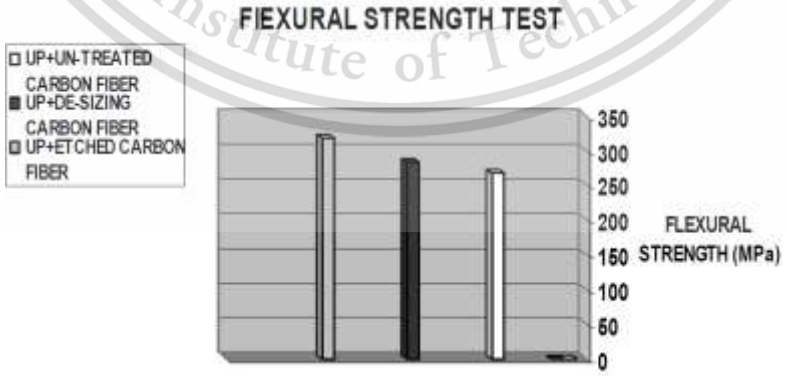


Figure 2.16 Flexural strength of different treated carbon fiber composite [17]

2.5 Carbon Fiber / Glass Fiber and their Composite

2.5.1 Composite of Carbon and Glass Fiber

Carbon and glass fiber is the main usage fiber in composite field for automotive and other several fields as mentioned above. There are the main two materials that were used in composite first is fiber reinforcement and another one is matrix epoxy to reinforce the fiber in the composite. For Fibers, various fibers are available in the market based on different strength and different fabrication process. Generally, there are three types of fiber for composite field, those are carbon fiber or graphite fiber, glass fiber and aramid (Kevlar) [19]. Fibers are the most important component of the composite. The main responsible for fiber are to contribute strength and stiffness of composite and to carry the load from external in composite. Matrix resin also one of the most important to reinforce fiber in the composite. Several fabrication methods are found in these days to make a batter composite with the simplest and the cheapest composite manufacturing process such as

1. Hand Lay up (Wet lay up) process
2. Vacuum Bag Molding/ Autoclave
3. Pultrusion
4. Compression Molding
5. Filament Winding
6. Resin Transfer Molding
7. Vacuum assistant resin infiltration
8. Injection molding and etc.

Several papers show different method of composite fabrication process. For composite, there are many factors such as parametric, temperature, pressure and environment to consider in the fabrication process besides fiber and epoxy. In composite, the main effect is fiber orientation not only in carbon fiber, but also in glass fiber due to the geometry of fibers are stronger in its axis and also depend on applied load direction. Figure 2.17 shows the orientation of fiber with different angles. Different orientation of fiber also gives different result in composite. Mostly 0/90º woven pattern of fibers are common usage with different fiber filament quantity.

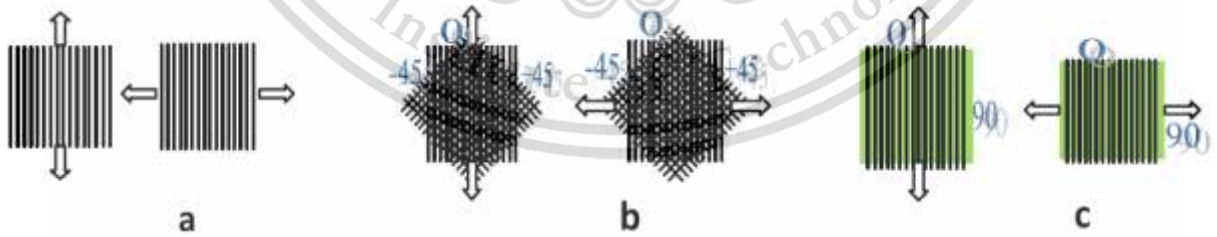


Figure 2.17 Stacking sequence and tensile loading direction of composite; a) 0/0/0/0, b) 0/+45°/-45°/0 and c) 0/90°/90°/0 [20]

Unidirectional 0º oriented fiber of a composite always obtain the highest mechanical strength especially in longitudinal axis in many literatures. Jute epoxy composite with glass fiber is fabricated by vacuum assisted resin infiltration (VARI) techniques with stacking sequences of (0/0/0/0), 0/+45°/-45°/0 and 0/90°/90°/0 [20]. It shows different result in tensile properties. The same fabrication technique are used to produce composite and test in several tests to the composite of different oriented fabric composite.

2.5.2 Mechanical Properties of Carbon and Glass fiber

The composite are need to produce based on user's usage need for all strength, properties, quality and morphology. Figure 2.18 shows a different mechanical test of different orientation laminated fiber to give different results of composite. The specimens were tested in a different mechanical test and the result are different based on different tested condition accordingly [21]. 0, 90° and 0/90° fabric are laminated differently to produce a composite of both carbon and glass fiber by hand lay up process and the composite are tested in tensile and flexural test. As a result, 0 laminated composites have the highest tensile strength in both CFRP and GFRP [18]. Therefore, 0 degree oriented fibers have the highest strength when the tension are test in the longitudinal axis of the fiber. CFRP result is higher compared to GFRP except 90degree orientation, fiber in flexural test of composite.

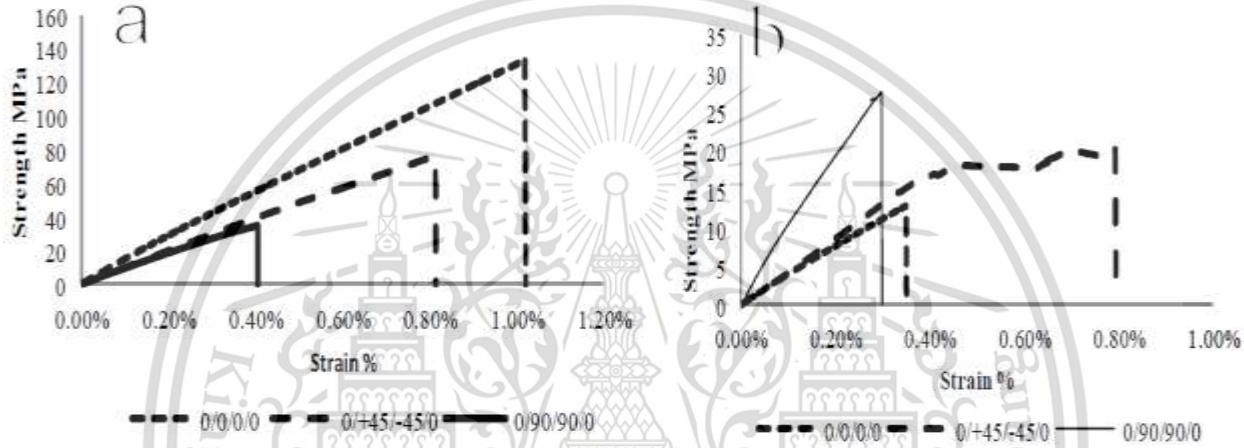
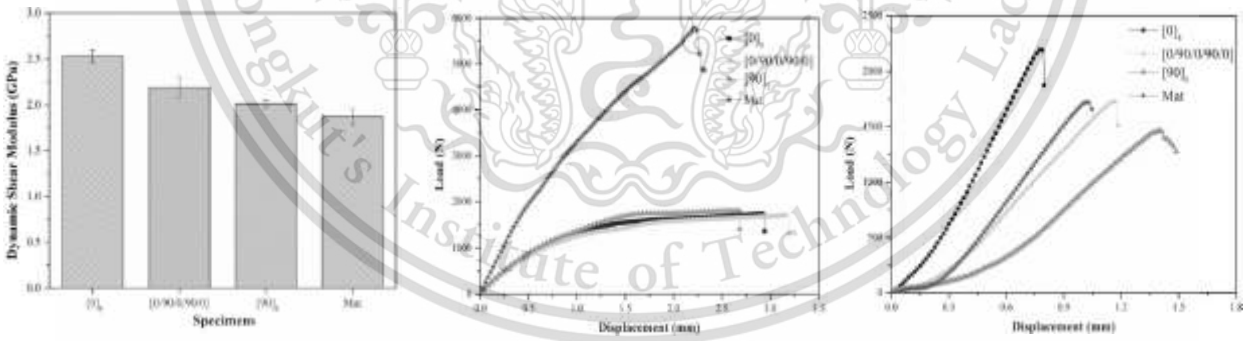


Figure 2.18 Tensile test result of jute epoxy composite with different orientation fiber a) longitudinal and b) transverse direction [20]



(a) Dynamic shear modulus (b) V-notched shear specimens (c) Double Notch Shear specimens.

Figure 2.19. Load and displacement curve of a composite of (a) Dynamic shear modulus, (b) V-notched shear specimen test and (c) Double Notch shear test [21]

There are several composite that are fabricated by hybrid of carbon fiber and glass fiber with matrix epoxy, and also carbon fiber and aluminum hybrid composite. Jaganatha and Harish [23] studied about hybrid of carbon and glass fiber composite with different percentage 15%, 30%, 45%, 40% and 60%. All of them are fabricated using vacuum bag process. And found that 60% of pure carbon fiber and 0% of glass fiber have the highest mechanical strength, such as ductility, load and ultimate tensile strength. Hybrid composite of carbon and glass fiber were also studied by Turla [28] especially in flexural mechanical test

to composite of filament winding fabrication technique. The result of hybrid composite with carbon and glass fiber is slightly higher than pure carbon and glass fiber composite. The result are shown in figure 2.20 of hybrid composite and all results of composite. Surprisingly, carbon fiber composite was the lowest flexural strength in the chart.

Specimen #	Maximum Load (N)	σ_f (MPa)
1	893	570.00
2	857	547.02
3	878	560.43
4	804	513.19
5	821	524.04
Average	850.6	542.94
Standard Deviation	37.54	23.96

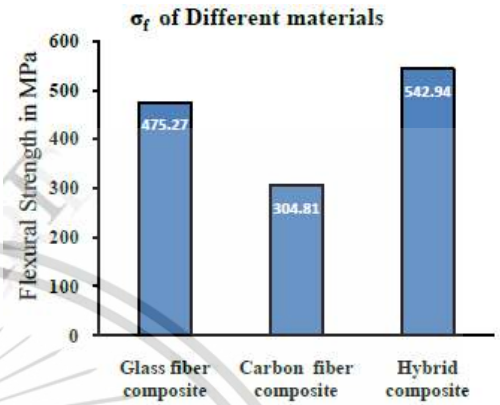


Figure 2.20 Flexural result of hybrid composite in table and comparison chart of all composite.

The mechanical properties in hardness and modulus of the composite can obtain by nanoindentation test on the composite test. This test can be done with small pyramid shape spindle diamond knife to investigate hardness and modulus of composite. Nanocomposite are used to analyze of their hardness and modulus of composite [29] with equation 1-5 with the principle of the indentation test of composite. Nano carbon fiber composite test condition are shown in figure 2.21, it describe the indenter contacting point with shape and diameter of indenter. The behavior of indentation in nano composite are also shown in figure 2.21 with the depth displacement of carbon fiber composite.

$$H = \frac{P}{A_c} \qquad (1)$$

Where H is defined as hardness of contact pressure under the indenter, P is the load and Ac is contact area.

$$S = \frac{dP}{dH} = \frac{2\,E_r\sqrt{A_r}}{\sqrt{\pi}} \qquad (2)$$

Where S is the initial slope of unloading curve, Er is the reduce modulus.

$$A = Fh_c = 24.56{h_c}^2 \qquad (3)$$

Where h_c is contact depth base on Sneddon's expression and its indentation.

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{\frac{dP}{dh}} \qquad (4)$$

h_{max} and P_{max} are the maximum of depth and load, ε is 0.75 for paraboloid revolution.

$$\frac{1}{E_r} = \frac{1-v_s^2}{E_s} - \frac{1-v_i^2}{E_i} \quad (5)$$

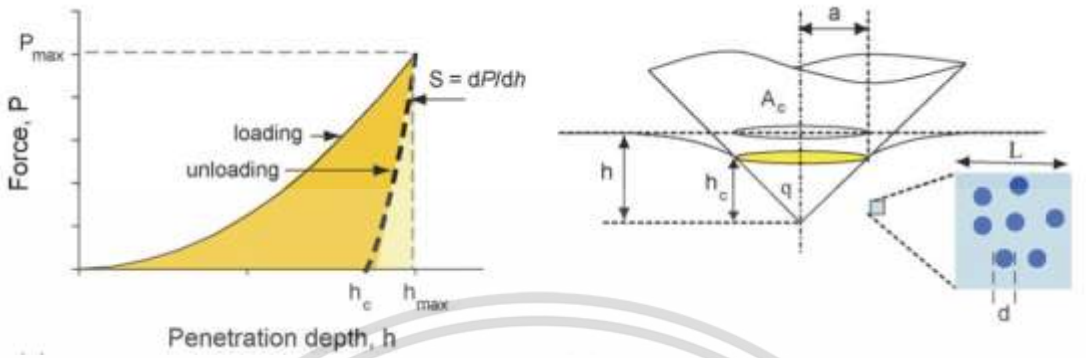


Figure 2.21 Principles of indentation testing. [30]

The equation are constructed with E_r is reduced modulus, E_s is specimen modulus, E_i is indenter modulus, v_s and v_i are Poisson's ratios of specimens and indenter respectively. Single carbon nanofiber was composed with epoxy matrix and tested in indentation to investigate the mechanical properties and the fracture behavior of nanocomposite in micro scale [31]. The behaviors of composite can easily obtain by this indentation testing method. Figure 2.23 shows the behavior of composite test result from nano composite of carbon fiber with its indenter points.

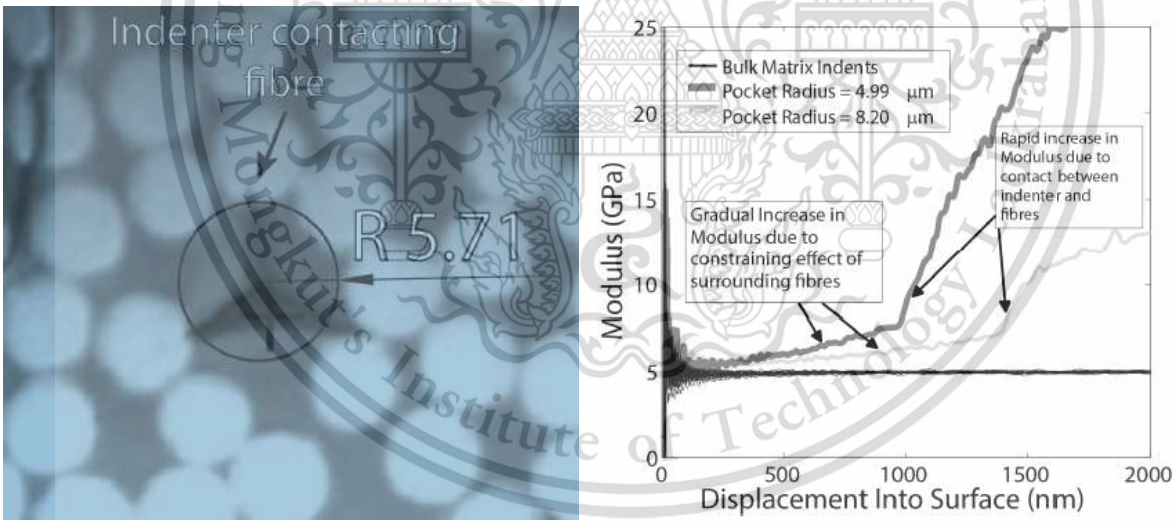


Figure 2.22 Optical Microscope image of indenter dent and Modulus Vs depth data for indentation behavior [29]

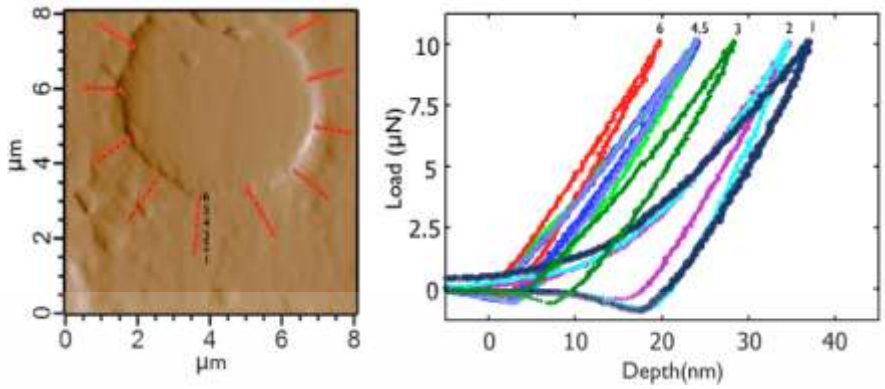
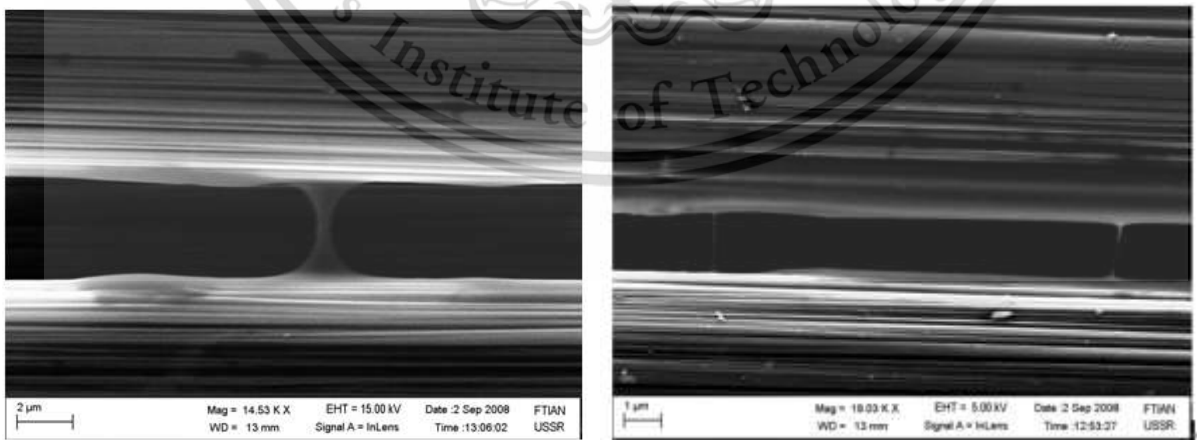


Figure 2.23 AFM image of the sample and indents locations and load and unload curves for indents at various radial distances of fiber composite [31]

2.5.3 Fracture Mechanism of Carbon and Glass Fiber Composite

Fracture mechanism is the most important part composite because knowing the weakness of the composite can give us how to prevent it. Fracture mechanism can be used to correct to the right way in the fabrication process for better mechanical properties. In composite, the interfacial bonding between fiber and matrix epoxy are very important to enhance mechanical properties. If fibers and matrix are well bonded both in mechanical interlocking and chemical bonding, it will bring the better mechanical properties. Therefore, the fiber surface is very important to adhere with matrix epoxy. Baurova [25] studied about two type of carbon fiber from different stage of carbonization and graphitization stage of fiber to make composite. Both of fibers are almost similar but the interior of fiber are different in structure. Carbonization fiber is constructed with crystallites and it cluster, therefore the graphite structure are connected with a cluster of defects crystalline while graphitization fiber structure is full of graphite crystalline. The curing temperature is different in two types of fiber at 1000-1500ºC temperature for carbonization while graphitization performed at 2500-2800ºC temperature. Morphology of cross section area and surface character between carbonize fiber and graphite fiber are as shown in figure 2.22 and 2.23. Carbonized fiber have the impurities around fiber, it wasn't removed completely from particles of organic polymer. It gives big effect to electrochemical properties of carbon fiber. On the other hand, graphitized carbon fiber have only a few of impurities, therefore it gives more stabilization of electrochemical properties in carbon fiber.



(a) Graphitized fiber (b) Carbonized Fiber

Figure 2.24 Appearance of a mono thread graphitized (a) Carbonized; (b) Fiber.

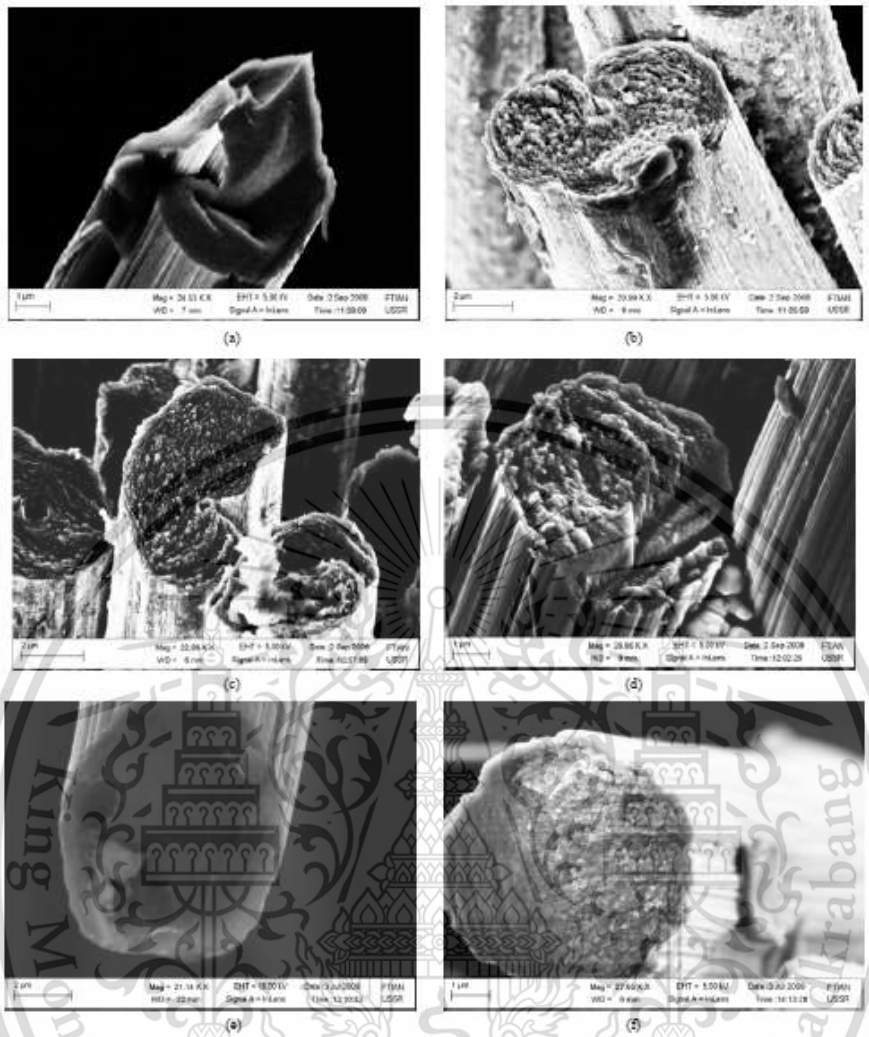


Figure 2.25 Structure of a mono thread of carbon fiber: (a) Elur; (b), (c) Culon; (d) LU; (e) UKN-P; (f) UKN-2-500 [25]

Another study of composite was conducted by Rahmani [24] different orientation of fiber at 0, 35, 45 and 90 oriented fibers were fabricated by using hand lay up process with three different types of epoxy. The ratio of epoxy to fiber is 60:40. The mechanical test of tensile, flexural and impact strengths were tested. The fracture mechanism of carbon fiber composite is shown in figure 2.26. Their mechanical properties are quite high compare to others result, and the fracture mechanism are also clearly shown that there is no pull out or separation of fiber from the matrix. Strong adhesion is observed at the interface of fiber and epoxy resin for their bonding. Polymer between fiber and matrix are very strong that provide good mechanical properties of the composite. The main important part of composite is fiber surface and fabrication process to follow the instruction of material curing with the right environment. The nanoparticles of rubber and nanoparticles silica was used to form a hybrid toughness composite by a resin infusion process in composite [33]. The result of mixing component shows significant increase in the interlaminar of fracture energy in mechanical test mode I and II.

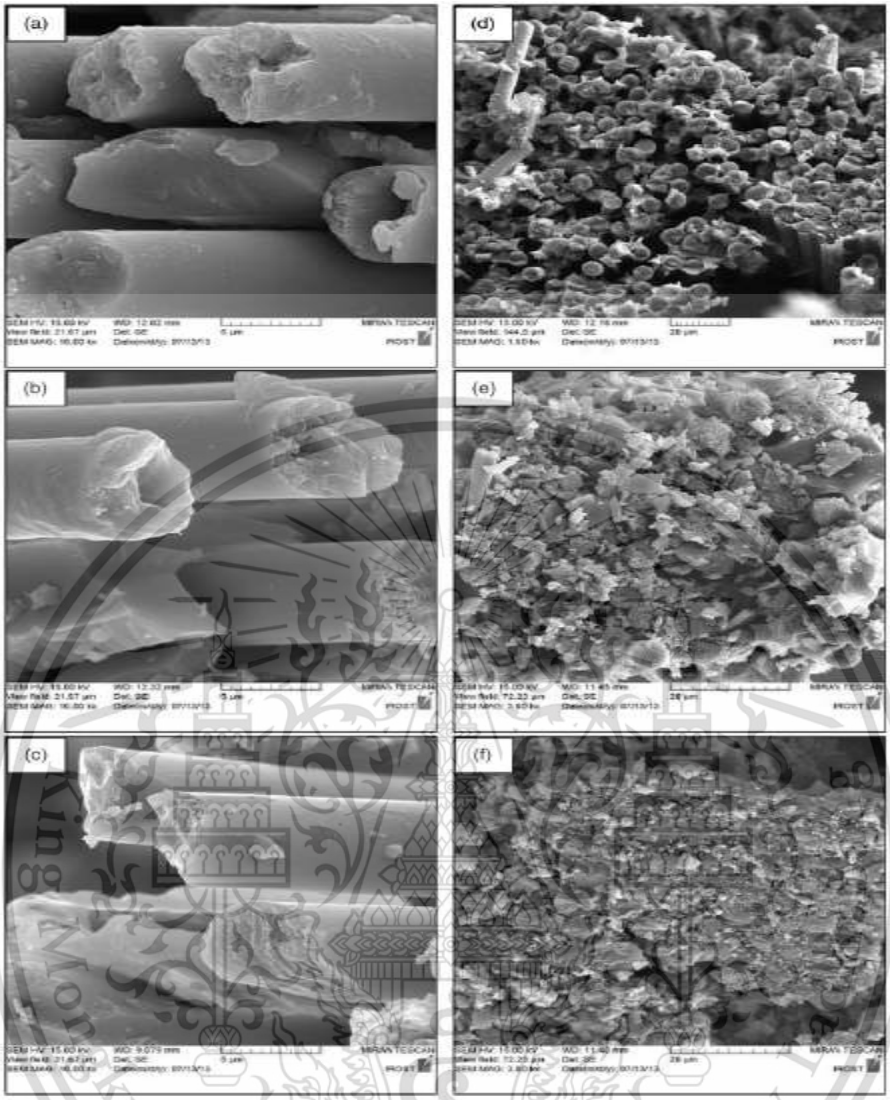


Figure 2.26 SEM micrographs of tensile fracture surfaces of the composite types of LY5/4 (a and d), EM5/4 (b and e), and EP5/4 (c and f) [24]

2.6 Temperature Effect to Composite

In the composite, there are several effects of various temperature curing to composite in different mechanical test result. Composite without treated fiber are fabricate to test mechanical properties and found the improvement strength of composite. Takamura [37] studied about tow types jute fiber for unsaturated fibers and other fiber that was immerse in the solution of astringent persimmon until it absorb and dry in oven for five hours. And the fiber was cured at different temperatures from 80-140ºC for 0.25h, 0.5h, 0.75h and 1hr before molding the composite. The composite are fabricate to test in a static tensile test and found that surface treated fiber got higher mechanical tensile strength. The highest mechanical properties was achieve from 0.5h cured composite. Heat treatment composite influence on the flexural strength of the composite and the hardness is not change significantly by heat treatment [36]. Glass fiber composite with

thermosetting polymers are curing at different temperature for various times to observe the impact of temperature post curing. The composite was fabricated by hand lay up process and cured at 60ºC temperature hydraulic press with 1MPa pressure for 20 Mins. The dries composite was post cure again at 80ºC, 110ºC and 140ºC for various hours. Interlaminar shear strength (ILSS) and Tg was evaluated using differential scanning calorimetry (DSC) as shown in figure 2.27. As the result, both of them are affected by post curing. Among several post curing temperature with varying time, 140ºC for 6hrs was the optimum value in composite [39].

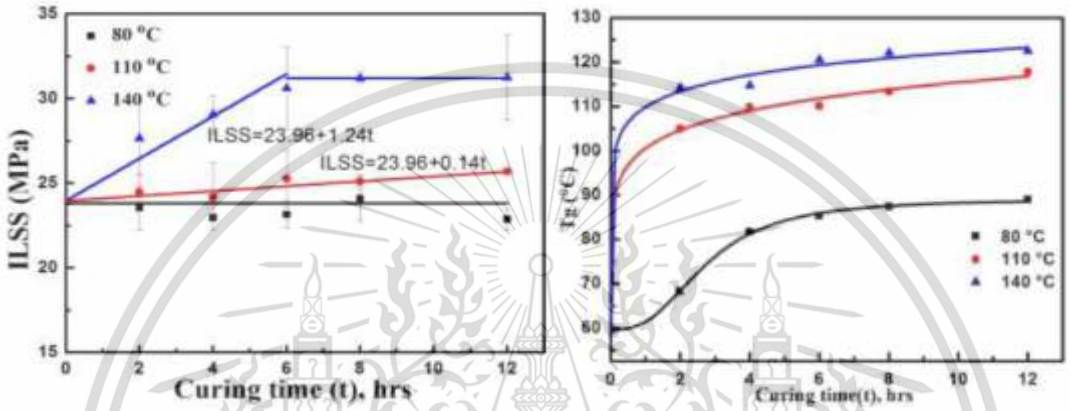


Figure 2.27 Effect of temperature and time on ILSS and Variation in Tg with curing at different Temperature [39]

Rudd [40] studied about glass fiber composite with novolac matrix which is produced by compression molding. The fiber are desized by dipping in ethanol to remove the sizing layer of fiber and then cured at 160ºC for one hour. The dry composite was post cured again at different temperature for 2hrs each temperature to observe the mechanical properties effect on tensile, flexural and impact test. The result shows that flexural and tensile properties were drop base on temperature effect while impact test result are improved. Aruniit [41] studied the composite that was fabricated by filler with special vacuum assisted casting machine to get rid of porous due to vacuum release air out from the mold and it was cured at room temperature for 12hrs. The composite was followed by further post cured at different temperature and time to increase cross link condition to obtain better mechanical properties at elevated temperature. As a result, elevated curing temperature increase heat resistance and cross link density. On the other hand, mechanical properties ascending with the increase of post cure temperature. Therefore, the result suggest that brittleness and the impact strength was need to be considered in composite post curing.

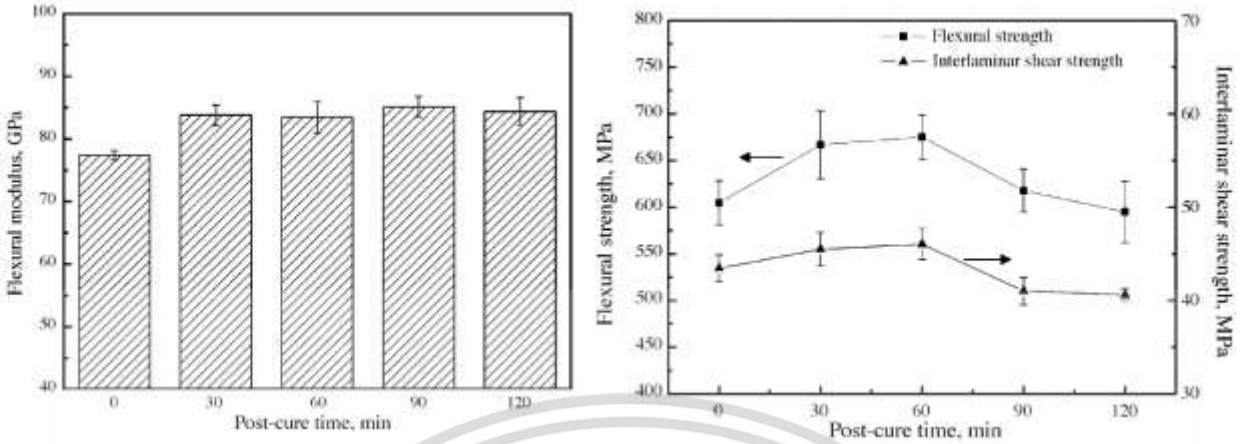


Figure 2.28 The effect of post-cure time on the flexural strength and the ILSS of the composite laminate

Figure 2.29 SEM micrographs from the fracture surface after flexural test under different post-cure duration: (a) 0 min (b) 60 min (c) 120 min [43]

Wang studied about a rapid curing resin in composite with thermoset matrix. The composite was fabricated using vacuum assisted resin transfer moulding (VARTM) for short curing time. The composite was cured under 120ºC for various mins to investigate mechanical properties, thermal properties and fracture mechanism of different timing cure composite [42]. As the result, figure 2.26 show that 60mins cure of composite have the maximum flexural strength and ILSS strength based on timing of curing composite. The flexural fracture mechanism proved that 0 min and 120mins cured composite fiber surface are very clear due to the weakness of bonding between fiber and matrix. 60mins curing composite shows fiber and epoxy are tightly bond and load transferring are well done. Therefore, the moderate post cure improve the interfacial bonding of matrix and fiber while the degradation are start with exceeded post cure temperature [43].

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Experimental Equipment

3.1.1 Carbon Fiber and Glass Fiber

Two type of carbon fiber (twill and plain) and two type of glass fiber (woven and fabrics) are use in current research with different woven pattern. All of the data are shown in table1. All of materials are commercial materials which are available in the market and bought from concrete composite. Figure 3.1 shows four different type of carbon fiber, glass fiber and matrix call epoxy.

Table 3.1 Material Properties

Material	Based	Fil Dia (μm)	Weigth (g/m^2)	Thickness (mm)	Tensile Strength (MPa)	T Modulus (MPa)	Density (g/cm^3)	Chemical Comp
Carbon Fiber Twill	PAN	5	240	0.32	-	-	1.8	
Carbon Fiber Plain	PAN	5	200	0.29	-	-	1.8	CF - 95-99%
Glass Fiber Woven	E-Glass	17	400	0.32	286	18,800	-	
Glass Fiber Fabrics	E-Glass	17	200	0.2	260	16,900	-	Alumina Silica
YD 582 (Resin)	Thermoset				440	2,850		
TH 7253 Hardener	Thermoset							

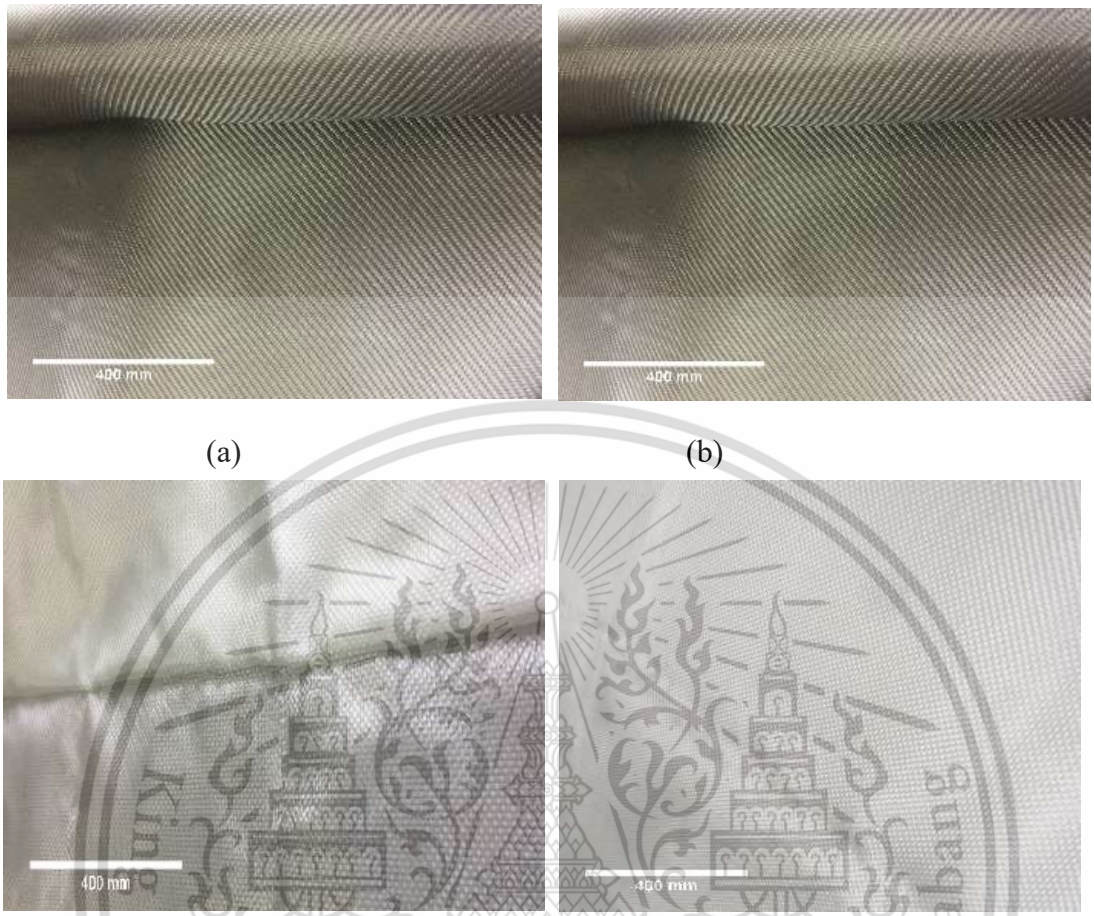


Figure 3.1 (a) Carbon fiber twill weave (CFT), (b) Carbon fiber plain weave (CFP), (c) Glass fiber woven (GFW) and (d)Glass fiber fabrics

Orientation for all fiber are bidirectional 0/90º weave pattern. Two carbon fiber are different in weave pattern such as twill and plain weave pattern and fiber bundle filament are different either. For glass fiber woven and fabrics, the quantity of filament in one bundle are different in bundle. Based on fibers bundle thickness and woven pattern, the strength are changed in composite and also in fabric itself. These four types of fibers are choose to observe the structure, morphologies, mechanical properties of different fiber pattern and fracture mechanism of composite.

Most of researcher requests special material from company to make a research deeply, but in this research, I would like to study the fiber that available in Thailand market which is commercial material. Therefore, all of materials was order from Concrete Composite Company. Most of the specification are receive from company and glass fiber properties result are similar to the supplier specification while carbon fiber mechanical properties did not show in specification. Glass fiber is not the best one, the best mechanical properties are come from carbon fiber due to its stiffness and brittle. Compare to carbon fiber, tensile strength and modulus of glass fiber are quite low about half or more than half. But some researcher make hybrid composite which are mix with carbon fiber and glass fiber in a composite, which make the higher mechanical properties.

3.1.2 Epoxy Resin

In composite field, there are several matric epoxy resin to reinforce fiber such as thermoplastics and thermoset. Thermosetting material is one of the best matrix for composite and it was used in current research. There is always resin and hardener to mix together to reinforce fiber in composite. The ratio of this two component resin: hardener is 100:35: ratio. YD 582 resin the mixture of 1) Bisphenol-A-(epichlorohydrin) epoxy resin (number average molecular weight _ 700) – 75-95% and 2) 1, 6-Hexanedioldiglycidyl ether- 5-25%. TH 7253 hardener is choose to use in this composite. Glass transition temperature for this material is at 65ºC for curing at 25ºC for 8 days. The mixing ratio of resin and hardener are 35:100. The main operating temperature is available between -60ºC and +80ºC.

Figure 3.2 (a) epoxy resin and (b) hardener

3.1.3 Raman spectroscopy

Raman spectroscopy is one of the tool that can detect the structure of nano material such as Silicone, diamond, amorphous and graphite structure of all materials. In this research, Raman spectroscopy is use to reveal the structure of carbon fiber and glass fiber. Operation condition of Raman spectroscopy for carbon and glass fiber are 514nm of wavelength (λ) and lens operate with 5μm. Based on the data from Raman spectroscopy, data analysis tool original peak (6) software was use to analyze the data to obtain respective peak intensities that can represent the structure of the materials. From peak analysis intensity of G (graphite) band and D (disorder) band, the intensities ratio(R) of D band and G band, the length of nano crystallize fringe can obtain from data analysis with original peak based on Gaussian law calculation method.

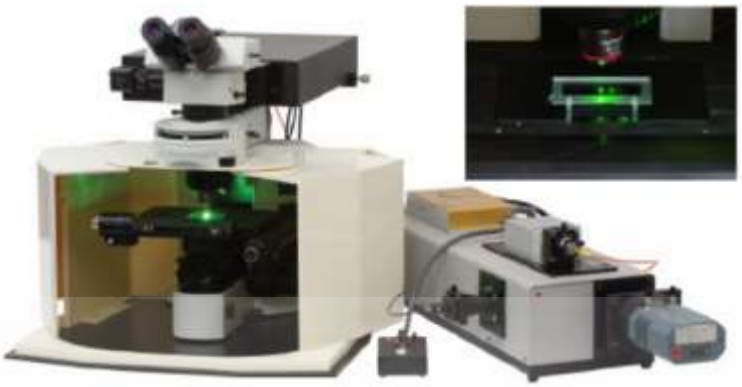


Figure 3.3 Raman spectroscopy

3.1.4 X-Ray Diffraction (XRD)

X-ray diffraction machine is one of the tool that can reveal the structure of the material in nano material field. In this research, XRD machine is from Tokyo Institute of Technology, Japan. Carbon/glass fiber and their composite are observe their structure in this research. The data of 2θ (theta) was start from 13º until 90º degree for all of data. Crystallite size (L_a), crystallite height (L_c), spacing (d_{002}) between two graphene at plane 002 and number of graphene in one graphite lattice fringe are calculated by using Scherer's formula and Bragg's law equation 1-4, Which can reveal the nanocrystalline of carbon and glass fiber.

$$Lc = \frac{0.9x\lambda}{\beta 002 xCOS(\theta 002)} \quad \text{..... (1)}$$

$$La = \frac{1.94x\lambda}{\beta 001/101 xCOS(\theta 001/101)} \quad \text{..... (2)}$$

$$n = \frac{Lc}{D002} \quad \text{..... (3)}$$

$$D_{002} = \frac{\lambda}{2xSIN(\theta 002)} \quad \text{..... (4)}$$

3.1.5 Optical Microscopy

Optical Microscopy is use to investigate the structure of material in macro scale. In this research, optical microscopy ks used to observe the structure of composite, fracture of composite and their porous. The composite are check before and after cured in high temperature oven to investigate the changes of composite due to high temperature cured. The fracture mechanism of composite are also investigate by OM.

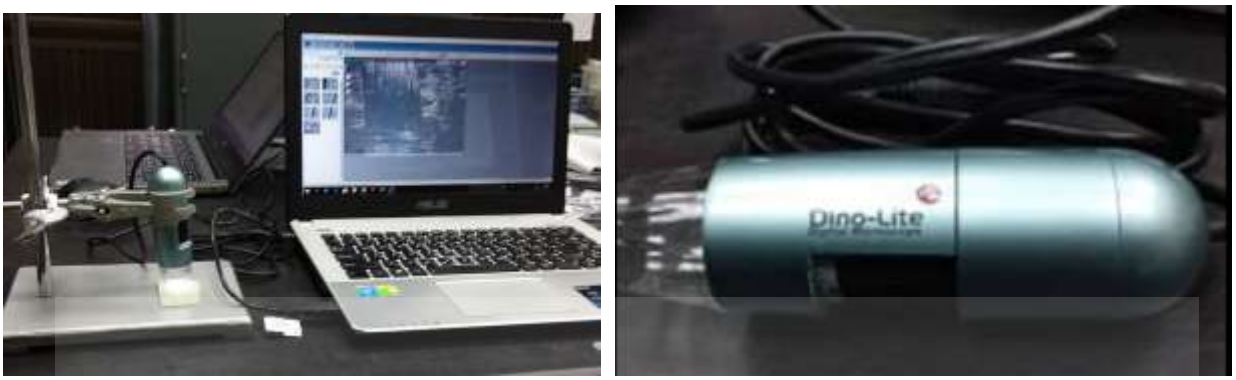


Figure 3.4 Dino-Lite Optical Microscope

3.1.6 Field Emission Scanning Electron Microscopy (FESEM)

Scanning electron microscopy (SEM) with energy dispersion spectrometer (SEM/EDS) is the tool that can investigate the morphologies of carbon/glass fiber and chemical composition of materials. In carbon and glass fiber, the surface and cross section of fibers are investigate using SEM. The containing percent of chemical are also observed using SEM/EDS. FESEM machine (Hitachi SU8200 Series) and Joel Machine (JCM- 6000) are used in this research.

Figure 3.5 Scanning electron microscopy Hitachi SU8200 series

3.1.7 Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) is use to observe the structure of carbon fiber and glass fiber in nano scale. Energy dispersion spectroscopy and diffraction pattern are also use to determine the chemical composition and diffraction of specimens. 200kV and high magnification of series JEM-2100 (URP) is use to detect the structure of carbon fiber and glass fiber. Carbon fiber and glass fiber were cut to be the smallest piece around 100nm by Microtome (diamond knife) method due to its original softness.

Figure 3.6 Transmission Electron Microscopy machine

3.1.8 Fourier Transform Infrared (FT-IR)

FT-IR is one of the tool that can observe the element bonding based on spectra peak of each materials. There are two type of sensor to observe different material. Normal illumination and ATR. For current research, illumination sensor use for fiber alone and ATR sensor use for composite. JAS.CO FT/IR – 4700 serial number A080061809 is used in this research with the wavenumber 400 – 4000cm^{-1}.

3.1.9 Tensile test

Tensile test is one of the main tool to investigate mechanical properties composite field and other materials . In this research, all of the fabric and the composite were tested by the machine based on ASTM D638. Fabric fibers are cut to be the shape of rectangle with 127mm length and 12.7mm width. The thickness of samples are based on supplier's material thickness. Tape is used to cover the fabric at both side not to freak out the pattern while tensile test are perform in fabric. The prepared specimens are tested in

tensile machine INSTRON 8501 with the condition of 50mm/min, 95KN load cell. Composite specimens are also test by this machine with the same condition. ASTM D683 is used as a testing method of composite.

Figure 3.7 Tensile machine with 95 Load cell, INSTRON 8501 tester and Composite Specimens

The main standard of ASTM 638 was used to calculate tensile strength in the mechanical properties as describe in equation (5-7).

$$\sigma = \frac{P_{max}}{A} \qquad (5)$$

Where, σ = Tensile stress, MPa

P_{max} = Maximum Load to failure, N and A = Average cross sectional area, m^2

$$\varepsilon = \frac{\delta}{L} \qquad (6)$$

ε = tensile strain at *i*th data point, δ = extensometer displacement at *i*th data point, mm; and L = gage length, mm

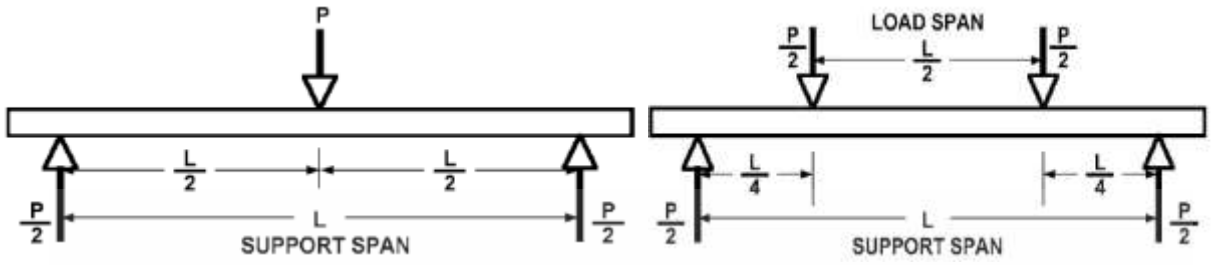
$$E = \frac{\Delta\sigma}{\Delta\varepsilon} \qquad (7)$$

Where; E = tensile chord modulus of elasticity, GPa; $\Delta\sigma$ = difference in applied tensile stress between the two strain points, MPa; $\Delta\varepsilon$ = difference between the two strain points.

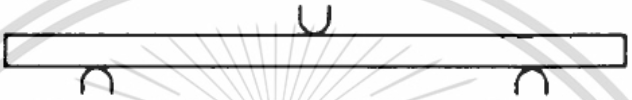
3.1.10 Flexural Test

Flexural test is one of the important mechanical properties of material at maximum applied load without any permanent deformation. Flexural test or bending test is test to know the extent to which object resist when the object are bent and also to know the stability of material. The testing condition was

differ base on the materials and the usage function. The figures are shown about three point and four point bending test of composite base on ASTM 790.



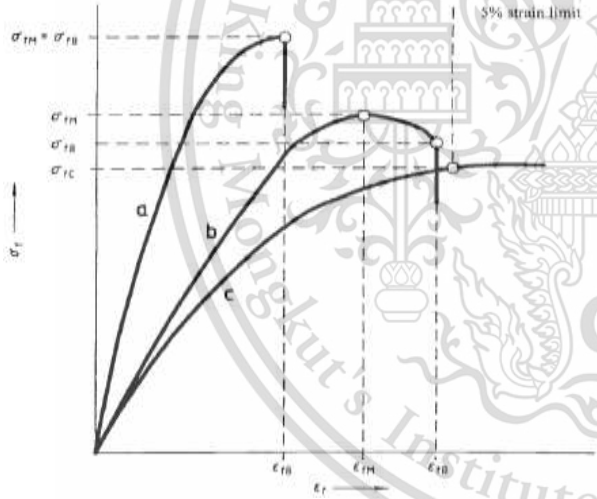
(a) Three point bending test (b) Four point bending test.



(c) Bending nose radius with 3.2 mm

Figure 3.8 The testing method of flexural strength.

The mechanical test of composite



σ_f = Flexural Stress,

ε_f = Flexural strain,

σ_{fm} = Flexural Strength,

σ_{fB} = Flexural Stress at Break,

ε_{fm} = Flexural strain at maximum load,

ε_{fB} = Flexural strain at Break,

Curve a: Specimen that breaks before yielding, curve b: Specimen that yields and then breaks before the 5 % strain limit and curve c: Specimen that neither yields nor breaks before the 5 % strain limit.

Figure 3.9 Typical Curves of Flexural Stress (σ_f) Versus Flexural Strain (ε_f)

$$\sigma = \frac{3PL}{2bd^2} \quad (8)$$

σ = stress in the outer fibers at midpoint, MPa
P = load at 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

$$\varepsilon = \frac{6Dd}{L^2} \quad (9)$$

ε = strain of outer surface, mm/mm
D = Max deflection of the center of beam, mm

$$E = \frac{L^3 m}{4bd^2} \qquad (10)$$

E = Modulus of elasticity in bending, MPA
m = slope tangent of load deflection, N/mm

Flexural test (three-points bending) was performed at 24ºC temperature, 49% RH with INSTRON 55R4502 machine. Testing rate speed is 5.2mm/min with cell load 10KN. All of the composite are test in the same conditional. ASTM D790 method was use for flexural test of all composite. For load crosshead motion of flexural test, the following equation (6) was used to determine the rate of crosshead speed. The support span length was use 32:1 ratio due to it was composite material based on the composite thickness. To determine the flexural stress, equation (7) is used and it was automatic calculated in machine.

$$R = \frac{ZL^2}{6d} \qquad (11)$$

Where, R = rate of crosshead motion (mm/min)

L = support span length, mm

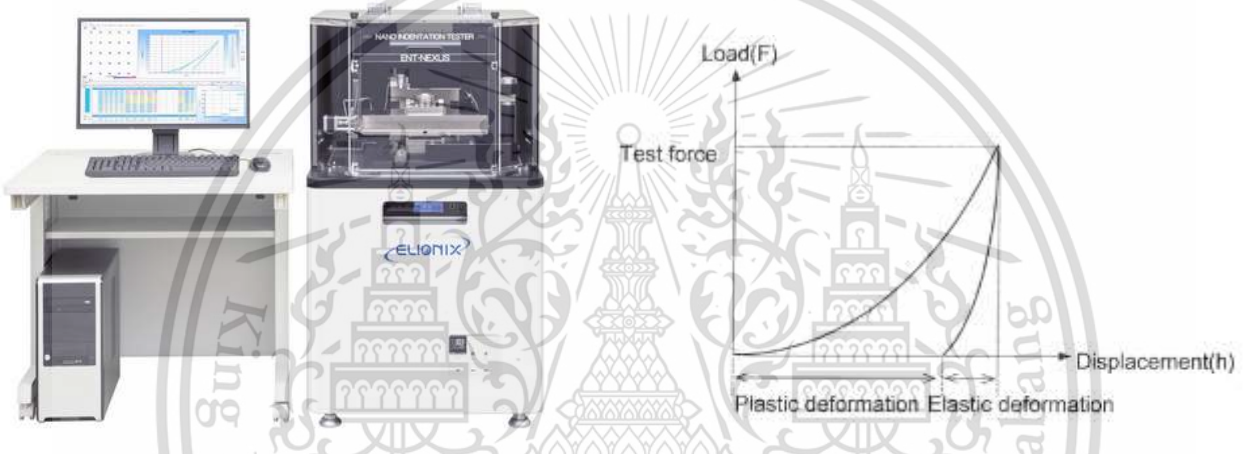
D = depth of beam /thickness (mm)

Z = rate of straining of the outer fiber, mm/ (mm^{-1}) min, Z=0.01 was used

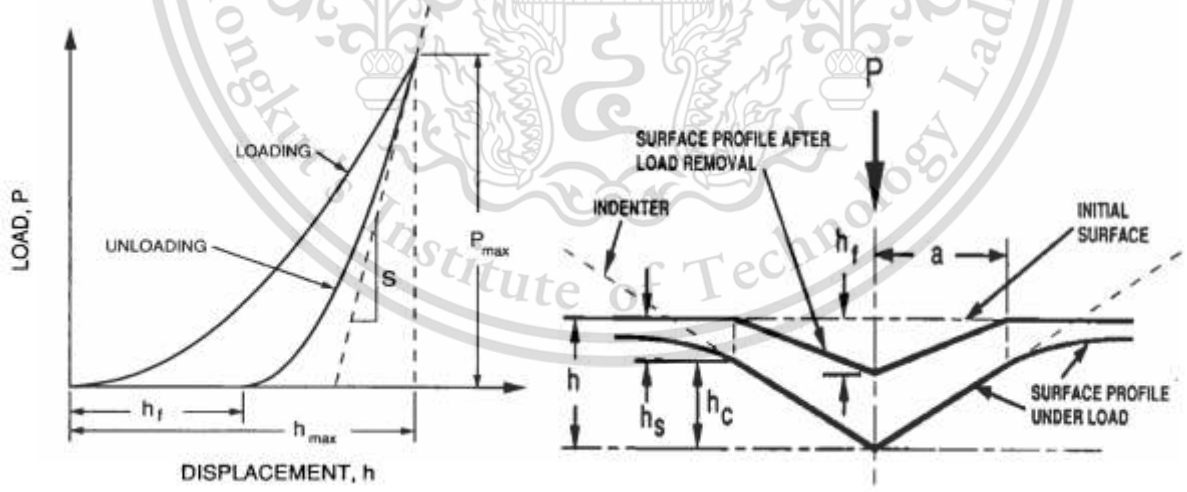
Figure 3.10 Flexural tester, INSTRON 55R4502

3.1.11 Indentation Test

Indentation test is the tool to observe the mechanical properties of the materials especially modulus and hardness of material. In this research, indentation testing method is used to investigate the hardness and young modulus of composite that are cured at room temperature until it is dry and cured composite at 80ºC for 6hrs. Elionix (ENT – 1100a) indentation tester is used in this research. Operating condition at room temperature with max load 200mN, set up time for a test is 10,000sec and 500 steps (200sec/ step). This result shows increasing hardness and modulus of materials with the increased of curing temperature especially in carbon fiber composite while glass fiber do not improve significantly.



(a) Testing machine Elionix (b) graph of indentation loading



(c) Graph of indentation (d) representation of indentation analysis

Figure 3.11 Indentation machine Elionix (ENT-1100a) and operating graph of indentation curve

Figure 3.11 shows the graph of operating indentation test. Three sided pyramid diamond indenter known as Berkovich indenter is used to determine modulus and material hardness. The following equation (12-15) are used to calculate mechanical properties.

$$H = \frac{F_{max}}{A_p(h_c)} \ldots\ldots\ldots\ldots\ldots\ldots (12)$$

Where H is hardness, F is maximum applied force, A is contact area and h_c is depth of contact at maximum force. Elastic modulus can be calculated with equation (13-15). Where E is modulus, V_s: Poisson ratio of the test piece, V_i: Poisson ratio of indenter, E_r: elastic modulus from contact point, E_i: indenter elastic modulus A_p (h_c): contact area.

$$E = \frac{1 - \nu s^2}{\frac{1}{Er} - \frac{1 - Vi^2}{Ei}} \quad \ldots\ldots\ldots\ldots\ldots (13)$$

$$Er = \frac{\sqrt{\pi}}{2C\sqrt{Ap(hc)}} \quad \ldots\ldots\ldots\ldots\ldots (14)$$

$$h_c = h_{max} - \varepsilon(h_{max} - h_r) \quad \ldots\ldots\ldots\ldots\ldots (15)$$

3.1.12 Thermo gravimetric Analysis (TGA)

Thermo gravimetric analyser is one of the powerful tools to investigate fiber volume fraction and thermal stability of composite. In this research, carbon and glass fiber were burned out in TGA with the condition of temperature within 30ºC-900ºC with 10ºC/min, supplied pure air with 50ml/min. Different result are observe in carbon fiber and glass fiber. The composite with different curing temperature are also analyse using TGA. The result of composite is different with different curing temperature of composite. METTLER TOLEDO TGA/DSC 3^+ Reference No. SCL17060040-1 machine was used in this research as shown in figure 3.12.

Figure 3.12 Thermo gravimetric Analyzer TGA/DSC 3^+

3.1.13 Treatment Fiber and DLC Coating

Most of fibers are produced by wet or dry spinning method. Most of manufacturer coated the fiber by coupling agent or epoxy after treatment fiber to become pitting valley on the surface of fiber that can help interlocking with matrix. The coating layer of epoxy or polymer may cover the pitting valley of fiber surface which can make the weakness in interlocking. Therefore, treatment (removing coating layer) is used in this research and coating diamond like carbon (DLC) to increase the mechanical interlocking. Treatment process as below procedure.

Treatment Procedure

1. Kapton tape is used not to freak out fabric
2. clean with Ethanol for : 30Mins t
3. Wash with Distilled water for : 10 Mins
4. Curing at 80ºC for 1hr and 30 mins

After treatment the fiber, the fiber were coated by DLC to enhance the adhesive and hardness properties by plasma assistance chemical vapor deposition (PACVD). The machine was operated with 3Pa pressure, 3.5kV and apply argon (Ar) for 10 mins with 20 cm^3/min to clean out the room and the specimens and apply acetylene (C_2H_2) for 30mins in each side as the main materials as shown in figure 3.13. The coated fabric were test in tensile test as the previous method of section 3.1.9 to know DLC coated fabric property. DLC coated fabric are not used to fabricated composite.

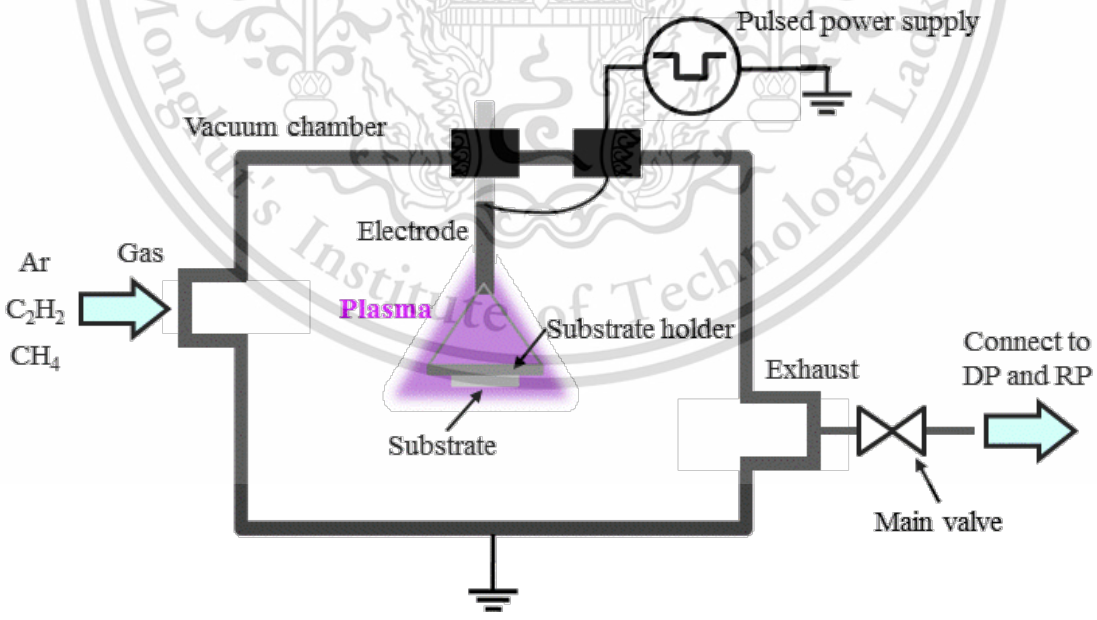


Figure 3.13 Procedure Schematic of DLC machine and Performance

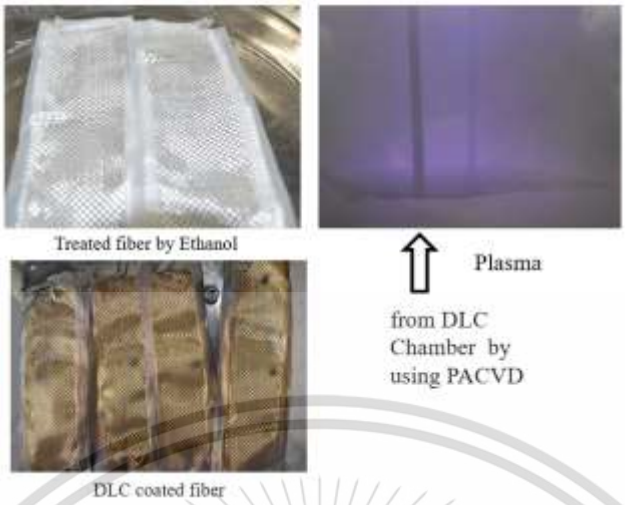


Figure 3.14 Treated fiber DLC deposition by Plasma Assistant Chemical Vapour Deposition

3.2 Fabrication Composite

There are several method to make composite. In this research, all of the fibers are fabricated by using hand lay up process to 0/90º orientation weave pattern. In epoxy mixing ratio, part A: epoxy and part B: hardener are mix for 3:1 ratio based on the fiber ratio and equations (16-18) to calculate the ratio. The thickness of composite are control based on the fabric thickness of each layer. The same method is use for all composites with hand lay-up process. All of laid up conditions can see in table 3.2 and composite are fabricate based on ASTM D638[1] for tensile specimens and ASTM 790 [2] Flexural test. Composite thickness are calculated by fabric thickness. The composite are cured at room temperature until it was dried. Room temperature is consider to be 30ºC in Thailand.

$$Hardener\ weight\ (g) = \frac{Weight\ of\ Fibers\ (g)}{4} \quad (16)$$

$$Epoxy\ weight\ (g) = \ weight\ of\ hardener\ (g)\ x\ 3 \quad (17)$$

$$Epoxy\ and\ hardener\ ratio = 3:1 \quad (18)$$

Composites are cut by computer numerical control (CNC) machine Mazak FJV-20 with the parameter of spindle speed 150 mm/min and feed speed 50-100mm/min according to ASTM specimens shape. Samples are prepared according to ASTM 638 for tensile test and ASTM 790 for flexural test specimens. All four type of fiber are produced composite with three plate in each type of fiber. Six specimens were test in each materials but all of six specimens are not from the same plate composite, all of specimens are collected two pieces each from each plate of composite. This is the reason to know the

deviation of each plate and the stability of hand lay up process. Additionally, after cutting dried composite, some specimens were cured at 80ºC for 6hrs and another sample at 120ºC for 3hrs.

For better quality control, the composite are need to check deeply not only in visual by eye but also by scanning machine which can investigate the defects in the composite. In current research, only human eyes are used to check the defects or the imperfection of composite. But in the future, composite material usage are increase widely across the whole world in many field, therefore better or advance tool such as ultrasonic scan are need to use to control the quality. Some of researcher also used Ultrosonic sensor or scan to investigate, the porosity of composite, microcrack, defects area, delaminations area and impact damage[52,53]. These are the tool that will use to inspect the composite in the near future.

3.3 Treatment Fiber

Composite were tested in tensile and flexural test to collect the highest mechanical properties of all composite. The highest mechanical properties of fiber were carbon fiber twill weave and glass fiber woven composite. The fracture mechanism of composite are investigated and it was due to the sizing of material on the fiber surface. The bonding between fiber and matrix are not well adhesive, this is one of the reason that interface between fiber and matrix is not good enough. There is another way to make a treatment on the fiber surface to remove the sizing material and make composite again. Treatment fiber will make the fiber rougher and more pitting on the fiber surface, this will help to make mechanical interlock between fiber and matrix. Treatment fibers process are as show in figure 3.15 and the process are as follow,

1. Material were choose carbon fiber twill weave and glass fiber woven
2. For treatment, the fabric edge were cover with tape not to freak out the pattern of the fiber
3. For De-sizing or treatment fiber, the fabric was immerse in acetone for 30mins to soak out all of the sizing material from the fiber surface.
4. The treatment fibers was wash out by immersing the fibers in DI (deionized water) for 10mins
5. After wash out by DI, the fiber were dry at 120ºC for 30mins.

After all of the fabric was treated, it was used to fabricate the material as the previous composite fabricated method. Table 3.2 shows the composite thickness, layers and condition of all composite which are used in this research. All of the fibers were cured at room temperature (RT is consider at 30ºC) until it was dry after that, the composites were cured at 80ºC for 6hrs and 120ºC for 3hrs to investigate the effect of high temperature cured on the composite.

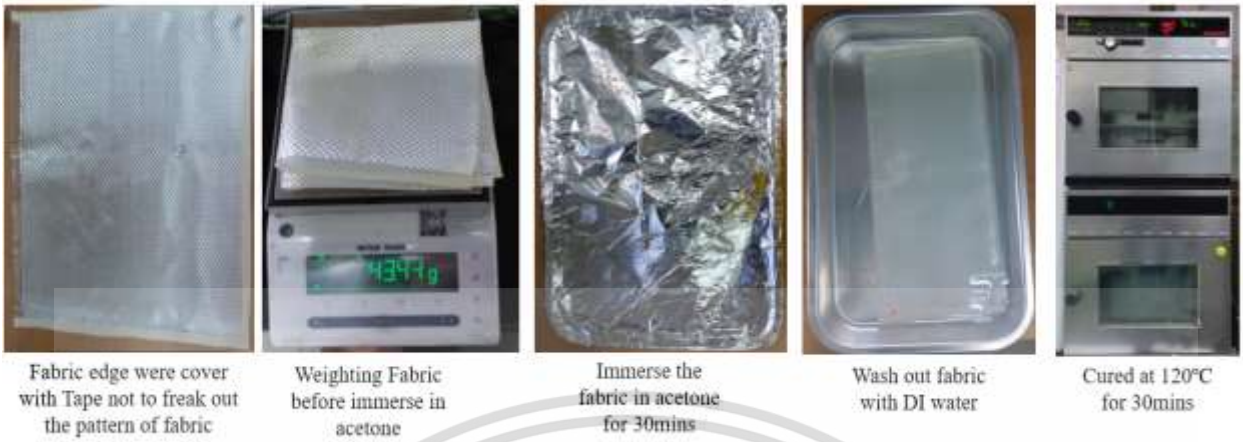


Figure 3.15 Treatment fiber process

Table 3.2 composite parameter and name in this research.

No.	Material	Condition	Filament Dia (μm)	Weight (g/m^2)	Layers	Cured Condition	Name
1	Carbon Fiber Twill	-	5-7 um	240	10	(1) at 30ºC	CFT30
						(2) 80ºC for 6hrs	CFT80
						(3) 120ºC for 3hrs	CFT120
2	Carbon Fiber Plain	-	5-7 um	200	10	(1) at 30ºC	CFP30
						(2) 80ºC for 6hrs	CFP80
						(3) 120ºC for 3hrs	CFP120
3	Glass Fiber Woven	-	17	400	10	(1) at 30ºC	GFW30
						(2) 80ºC for 6hrs	GFW80
						(3) 120ºC for 3hrs	GFW120
4	Glass Fiber Fabric	-	17	200	16	(1) at 30ºC	GFF30
						(2) 80ºC for 6hrs	GFF80
						(3) 120ºC for 3hrs	GFF120
5	Carbon Fiber Twill	Treatment F	5-7 um	240	10	(1) at 30ºC	TCFT30
						(2) 80ºC for 6hrs	TCFT80
						(3) 120ºC for 3hrs	TCFT120
6	Glass Fiber Woven	Treatment F	17	400	10	(1) at 30ºC	TGFW30
						(2) 80ºC for 6hrs	TGFW80
						(3) 120ºC for 3hrs	TGFW120

3.4 Experimental Procedure

The commercial material of carbon fiber and glass fibers such as CFT, CFP, GFW and GFF were investigated the morphologies and their characteristic by using SEM, TEM, Raman spectroscopy, x-ray diffraction and Fourier transform infrared. Tensile tester are used to investigate the properties of mechanical properties of all fabrics.

The composite is fabricate by hand lay up process as describe in section 3.2.13 and cured at different temperature. The composites is test in mechanical properties through tensile test (ASTM 638) and flexural test (ASTM 790). Nano indentation were test to know the hardness and modulus of each composite at 30ºC cured fiber and 80ºC for 6hrs cured composite. Among four type composite of different fiber, carbon fiber twill (CFT) and glass fiber woven (GFW) have the highest mechanical properties, therefore they are chosen to investigate in treatment fiber composite. The chosen fiber CFT and GFW are treated by acetone and use them to make composite. This composite also tested in tensile and flexural test to observe the mechanical properties. The overall brief description are as shown in figure 3.16.

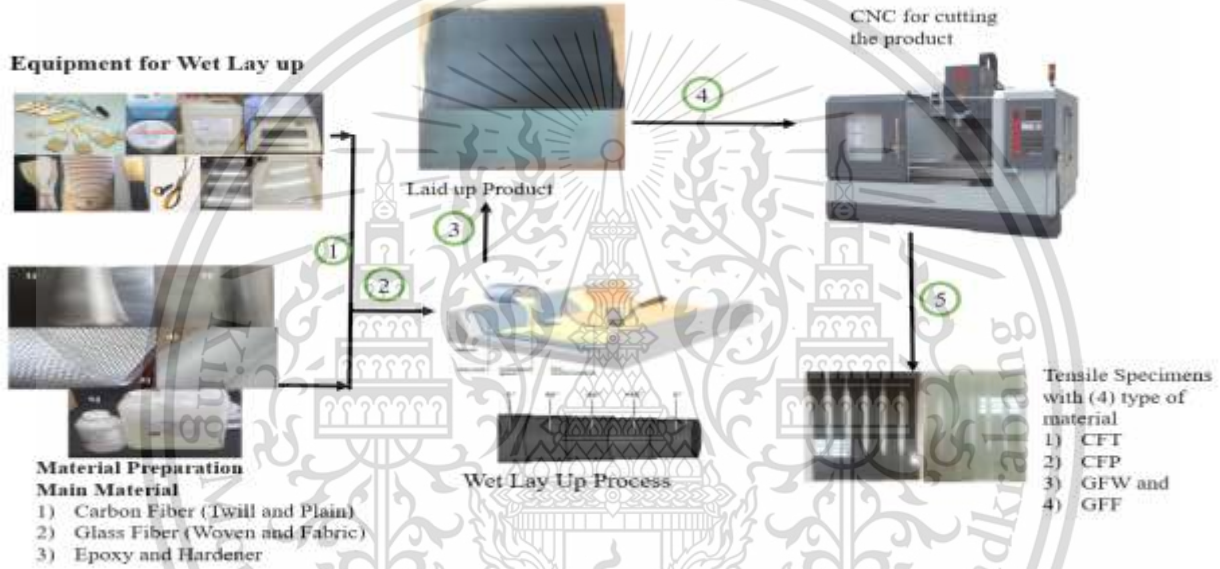


Figure 3.16 Schematic diagram of the overall research

Fracture mechanism of composite are examine by scanning electron microscopy and optical microscopy.

CHAPTER 4

RESULT AND DISCUSSION

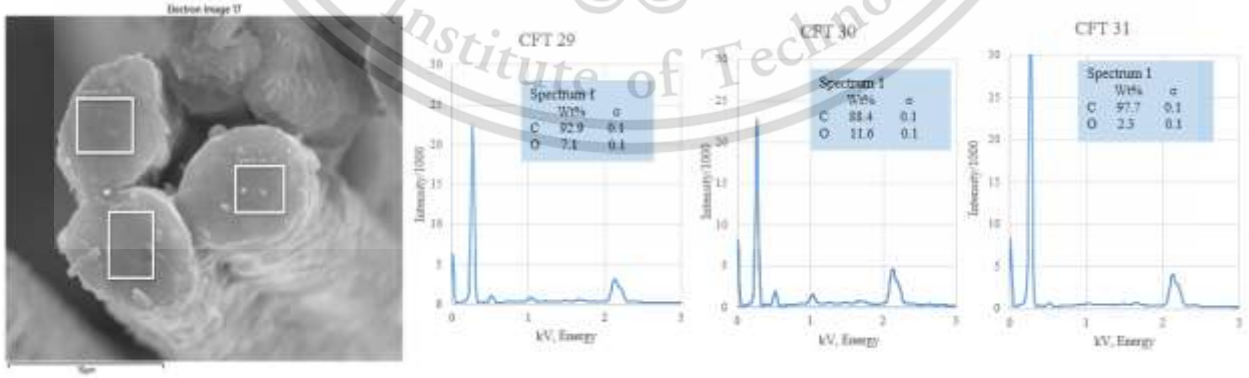
4.1 Characteristic and Morphology of Carbon and Class Fiber

The morphologies of carbon fiber and glass fiber both were studied by field emission scanning electron microscopy (FESEM). From SEM, the surface of fiber and cross section of fiber parts were investigated. First of all, morphologies of carbon fiber twill weave will discuss.

4.1.1 Carbon Fiber Twill (CFT)

The fiber woven pattern of twill weave with around 3000 filament in one bundle. In CFT, the commercial fiber is used to observe its morphologies, and found that the whole fibers surface are rough with small pitting line along fiber surface. This will help mechanical locking between fiber and epoxy to the composite. The surface of fiber pitting line are not stable and it can assume the structure of fiber are mixing with graphite and cluster structure. The cross section of carbon fiber is not quite smooth due to the fiber cutter scissor. It seems the fiber structure are well organize without voids and porous in the fiber cross section as shown in figure 4.1(a). The chemical composition of fiber are investigated by SEM/EDS. Three area of fiber were detected as shown in figure 4.1 (b).

(a) Surface and cross section of Carbon fiber with different magnification

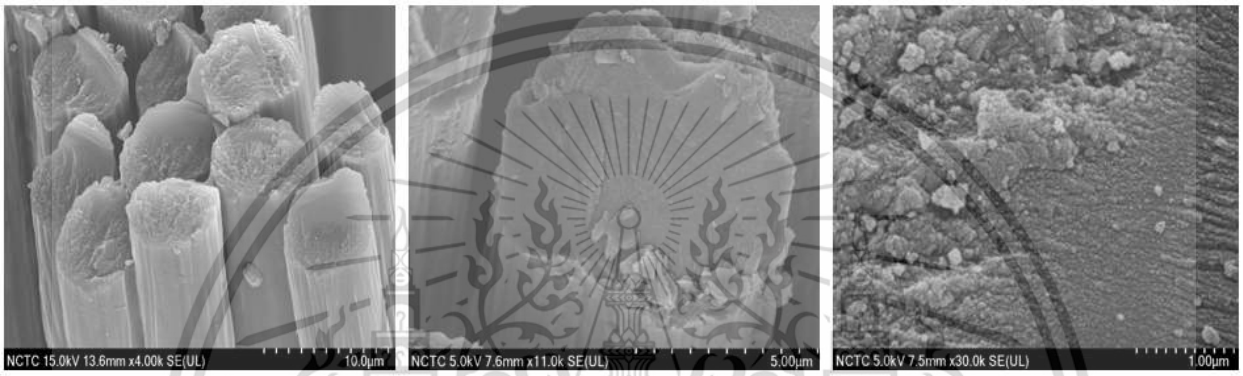


(b) The chemical composition from energy dispersion spectroscopy

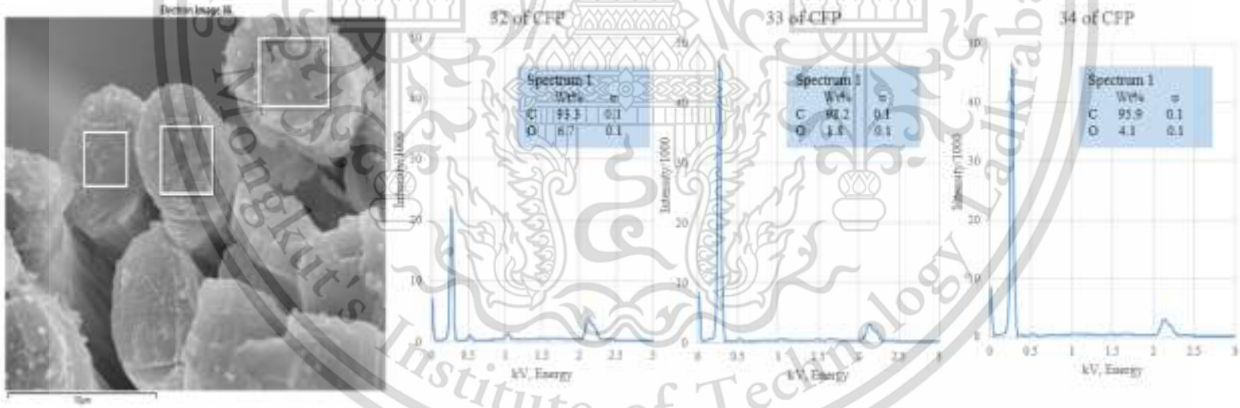
Figure 4.1 morphologies of carbon fiber twill weave

4.1.2 Carbon Fiber Plain (CFP)

The carbon fiber plain is weave pattern fiber is plain fiber bundle one by one. The filament is around 2000 filament in one bundle of fiber. The diameter of fiber filament are 5-7μm. This is one of the commercial carbon fiber in the market. CFP is investigated using SEM/EDS and the morphologies are similar with CFT result. The surface of CFP also have the roughness and pitting valley on the surface of fiber. In cross section area, there is no porous in the cross section of fiber. Around fiber surface, there is no porous are found. The chemical composition of CFP is carbon 92-99% and oxygen 2-7%. Carbon containing percent are 93%, 98% and 96% respectively from fibers cross section area and oxygen contain 7%, 2% and 4% respectively.



(a) Surface and cross section of carbon fiber CFP



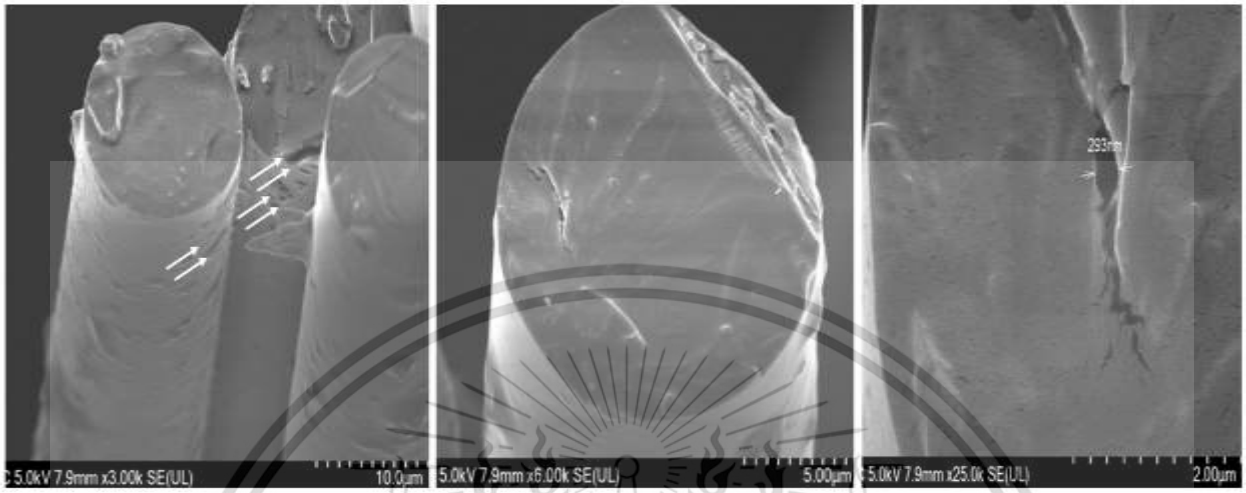
(b) Chemical composition of CFP

Figure 4.2 morphologies and chemical composition of carbon fiber plain weave

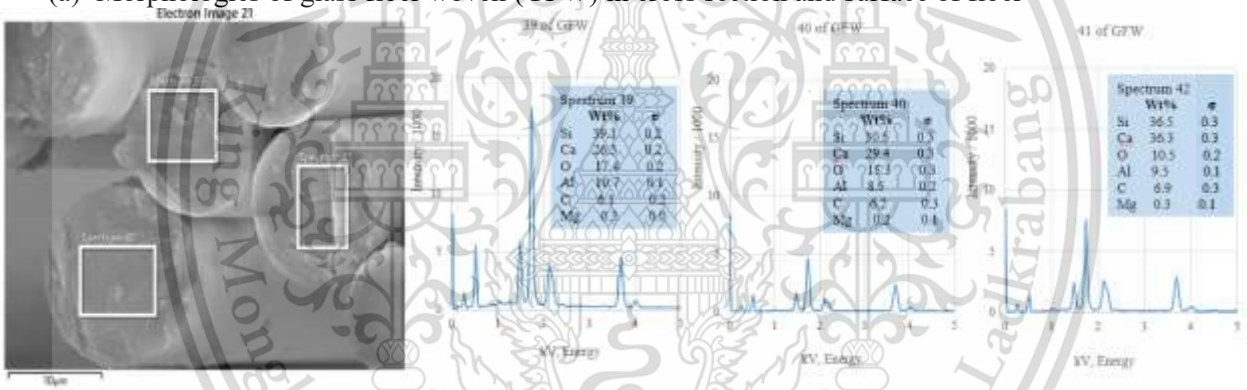
4.1.3 Glass Fiber Woven (GFW)

Glass fiber woven is the fiber that are weave in plain weave with around 3000 fiber filament in one bundle and the weight is 400g/m^2. The diameter of fiber filament is 15-17μm. The surface of the fibers are smooth than carbon fiber but it was full of defects along the fiber surface. On the other hand, there are several porosity along the fiber and also crack and fiber porous are on the cross section of fiber as shown in figure 4.3(a). The defective surface will be helpful for mechanical interlocking with epoxy and fiber. But it may be also one of the weakness to prevent the extension or elongation of fiber so it can break easily on

the porous and the crack. Based on SEM EDS, the chemical composition of GFW are Si, Ca, O, Al, C and Mg with different percentage of containing element. The highest containing element is Si between 30-40% due to it was base on silica. And the lowest element containing is Mg with around 0.2-0.3%.



(a) Morphologies of glass fiber woven (GFW) in cross section and surface of fiber

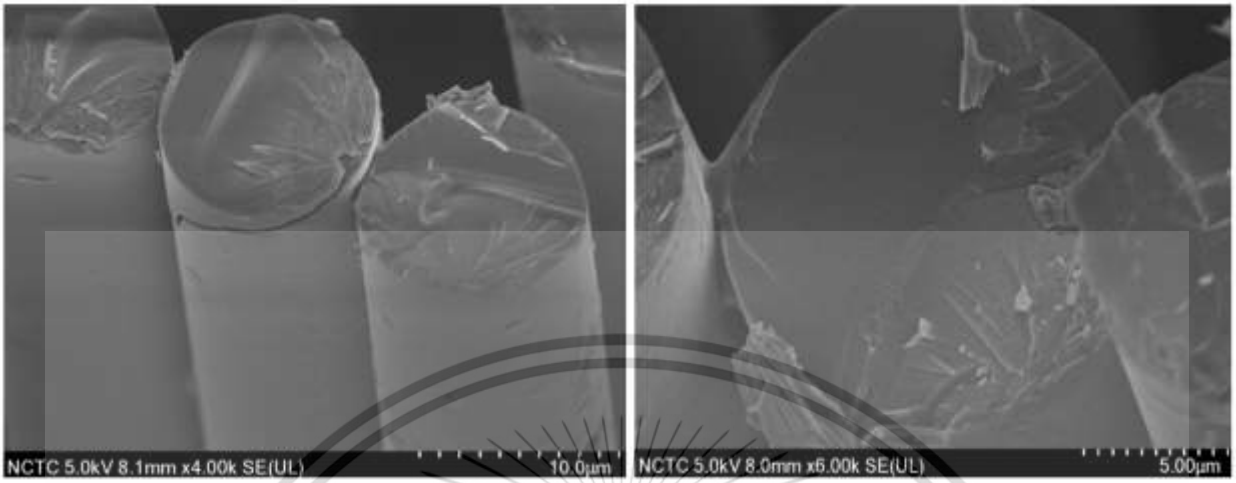


(b) Chemical composition of GFW

Figure 4.3 Morphology and chemical composition of GFW

4.1.4 Glass Fiber Fabric (GFF)

The glass fiber fabric is also one of the simple plain weave of glass bundle which has around one thousand fiber filament in one bundle of fiber. The diameter of fiber is 17μm. The color of fiber is white color. Based on SEM investigation of fiber, the fiber surface and cross section behavior is almost similar with GFW. There are several defects on the surface along the fiber axis, cracking sacra and porous are also found in cross section. The chemical composition difference between GFW and GFF is Zn, in GFW there is no any Zn element while GFF have in the range of 0.9-1.4% based of three filament measurement as shown in figure 4.1.4 (b). EDS chemical percentage of Al is not from the fiber only, some of them are come from the holder of the sample while SEM analysis are performed. Therefore, some Al chemical composition can be neglected from table 4.1.



(a) Surface of fiber and cross section of Glass fiber fabric (GFF)

(b) Chemical composition of glass fiber fabric from SEM/EDS

Figure 4.4 Morphology and chemical composition of GFF

Table 4.1 Fiber and their Chemical Composition

Element/ Fiber	C (Wt. %)	O (Wt. %)	Si (Wt. %)	Ca (Wt. %)	Al (Wt. %)	Zn (Wt. %)	Mg (Wt. %)
CFT1	92.9	7.1	-	-	-	-	-
CFT2	88.4	11.6	-	-	-	-	-
CFT3	97.7	2.3	-	-	-	-	-
CFP1	93.3	6.7	-	-	-	-	-
CFP2	98.2	1.8	-	-	-	-	-
CFP3	95.9	4.1	-	-	-	-	-
GFW1	6.1	17.4	39.1	26.3	10.7	-	0.3
GFW2	6.2	15.3	30.5	29.4	8.5	-	0.2
GFW3	6.9	10.5	36.5	36.3	9.5	-	0.3
GFF1	13.7	18.8	34.2	21.6	9.7	1.4	0.3
GFF2	11.6	25.2	32.5	20.1	9	1.2	0.3
GFF3	8.8	17.2	37.3	25.2	10.3	0.9	0.3

The description of each element or oxides group as follow:

Carbon (C): Carbon is the main element of carbon fiber and it was organic element.

Silica (SiO_2): It make increasing in strength and good acid resistance

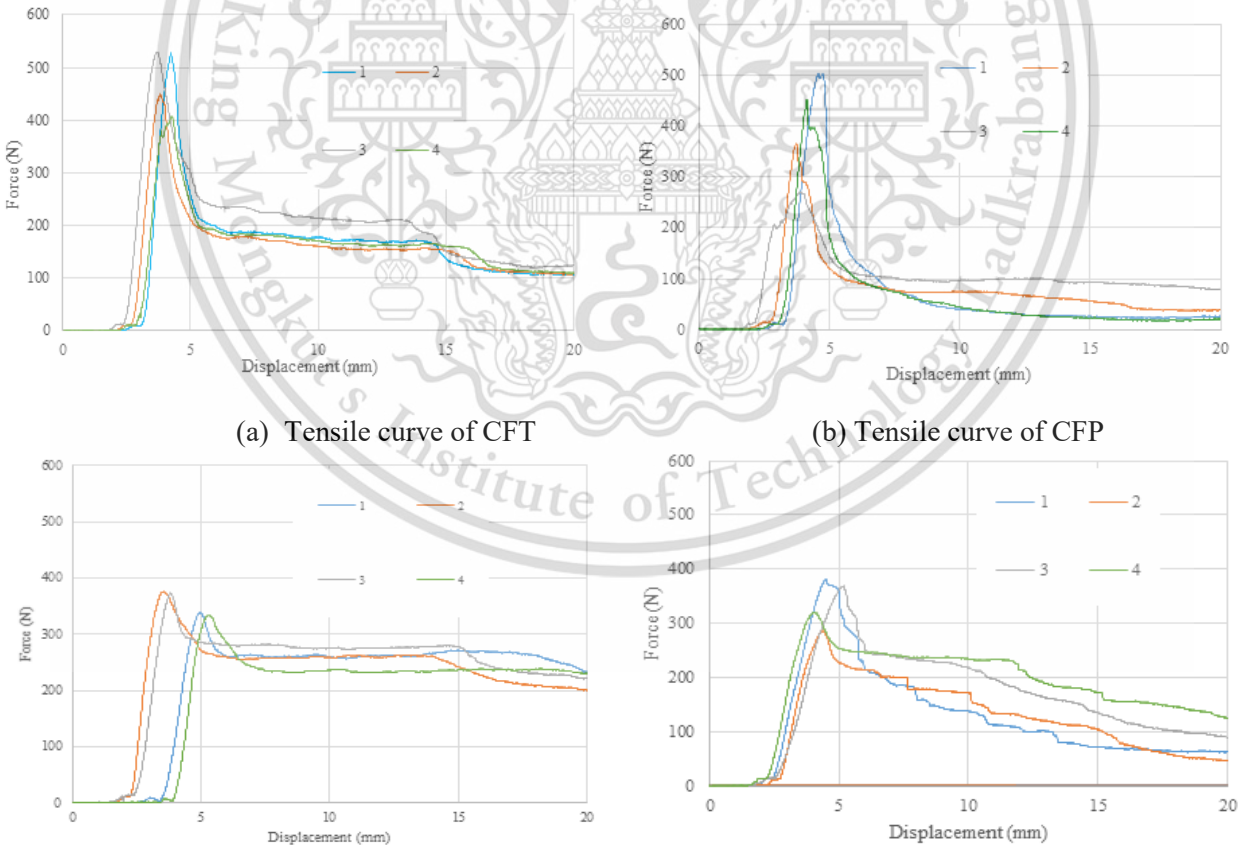
Na_2O and CaO: They make good fluid from their mixture but it was weak in durability and poor electric properties.

MgO: It make slow down to the rate of glass crystallizes.

Al_2O_3, ZnO: This function group make increasing durability and improve the moisture resistance in glass fiber.

4.1.5 Mechanical Properties of Fabric

The fabric of all of fibers were cut with the dimension of 12.7mm width, 127mm in length and the thickness of fiber are based on supplier data that was shown in table 3.1. The fabric were covered by tape in both side to test in mechanical tensile test and the properties of tape are deducted from the result of fabrics. All of fibers result are shown in figure 4.5



(a) Tensile curve of CFT (b) Tensile curve of CFP

(c) Tensile curve of GFW (d) Tensile curve of GFF

Figure 4.5 Force-Displacement curve of tensile (a) CFT, (b) GFW, (c) CFP and (d) GFF

The fiber graph shows different properties of fabric. In this graph, CFT result is the highest among fabrics due to its weave pattern of twill weave. Two of carbon fiber are same in fiber diameter and the bundle of fiber was different but how many quantity of filament are not mention. The main different point of the pattern woven are plain and twill, it make the difference in the properties of fabric. The result prove that woven pattern of twill weave is better in composite. In these all fabric test, dimensions are same in width and length, only different in the thickness of fabric that originally come from supplier and it can be base on filament quantity in one bundle. For both of glass fibers, load and displacement of tensile test was almost similar. The woven pattern of both glass fibers are plain weave but the bundle filament of fiber are different, it make the thickness different in fabric where GFW is 0.32mm and GFF is 0.2mm respectively. Therefore, the same pattern of fiber are similar in tensile result but the bundle filament quantity make small difference in fabric.

4.1.6 Characteristic of Fiber by X-Ray Diffraction (XRD)

All of the fiber filament are investigated in their behavior and characteristic in XRD machine analysis to know their structure in nano scale of the fiber. The fiber structure are examine using XRD. The data of XRD result are analyze through original peak using Gaussian method. Both of carbon fiber shows sharp peak intensity while the glass fiber do not show specific peak intensity in the graph. The peak of carbon fiber are obviously observe sharp peak at 25 of 2thelta at plane 002 as shown in figure 4.6 for both of carbon fiber. It indicate that there is the graphite structure of hexagonal carbon bonding behavior with highly order graphene sheet in the graphite structure crystallite. And another small peak were found at 001 and 101, they were represented the disorder and non-symmetric hexagonal of fiber structure. Equations 1-4, Scherrer's formula and Bragg's law are used to calculate the crystallite size and interspacing dimension of graphite structure. The original data peak and the calculated result are describe in table 4.2, the result show graphite crystallite size (L_a) 6.98 nm for CFP and 7.67nm for CFT. It represent CFP graphite order crystallite are less than CFT. Contrary, the high of crystallite of CFP is higher than that of CFT with the value 11.13nm for CFP and 9.32nm for CFT. The highly order graphite might be one of the better effect in mechanical properties. On the other, glass fiber do not have any specific peak for both of GFW and GFF, it represent the structure of amorphous in the fiber. It was agree with the previous glass fiber research.

$$L_c = \frac{0.9x\lambda}{\beta_{002}xCOS(\theta_{002})} \quad (1)$$

$$L_a = \frac{1.94x\lambda}{\beta_{001/101}xCOS(\theta_{001/101})} \quad (2)$$

$$D_{002} = \frac{\lambda}{2xSIN(\theta_{002})} \quad (3)$$

$$N = \frac{L_c}{D_{002}} \quad (4)$$

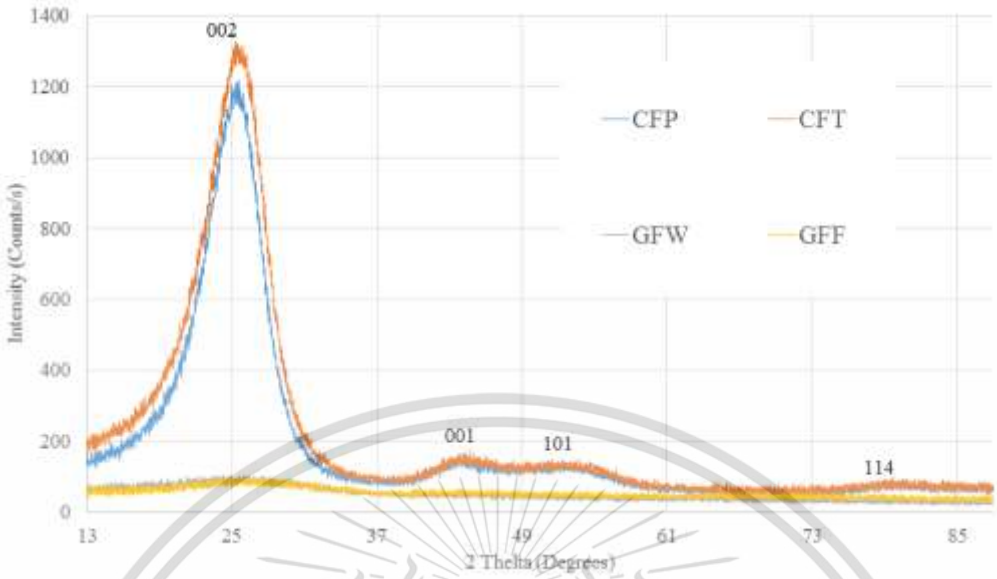


Figure 4.6 XRD intensity peak of CFT, CFP, GFW and GFF with 2 thelta in degree

Table 4.2. 2θ Value of Each Peak of Graphite Crystallite Structure

	Plane	2θ	FWHM (β)	L_a (nm)	L_c (nm)	D_{002} (nm)	n
CFP	200	25.01	7.31	6.98			
	100/101	47.56	26.8	-	11.13	0.36	27.82
CFT	200	25.08	8.77	7.67			
	100/101	47.4	24.37	-	9.32	0.35	23.30

The composite of carbon fiber and glass fiber are also doing in x-ray diffraction with the same procedure of raw fiber filament and the result are shown in figure 4.7. The graph are similar to the characteristic of raw fiber filament of carbon and glass fiber, the main intensity peak of fiber are not affect significantly by matrix epoxy. But the intensity peak of carbon fiber are quite lower than raw fiber intensity, the matrix of composite may block the fiber structure of carbon fiber. The peak are same at plane 002, 001, 101 and 144 as the raw fiber result. But the intensity of each plane peak are lower than raw fiber filament peak. Therefore, the graph show that there are more cluster behavior than raw fiber behavior in this composite due to 001 and 101 plane are become higher than raw fiber. But this small sharp intensity might come from matrix.

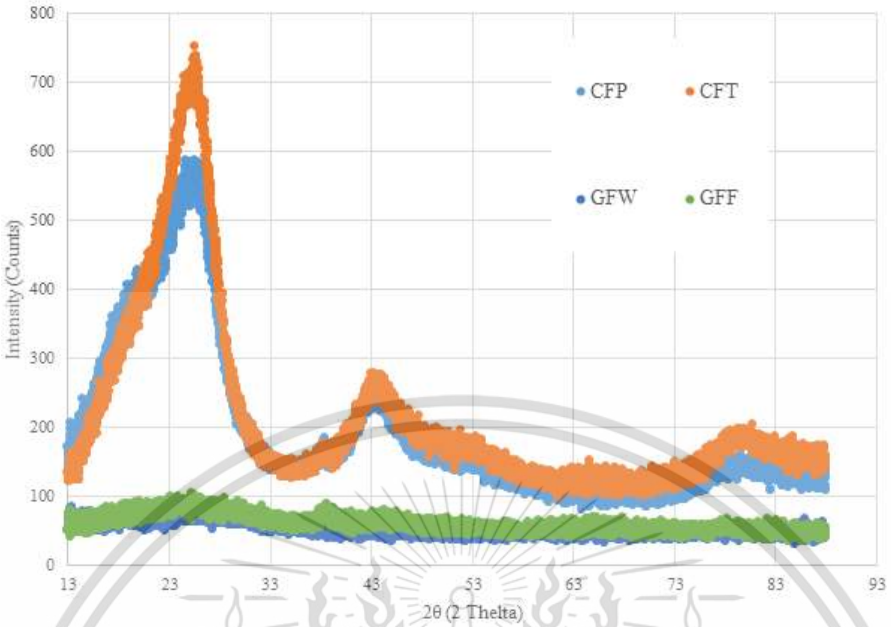
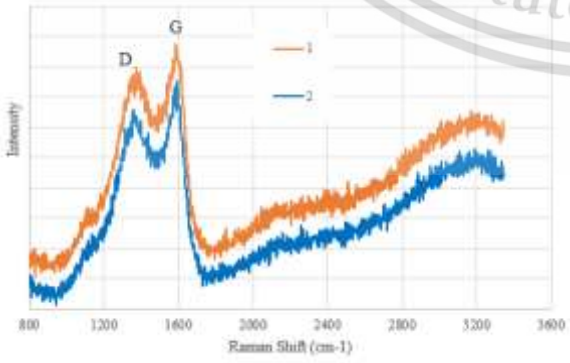


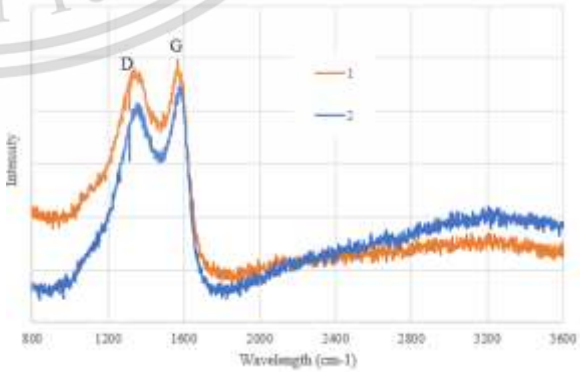
Figure 4.7 XRD intensity peak of CFT, CFP, GFW and GFF with 2 theta in degree

4.1.7 Characteristic of Fiber by Raman Spectroscopy (RS)

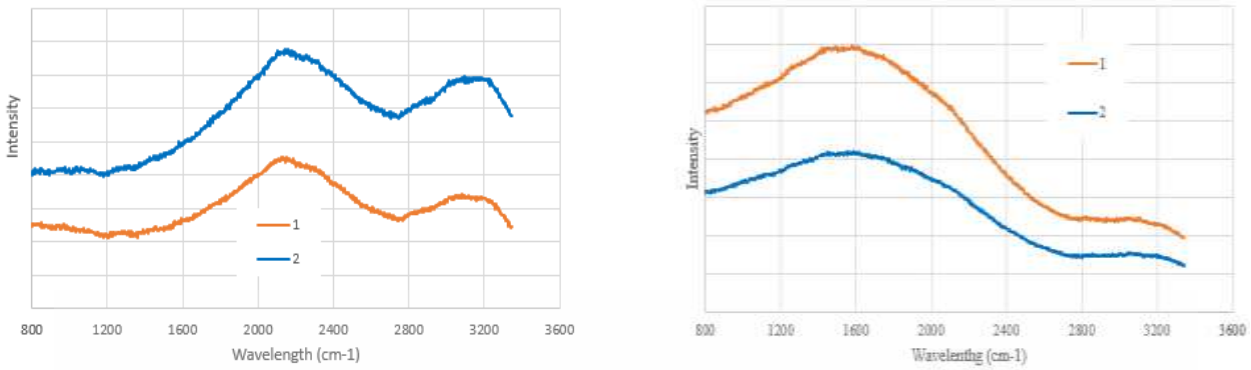
Raman spectroscopy is one of the useful tool to observe the fiber structure of fiber through D band and G band. The data of RS were analyze using original peak software. As the result, there are two peak in both of carbon fibers because both of carbon fibers was constructed by carbon as shown in SEM/EDS data. In figure 4.8, two different location were detected by laser illumination to know the behavior of carbon structure. The graph of carbon fiber twill and plain weave show two different peak with different Raman spectrum as D (disorder) band representing of the defective crystal or incomplete hexagonal bonding atom and G (Graphite) band which is represent about the fiber with pure hexagonal structure of carbon-carbon bonding graphene were stacking parallel. For carbon fibers, D band is at around 1350 cm^{-1} and D band is at 1590cm^{-1} of Raman shift. It was agreed with the previous research result shown in chapter 1 literature reviews. In the case of glass fiber, it does not show any of specific peak and it indicate the amorphous structure in fibers.



(a) Raman Shift of Raw CFT



(b) Raman Shift of Raw CFP



(c) Raman Shift of Raw GFW (d) Raman Shift of Raw GFF

Figure 4.8 Raman Shift of (a) CFT, (b) CFP, (c) GFW and (c) GFF

The result of Raman shift result were analyze by original peak to investigate the intensity of D band and G band. The result for both of carbon fiber CFT and CFP were shown in table 4.3. The peak of D-band and G-band value are similar to other researcher. Therefore, this research confirm that the structure of fiber was come for 1000-1500ºC carbonization as commercial material.

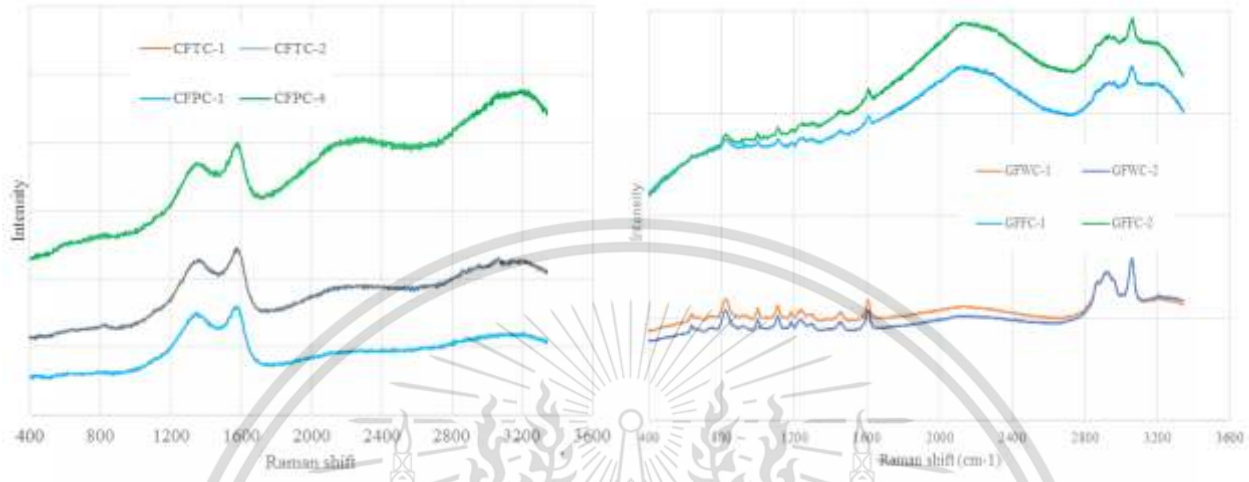
Table 4.3 Intensity peak in D band and G band of CFT and CFP

	Peak	Center of Peak	FWHM	I_D/I_G
CFT1	D - Band	1382.43	308.69	0.868472
	G - Band	1591.79	103.03	
CFT2	D - Band	1390.46	260.58	0.873726
	G - Band	1591.42	108.56	
CFP1	D - Band	1376.05	243.40	0.869422
	G - Band	1582.72	110.02	
CFP2	D - Band	1338.56	440.77	0.851065
	G - Band	1572.82	91.46	

The composite of both type carbon fiber and glass fiber were also check in Raman spectroscopy and different behavior are found RS. In Carbon fiber composite, there is still D band and G band but the intensity of them are not sharp as carbon fiber filament result. D band and G band value are still the same in Raman shift wavelength at 1380cm^{-1} for D band and around at 1590cm^{-1} for G band with broad peak. There is no specific peak for epoxy matrix but the epoxy is one of obstacle to detect the carbon fiber structure in Raman spectroscopy. The same broad peak are found in both carbon and glass fiber composite graph between 2000-2400cm^{-1} and 2800-3200cm^{-1}, it can assume that those peak are come from epoxy matrix. For glass fiber, several small peak are found in the range of 700-1700cm^{-1} Raman shift which are not found at the graph of raw fiber filament. The composite Raman shift graph are as shown in figure 4.9.

For both carbon and glass fiber filament, they have some peak at 2000-3000cm^{-1}, it comes from the epoxy that was coating on the surface of the fiber filament while manufacturing process [12]. For CFT, the intensity peak are higher than CFP because sizing coupling agent containing percent are different between two materials CFP and CFT as shown in figure 4.8 (a) and (b). The high intensity peak are also found on glass fiber both GFW and GFF due to coating coupling agent on the fiber surface and also the fiber itself

are amorphous as shown in figure 4.8 (c) and (d). Ferrari and Robertson [51] also studied about the resonant of Raman spectroscopy and it shown the polymeric CH or CH_2 stretching at raman shift 2000-3000cm^{-1}. The higher amorphous material are also found at the same raman shift.



(a) (b)

Figure 4.9 Raman shift of composite of (a) Carbon fiber and (b) Glass fiber

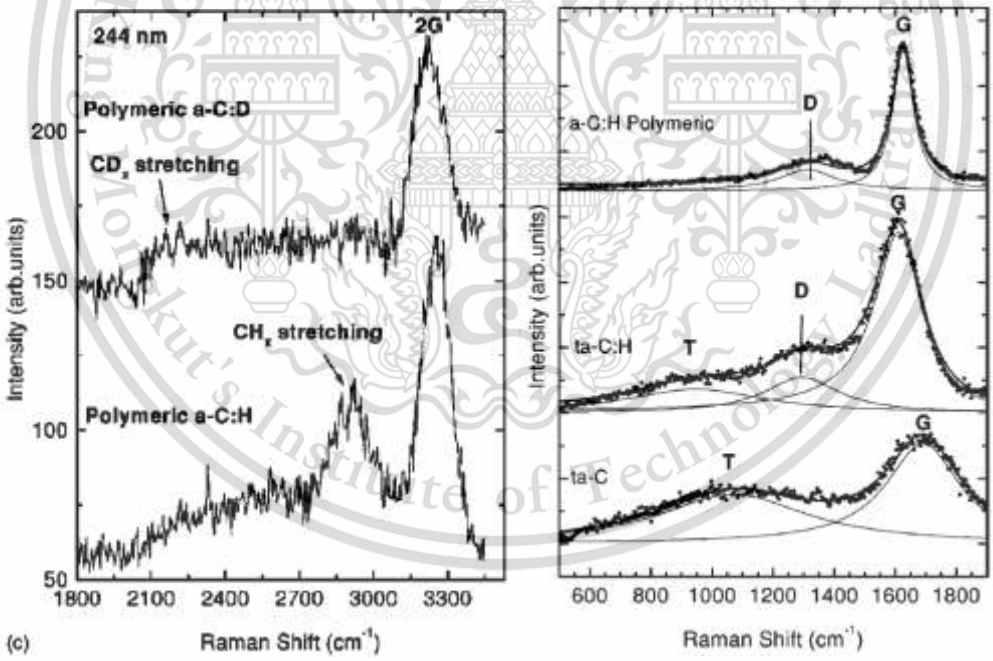


Figure 4.10 Disorder, Amorphous and diamond like carbon from Raman Spectroscopy [51]

In composite of carbon fiber, the intensity are higher than fiber filament due to most of the matrix are amorphous structure and it influent the intensity at 2000-3000cm^{-1}. The intensity peak are higher than D band and G band, it indicate the matrix behavior of composite as shown in figure 4.9 (a) and (b). The slope magnitude are slightly different between CFP and CFT at 2000-3000 cm^{-1} Raman shift.

4.1.8 Characteristic of Fiber by Fourier Transform Infrared (FT-IR)

The fabric of all fibers were examine bonding structure using FTIR. The bonding element in the fiber are reveal by Fourier transform infrared. Figure 4.11 shows the result of carbon fiber plain and twill weave. Both of fiber have similar element bonding, 1160-1350cm^{-1} band indicate acyl C-O bonding in the structure and C=C aromatic bonding at 1450-1650cm^{-1}, C≡N bonding at 1150-1250cm^{-1} are found in the carbon fiber. But it cannot show in EDS because the EDS detect only major and organic element in the fiber. Nitrogen may come from the synthesis of PAN fiber which did not remove fully in carbonization and it might be very a few that is not enough to detect by EDS. The main bonding are C=C aromatic and C=C due to more than 95% are carbon contains in carbon fiber which was investigated by SEM/EDS.

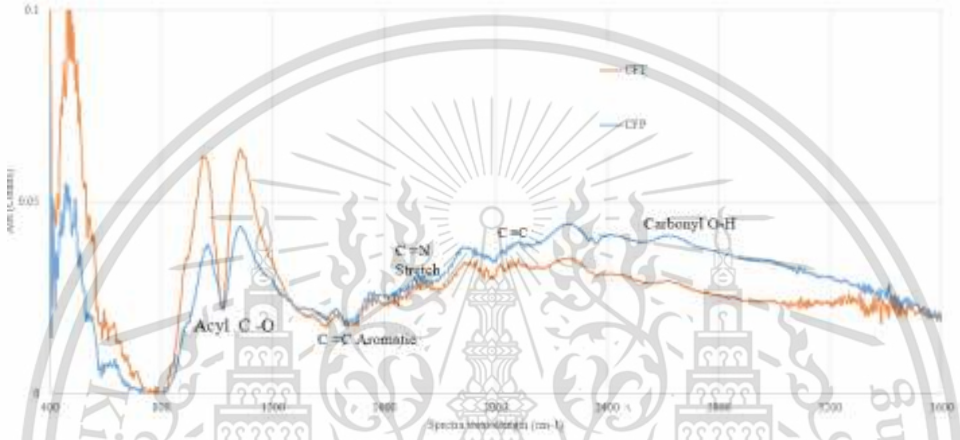


Figure 4.11 FTIR graph of carbon fiber twill and plain weave

Figure 4.12 FTIR data of glass fiber woven and fabric

C=C aromatic and C=C stretch might come from inside of the fiber because the main structure of carbon fiber are C-C bonding structure which was shown in TEM, RS and XRD result. There are some functional group of C=N stretch, N-H and carbonyl O-H group from both carbon fiber and glass fiber as shown in figure 4.11 and 4.12. Those are from carbon and glass fiber itself which are still remain from carbonization stage of fiber and epoxy coupling agent. Therefore, the main C=N or O-H bonding will come from the polymer or coating epoxy of fiber surface while the manufacturing are performed. But these are the main functional group of fiber which can make the main adhesive bonding between fiber and matrix.

Glass fiber filament are also detected the bonding element in FTIR which was shown in figure 4.13. The main element of glass fiber was silica (SiO_2) and the graph show the main stretching element of Si-O-Si band at 900-1100cm^{-1}. Epoxy resin behavior are found at band 1240-1183cm^{-1}, it may come from the coupling agent which was coated on the fiber surface for better handling. C=O stretching are also found at band 1700cm^{-1} as the main character element in glass fiber. In glass fiber, all of the element cannot detect by FT-IR because FTIR can detect especially organic material. Even though Mg and Ca are found in glass fiber by EDS, FT-IR did not shown in graph.

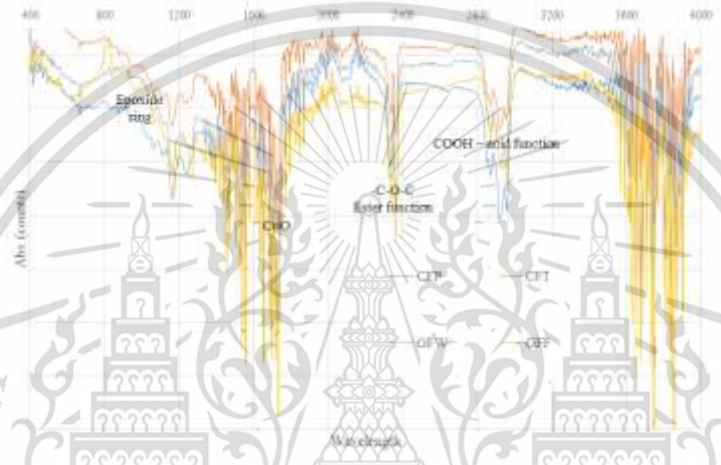
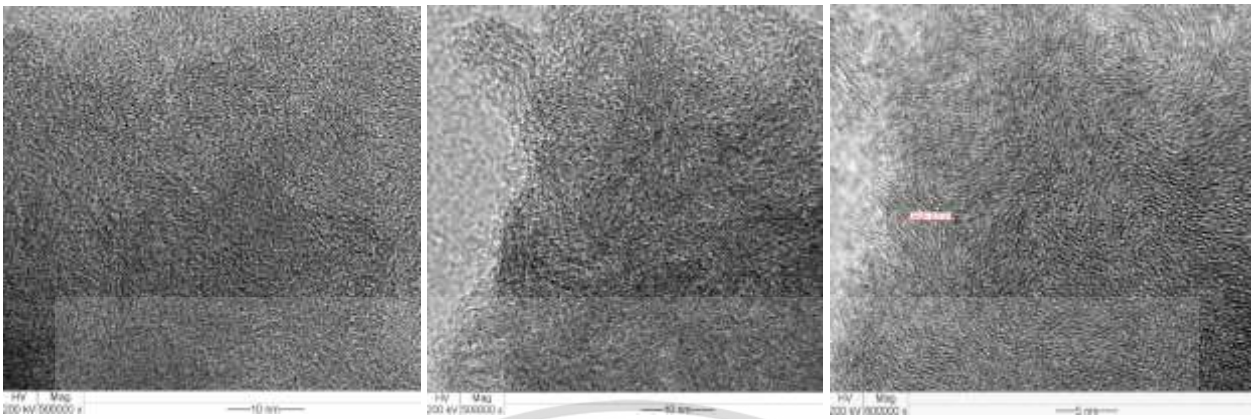


Figure 4.13 FTIR-ART data of composite of carbon fiber and glass fiber

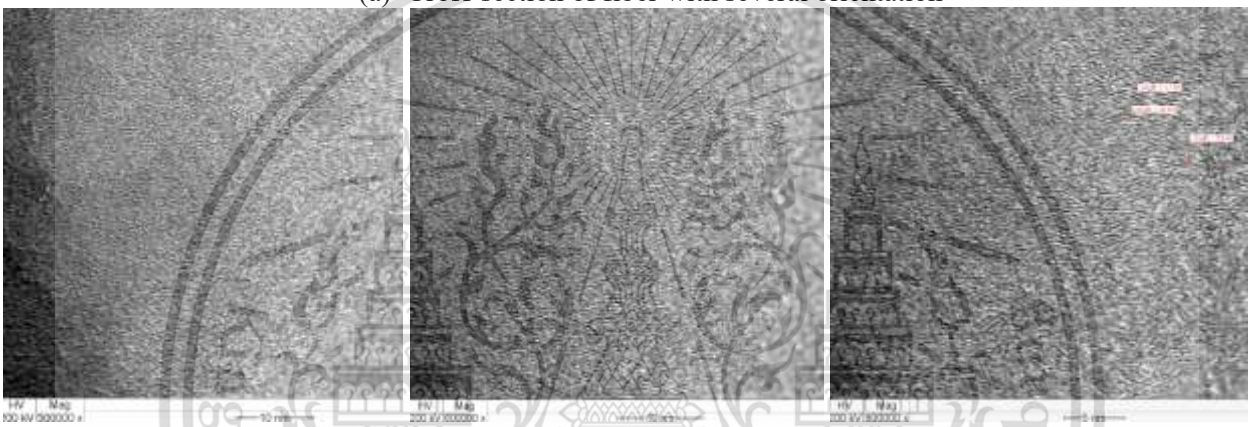
FTIR with ART was used to detect the behavior of carbon fiber and glass fiber composite and it shown main character of epoxy matrix as shown in figure 4.12. The behavior of carbon fiber and glass fiber was influence by epoxy matrix. Epoxy matrix ring was observe at 915cm^{-1} and C=O carbonyl group was found at 1732cm^{-1}. Ester functional group (-C-O-C-) was observe between1150-1380cm^{-1} and COOH acid functional group also observe at 2800-3060cm^{-1}. All of these functional group are found in all graph of CFP, CFT, GFW and GFF composite. And most of them might come from epoxy matrix.

4.1.9 Transmission Electron Microscopy (TEM) Analysis of Fiber

Nano structure of material are very important for mechanical strength especially in composite. TEM is one of the main tool that can reveal the behavior and the structure of material in nano scale. The filament of carbon fiber and glass fiber were cut by microtome diamond knife to be 100nm^2 to investigate the interior structure of carbon and glass fiber. 200kV transmission electron microscopy was used in this research. Nano structure of Carbon fiber twill (CFT), plain weave (CFP) and glass fiber woven (GFW) were investigated by TEM and all of result are as shown in figures. In CFT fiber filament, there are two type of oriented fiber in cross section and fiber axis although the fiber were cut randomly as shown in figure 4.14.The current result was agree with the result of Jean-Pierre [46].



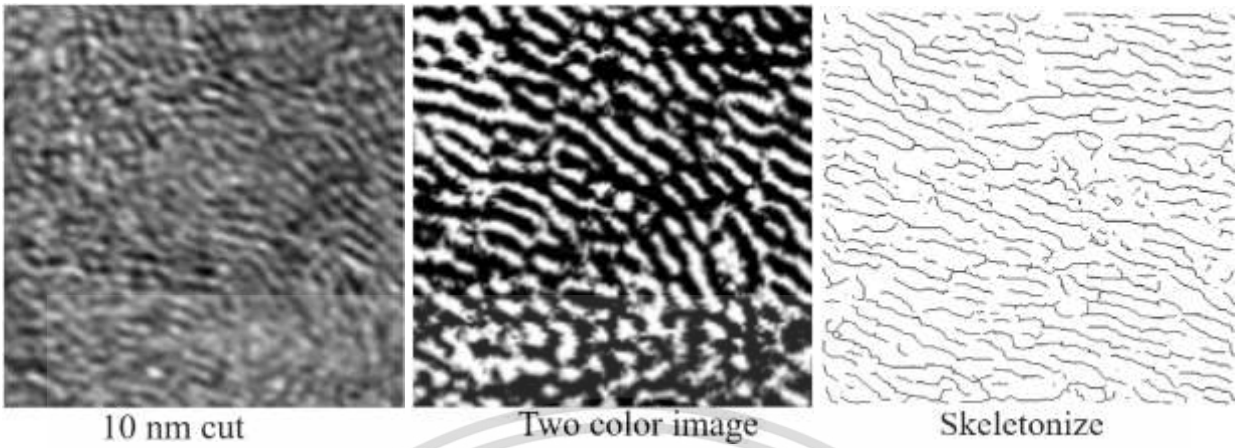
(a) Cross section of fiber with several orientation



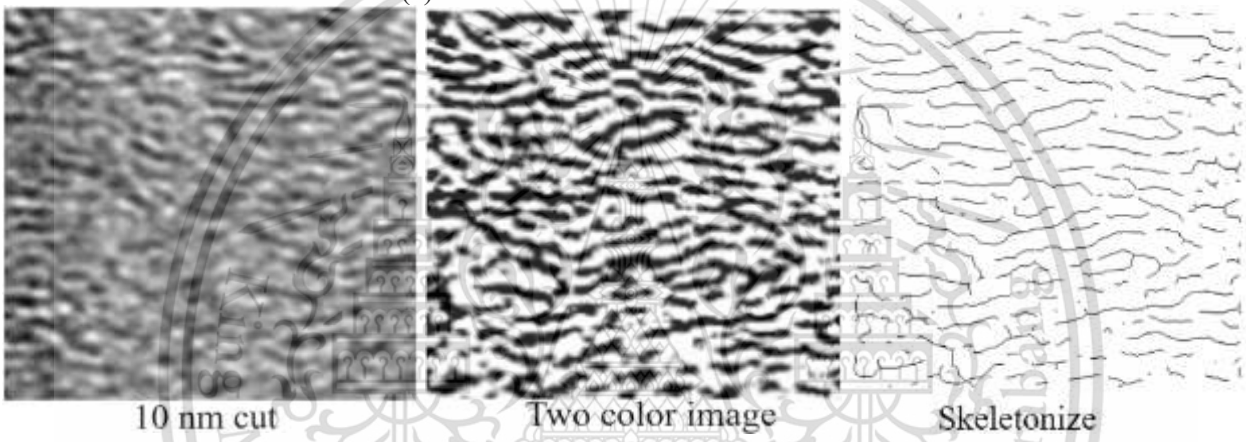
(b) Fiber axis with only one direction

Figure 4.14 TEM image of CFT (a) cross section of fiber and (b) fiber axis direction

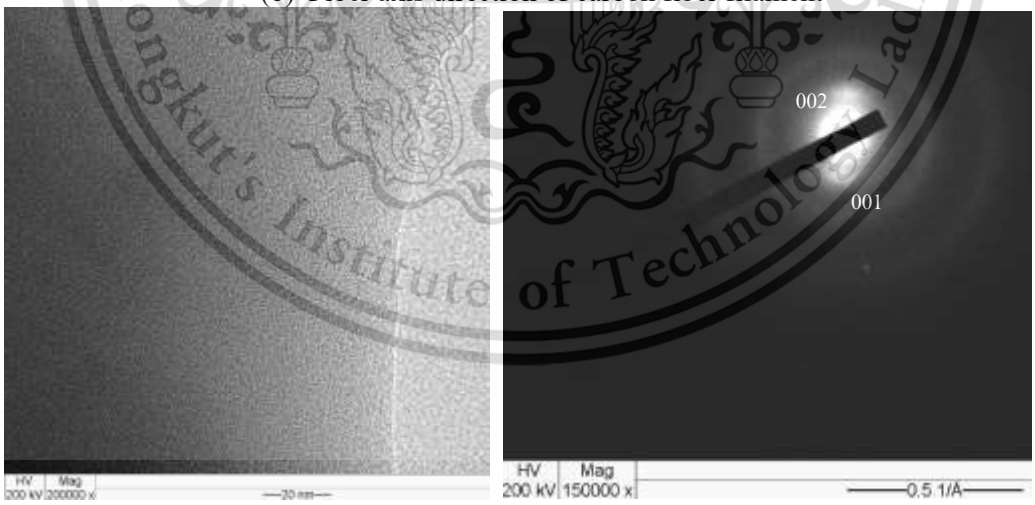
In CFT fibers, the orientation of fringe are mixing and oriented in different direction especially in the middle of fiber. The crystallite structure and some cluster fringe yarn are connected in different way but they are connect each other to hold each crystallite graphite structure. Amorphous cluster is one of the connecting bridge between graphite crystal structures. On the other hand, it may be one of the weakness in fiber for mechanical properties. When random orientation was stretch out, the cluster part of the structure will be the main parts of the structure that can break easily due to it weakness in bonding. On the other hand, the structure of crystallite was laid to the direction of fiber axis, but they are not very long crystalline size. Carbon fiber are constructed with many graphite and cluster connecting like turbostratic structure.



(a) Cross section of Carbon fiber filament



(b) Fiber axis direction of carbon fiber filament

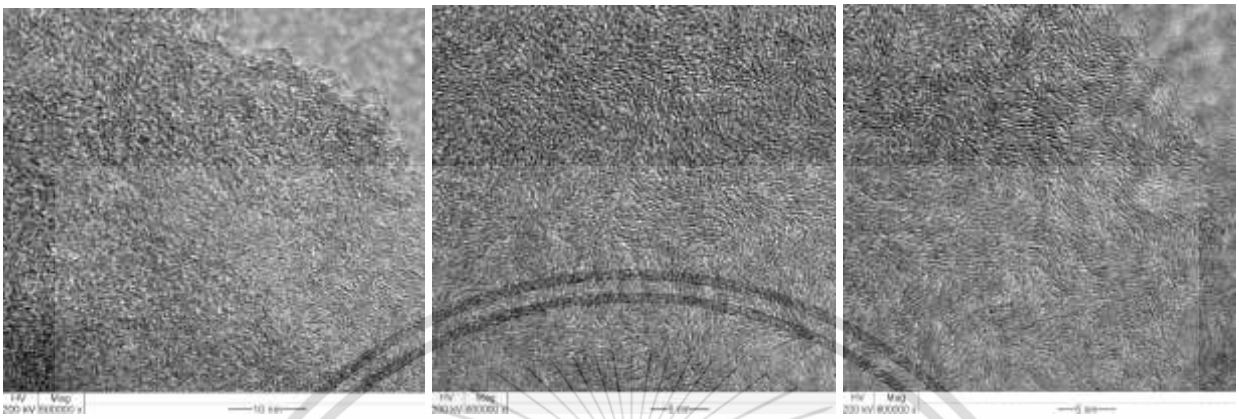


(c) TEM-XRD of carbon fiber filament

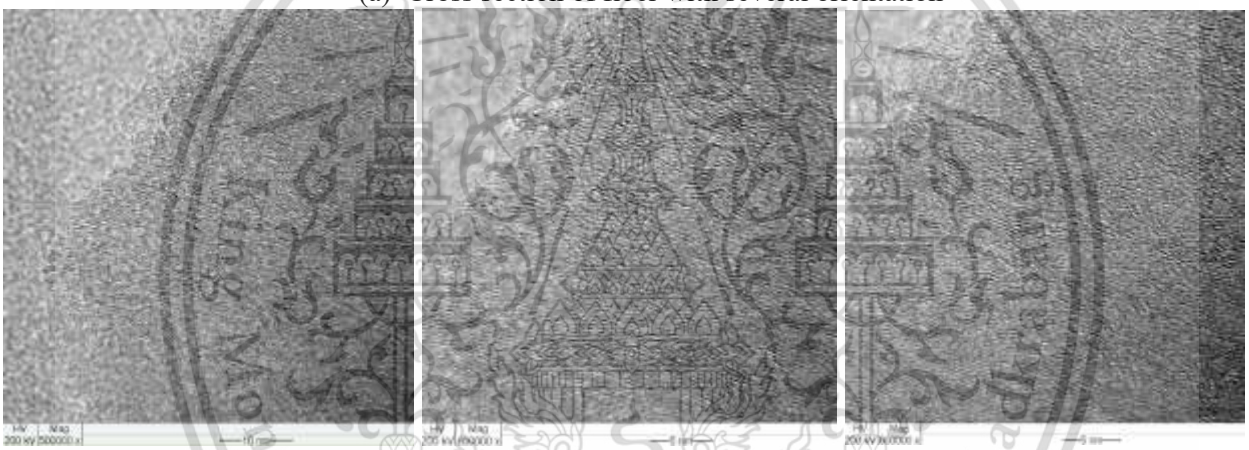
Figure 4.15 TEM image crystal of carbon fiber twill (CFT) weave filament

The structure and behavior of both carbon fiber CFT and CFP are similar that show graphite structure crystallite with mixing of cluster like turbostructure in the whole fiber filament. Figure 4.15 shows the structure of cross section and the fiber axis direction fiber that was analyze by image J to observe the

clear graphene. The graphite crystalline are laid in the same spacing height with different amount of crystallite fringe. TEM diffraction also approve that the peak of graphite at 002 plane and 001 of structure that was agree with XRD result.



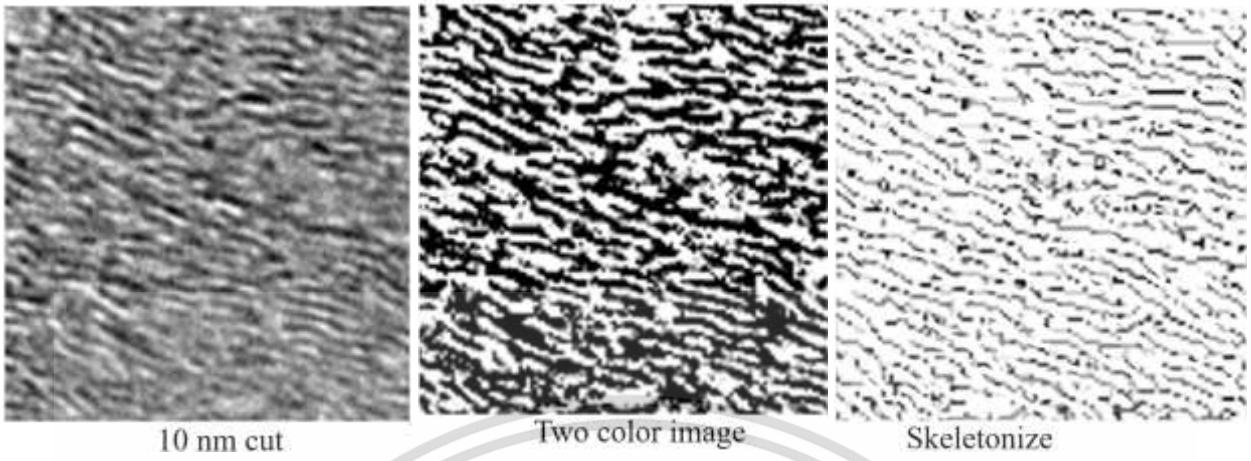
(a) Cross section of fiber with several orientation



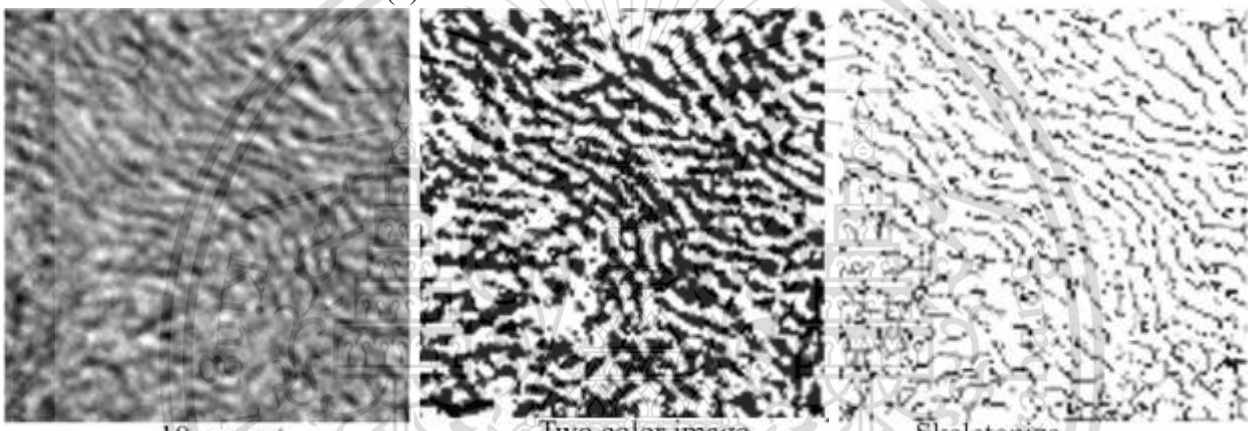
(b) Fiber axis with only one direction

Figure 4.16 TEM image of CFP (a) cross section of fiber and (b) fiber axis direction

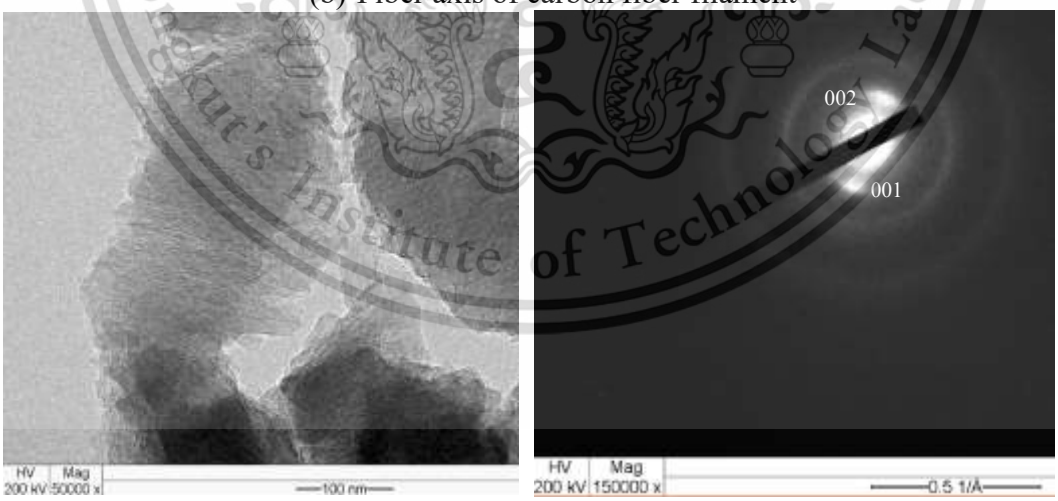
As mention above, the structure of carbon fiber are similar, only the spacing between each layer graphene are different where 0.35nm for CFT and 0.36nm for CFP. Spacing of parallel stacking graphene of CFT is smaller than that of CFP. Therefore, more graphene stacking layer will be construct and it will be more compatible for CFT. The compactible of graphite will create higher bonding strength between graphene that can give higher mechanical properties in both fiber and composite. Multiple orientation fringe of graphene layers in the cross section of fiber are found in figure 4.16(a). And figure 4.16(b) is assume that fiber axis direction. The graphite orientation is parallel to the fiber direction especially around the surface of fiber. The make the fiber surface unsmooth with various valley pits. As an advantage, it make the better mechanical interlocking between fiber and matrix in composite. Image J analysis picture of CFP show the clear paths of grahene laid in the same direction of fiber axis on the fiber surface. The diffraction pattern of the plain weave carbon fiber are also the same CFT as shown in figure 4.17(c).



(a) Cross section of carbon fiber filament



(b) Fiber axis of carbon fiber filament

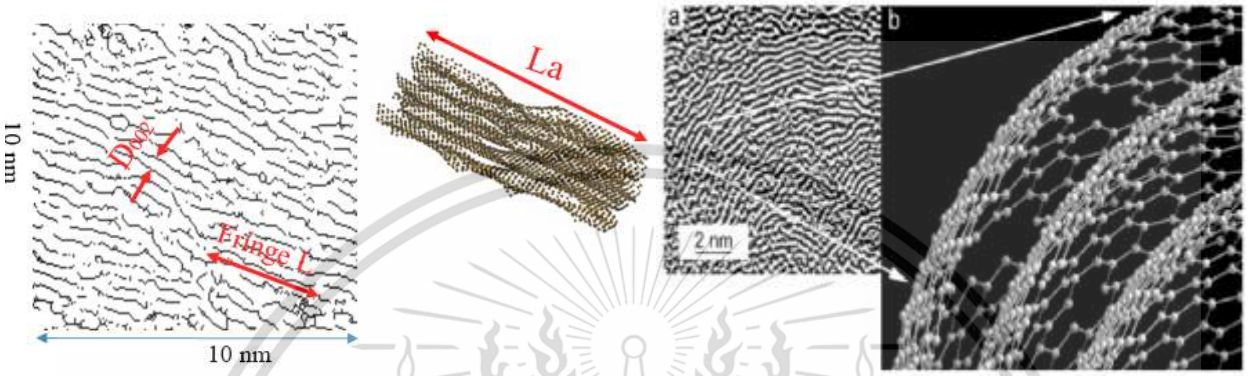


(c) TEM-XRD of carbon fiber filament

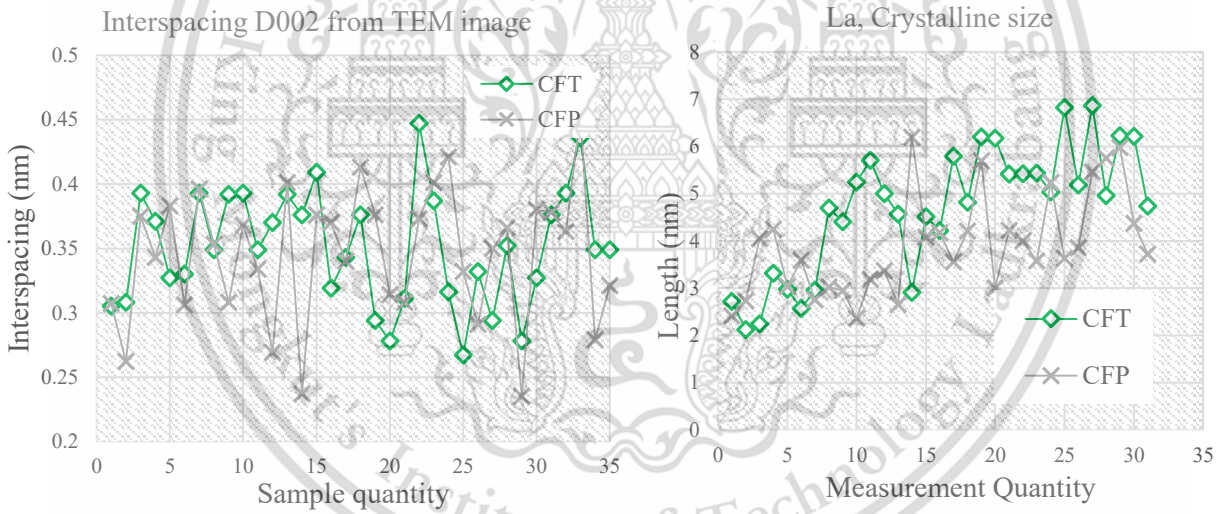
Figure 4. 17 TEM image of carbon fiber plain (CFP) weave

Skeletonize TEM image of carbon fiber is one the simplest image which can determine the structure of fiber and the orientation of fringe. Karolina [54] interpreted TEM image of carbon fiber to be 3D image of the graphene that was shown in figure 4.18 (a) and (b). TEM image of carbon fiber is analyze

by image J to measure the interspacing (D_{002}) and the crystal size (L_a) of nano structure of 10nm square skeletonize image as shown in figure 4.18(c) and (d). Skeletonize image of carbon fiber show several different fringe orientation in a 10nm square area. The graphite crystal or structure cannot see in TEM image but the result of XRD data can be compare with it



(a) Skeletonize image of carbon fiber (b) TEM to 3D image [54]



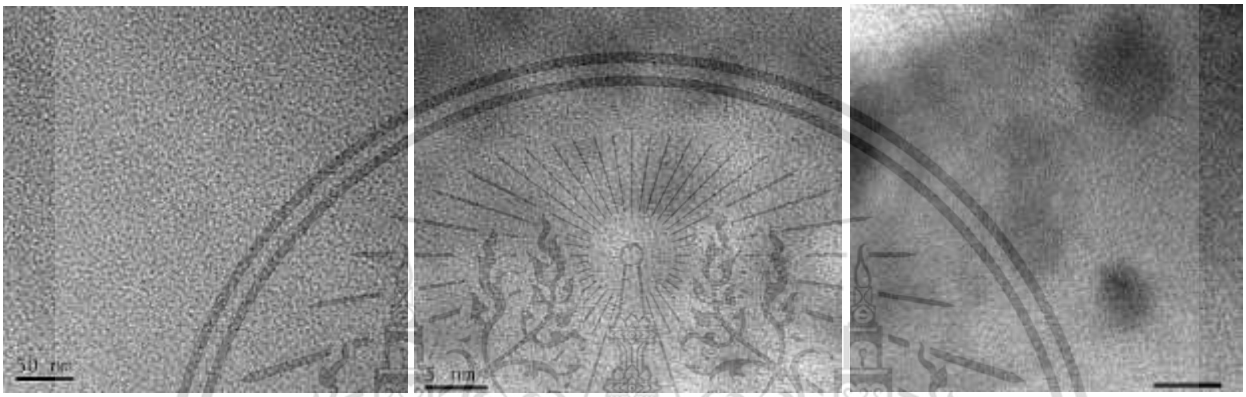
(c) Interspacing measurement from TEM image (d) Crystalline size measurement

Figure 4.18 TEM image analysis of carbon fiber and literature of TEM image analysis

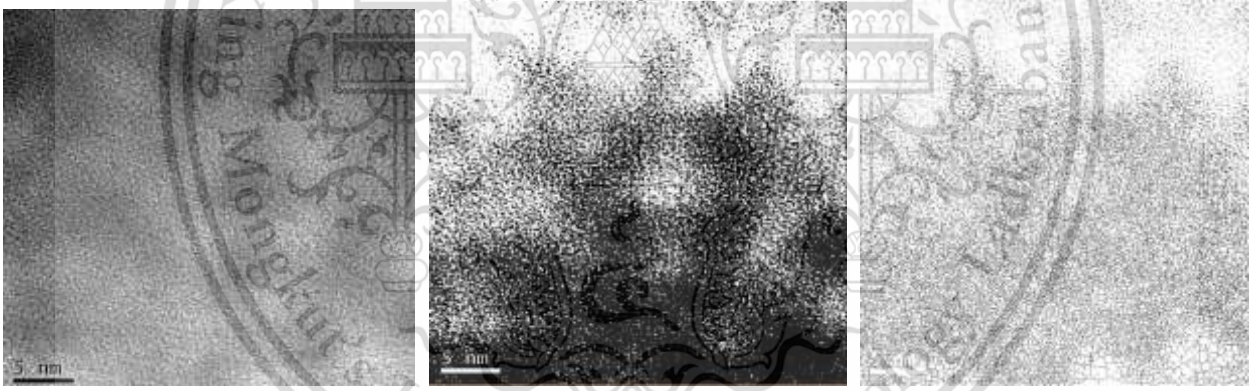
The measurement data of CFP TEM skeletonize give a result of interspacing around 0.347nm in average and it was a few different from XRD result with 0.36nm. But the result of CFP is almost similar to the literature of XRD result. Interspacing of CFT from TEM result are also smaller than XRD result but it is similar to the literature result of XRD. Crystallite size (La) from TEM image is much smaller than XRD result of carbon fiber. Some of the researcher also found the similar result of XRD. TEM image is one the useful tool to analyze the structure of carbon fiber and glass fiber in nano crystal. TEM image result of D002 and La are shown in table 4.4 with compares of XRD, TEM and some other literature review.

Table 4.4 Crystalline size, interspacing and stacking height of TEM image analysis of Carbon fiber

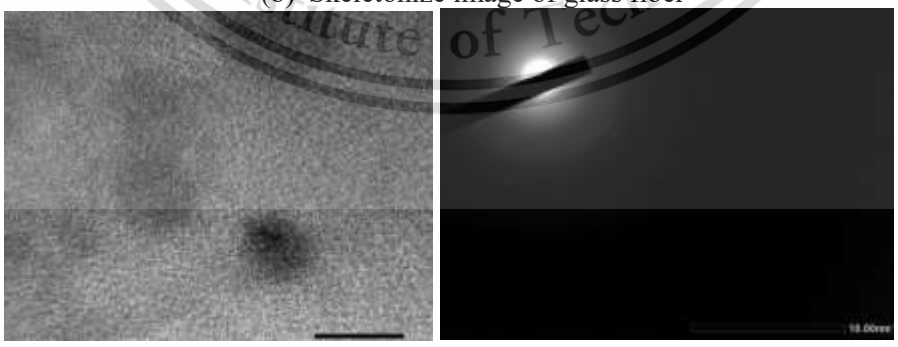
Sample	Current Experiment					Literature (XRD)			
	XRD			TEM		Dong Feng Li (2007)		Peter (2005)	
	La	Lc	D002	La	D002	La	D002	La	D002
	(nm)	(nm)	(nm)	(nm)	(nm)	(nm)	(nm)	(nm)	(nm)
CFP	6.98	11.13	0.36	3.91	0.347	4.37	0.357	9.8	0.34
CFT	7.67	9.32	0.35	4.69	0.348	14.7	0.345	4.8	0.345



(a) TEM image of Glass fiber



(b) Skeletonize image of glass fiber



(c) Diffraction pattern of glass fiber

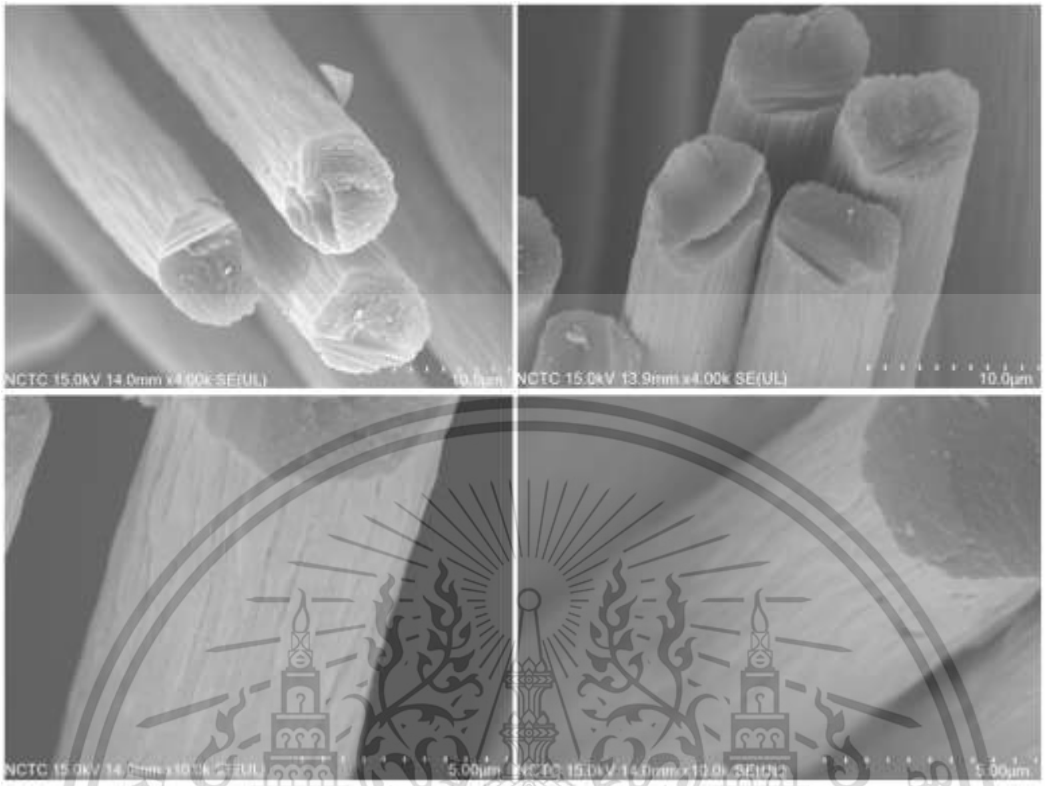
Figure 4.19 TEM image of glass fiber

On the other hand, structure of glass fiber are absolutely different from carbon fiber because glass fiber do not have crystallite structure. The result of glass fiber are agree with the previous research result with non-crystallite solid. The structure of glass fiber are investigated as amorphous in all of the result, there is noncrystallite structure inside of the fiber. All of the element inside fiber are bonded randomly and skeletonize image of glass fiber show that there might be the grain structure in the glass fiber. But it needs more research to confirm and compare to other result. The diffraction pattern of glass fiber also refer to the non-crystallite structure of glass fiber.

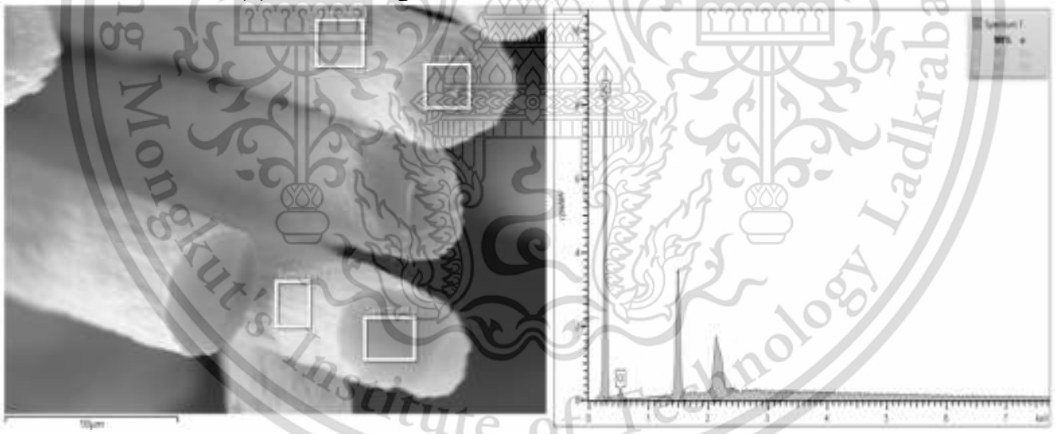
4.1.10 Treatment Fiber

Most of the fibers were produced by wet spinning method and then the fiber surface were coated by coupling agent or sizing epoxy to protect the fiber surface after the spinning fiber were treated. Most of coating layers are coupling agent or epoxy that can fill up the pitting valley on the fiber surface, therefore the mechanical interlocking between fiber and matrix would lost and that will bring weakness of interfacial bonding. The weakness of interfacial bonding between fibers and matrix will make the lower mechanical properties. Therefore, treatment fiber method was used in this research to remove all of the coating layer of fiber surface. Treatment fiber will dig back to the original pitting fiber surface, and that will make mechanical interlocking to improve the mechanical properties of composite.

In this research, the highest mechanical properties fiber CFT and GFW are choose to make a treatment on the fiber surface. In carbon fiber, the morphology of fiber filament is not change much but one of the important changes is the roughness of surface. Before treated, fiber surface are much rougher and the pitting are deeper. Even though, fiber surface roughness are not measure by machine like Atomic Force Microscope (AFM), SEM image give the information about the surface of fiber. Reducing roughness on the surface of fiber may make bonding weakness that can drop the mechanical properties. The weight reducing between before and after fiber treatment are 3% for carbon fiber because of the literature shows 0.5-6% of coupling agent are coated into fiber surface to prevent the damage or handling process.



(a) SEM image of treatment carbon fiber twill weave



(b) SEM energy dispersion spectroscopy of treatment carbon fiber (TCFT)

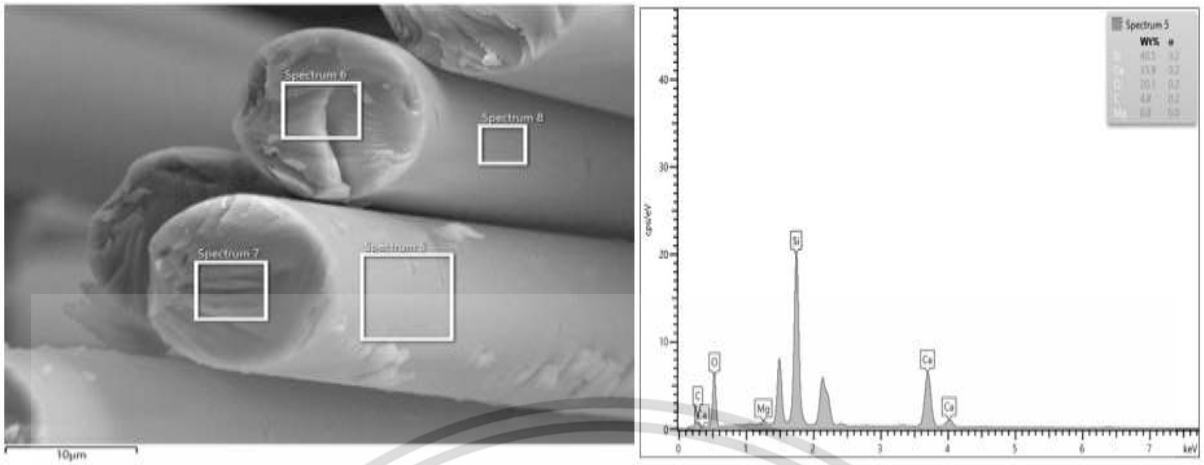
Figure 4.20 morphology of treated fiber of (a) carbon fiber and (b) its chemical composition by EDS

For glass fiber treatment, the weight reduce 3% after treatment and it is not effect to fiber diameter. Contrast with carbon fiber, the surface of glass fiber are smoother than before and the weight reduce only 1%. All of the particle are remove from the fiber surface and remove all of the defects from the fiber surface. It is good point of treated fiber surface to interact with matrix especially in chemical bonding. Mechanical interlocking between epoxy and fiber surface are not possible due to its smooth surface therefore chemical bonding is one of the important case in glass fiber. The surface morphology after treated fiber was shown in figure 4.20. And the chemical composition are not change in the fiber but carbon (C) and calcium (Ca)

are obviously lower percentage at the cross section area in spectrum 6 and 7. It can assume that most C and Ca are located on the surface of fiber. All of the chemical composition are summarize in table 4.5.

The treatment fiber are used to make a composite to the previous composite fabricated process. The specimens are prepared by CNC based on respective ASTM method, after that, the specimens are cured at 80ºC for 6hrs and 120ºC for 3hrs additionally after the composite is cured at room temperature until its dry. The morphology of treated CFT and GFF fiber filament was investigated by SEM and EDS to observe the changes of fiber surface and chemical changes. After treatment fiber, the weight are reduce 3% of CFT due to the particle from surface are remove all and make more pitting valley on the fiber surface. The fiber diameter and chemical composition of element are not change significantly. Pitting valley along the surface of fiber make mechanical interlocking with matrix. The treated fiber are as shown in figure 4.20. Four area of fiber surface and cross section are used to determine chemical composition as shown in table 4.5 for summary. Chemical composition at surface or cross section are same.

(a) SEM image of treatment glass fiber woven



(b) SEM energy dispersion spectroscopy of treatment glass fiber (TGFW)

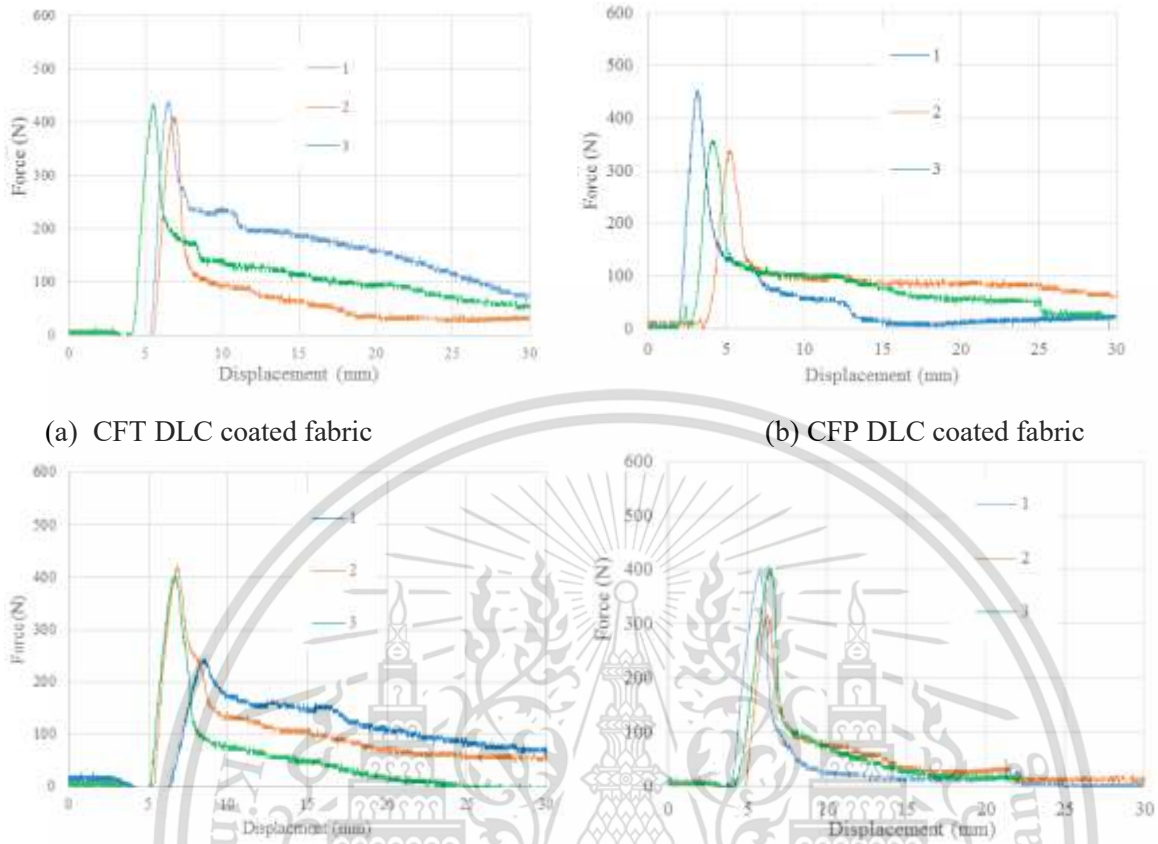
Figure 4.21 Morphology of treated fiber of (a) glass fiber (GFW) and (b) chemical composition by EDS

Table 4.5 Treatment Fiber Chemical Composition of Carbon and Glass Fiber

Element / Composite	Spectrum	Area	C (Wt. %)	O (Wt. %)	Si (Wt. %)	Ca (Wt. %)	Mg (Wt. %)
TCFT	1	Fiber Surface	95.5	4.5	-	-	-
TCFT	2	Fiber Surface	92.3	7.7	-	-	-
TCFT	3	Cross section	96.7	3.3	-	-	-
TCFT	4	Cross section	94.1	5.9	-	-	-
TGFW	5	Fiber Surface	4.8	20.1	40.5	33.9	0.8
TGFW	6	Cross section	1.1	27	43.5	27.5	0.9
TGFW	7	Cross section	1	23.9	45.2	29	0.9
TGFW	8	Fiber Surface	6.2	20.9	21.4	50.9	0.6

4.1.11 Diamond like Carbon (DLC) Coated on Treated Fiber

The treated fiber were coated with diamond like carbon (DLC) on the fabrics that was describe in section 3.1.13. The coated fiber were cut the same size of fabric which is mentioned in section 3.1.9 tensile test. The prepared specimen were tested in tensile test to observe the properties of DLC coated fabric. Fabric was cover by tape at both side not to freak out the pattern of fabric and the tape result was deducted from the total result of fabric. Force and displacement curve were shown in figure 4.22.



(a) CFT DLC coated fabric (b) CFP DLC coated fabric

(c) GFW DLC coated fabric (d) GFF DLC coated fabric

Figure 4.22 Diamond like carbon coated fabric result of (a) CFT, (b) CFP, (c) GFW and (d) GFF

Mechanical properties of DLC coated fiber was reduce obviously around 36% strength for carbon fiber and 3% change for glass fiber. The weight are reduce 3% for carbon fiber and 1% for glass fiber while the treatment are performed. The properties of DLC coated fiber are drop due to DLC plasma deposition is coated well especially on fiber surface while the fiber inside of fiber pattern are not coated well. It may affect the fabric properties to drop. If the deposited DLC the fabric was fabricated composite, it may give the high strength more than normal due to the deposited was become a particle stick on the fiber surface, it will help mechanical locking between fiber and matrix. Table 4.6 show the properties summary of naked GF/ CF and DLC coated GF/CF. The summary graph of naked fiber and DLC coated fiber was also shown in figure 4.23.

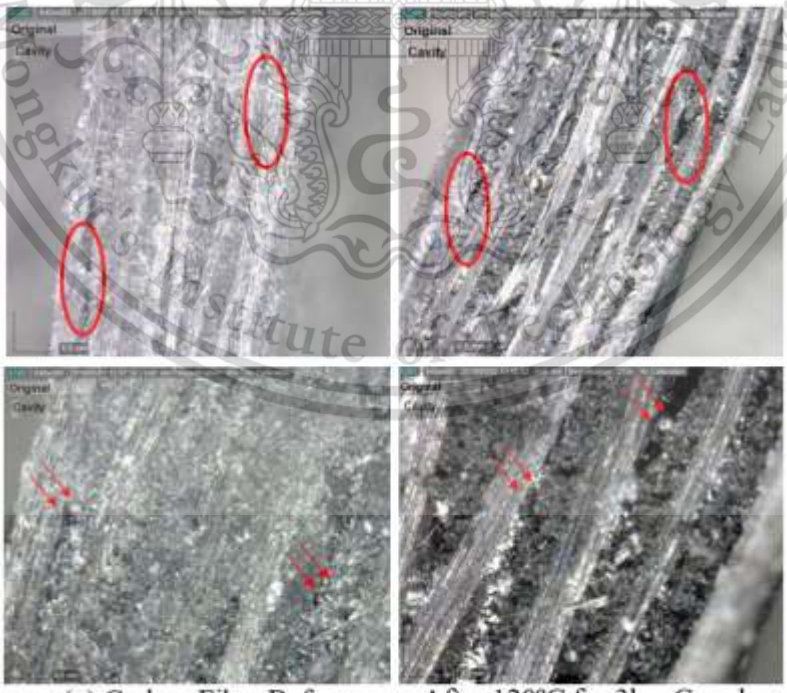
Table 4.6 Naked Fiber and DLC Coated Fiber Changes

	Force (N)	Displacement (mm)	Changes (%)
CF	440.87	4.05	
GF	271.1147	5.03	
DLC - CF	280.78	6.5	36.31
DLC - GF	261.715	6.266	3.47

Figure 4.23 Comparison of tensile test result of naked fiber and DLC coated fiber

4.2 Heat Treatment Analysis

In composite field, temperature is very important to change the mechanical properties or physical properties of composite. Especially in epoxy, it have glass transition temperature that can keep to make the balance curing or melting to the fiber. Before testing the mechanical effect on the composite, the physical changes on the composite was analyze using optical microscope in this current research. The small specimens were prepared by electric band saw cutter and analyze in OM. After analyze the composite by OM, the specimens were cured at 80ºC for 6hrs and another specimens were cured at 120ºC for 3hrs. After post cured at oven, the composite were analyze again by OM as shown in figure 4.24.



(a) Carbon Fiber Before After 120ºC for 3hrs Cured

(b) Glass Fiber Before After 120ºC for 3hrs Cured

Figure 4.24 Analysis of carbon fiber and glass fiber before cured and after cured at 120ºC for 3hrs

The composite of carbon fiber and glass fiber was analyzed by OM, it was found that carbon fiber composite are shrink as much as possible due to the high temperature after the liquid and moisture of the composite was removed. The cross link of epoxy matrix increase and it seems the fiber pull the epoxy to their side, therefore the middle of composite create small voids. On the other hand, even though the cross link increase make the higher strength, the more hole or small porous are created because of cross link. It may create crack initiating and crack propagate around matrix while the mechanical test in the composite. For glass fiber, the obvious changes are not found due to the color of composite. But the joining area of woven fiber bundles create big hole after cured for 3hrs at 120ºC and the color of composite was changed compare to before cured sample. The color changes to yellowish is also one of the sign of degradation of the composite.

4.3 Nano Indentation of Composite

All of composite with different type are test in nano indentation test with the spindle diameter of 10μm. Especially, nano indentation is to investigate the hardness of composite and the modulus of composite with the changes of cured temperature. 10mm^2 composite were prepared by band saw (electric saw) and test 5 times in different location. Only three result are used after excluded the maximum and minimum result. Two condition were test in this result such as the composite cured at room temperature (RT at 30ºC) and composite cured at 80ºC for 6hrs. The spindle sacra of composite are as shown in figure 4.25 at both temperature cured.

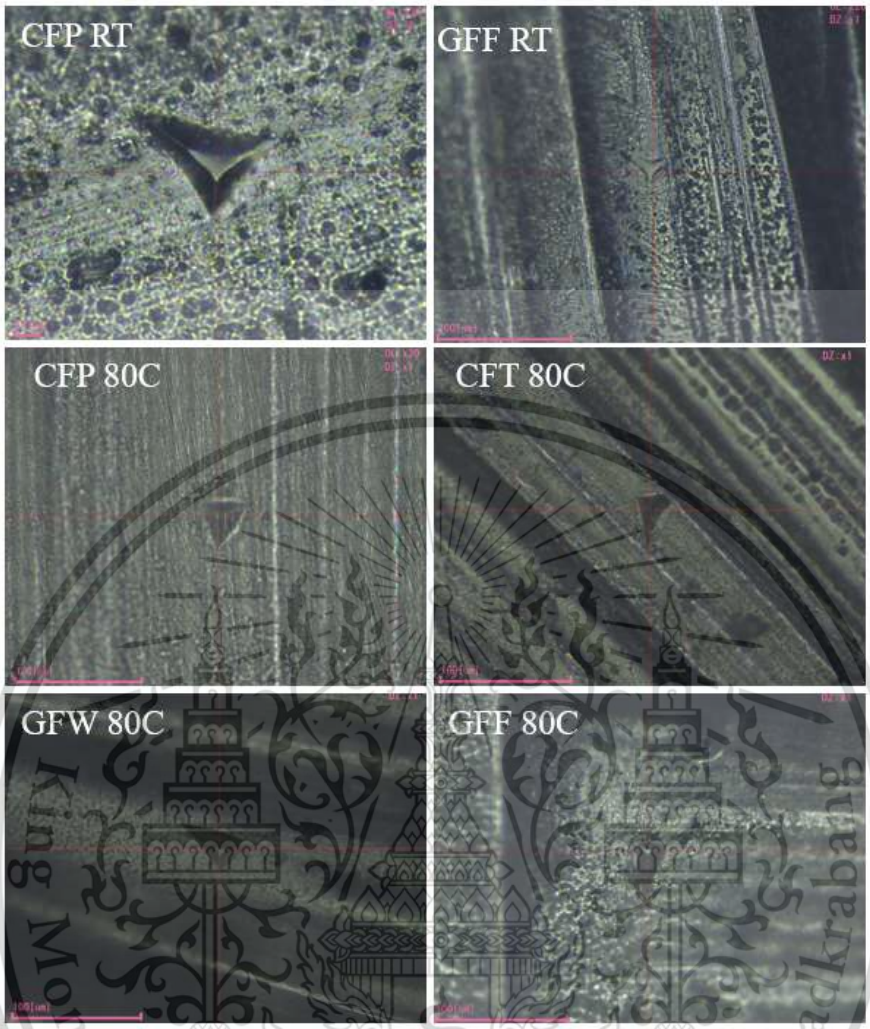


Figure 4.25 Nano indentation spindle dent area

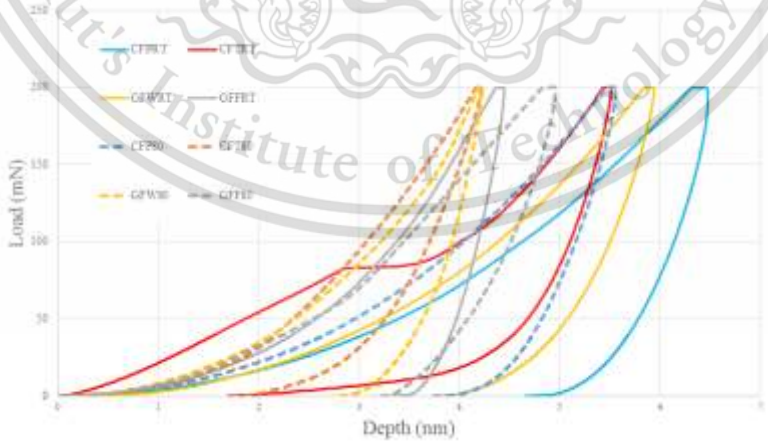


Figure 4.26 Nano Indentation graph for CFP, CFT, GFF and GFW cured at 30ºC and 80ºC for 6hrs

The nano indentation result show in figure 4.26 with solid line are cured at room temperature (RT is at 30ºC) and dot dash lines are composite cured at 80ºC for 6hrs. At 30ºC cured result, GFF have the

lowest depth at 4.3μm where CFP30 is the deepest depth at 6.4μm. The depth might be due to the pattern of fiber woven. GFF is plain weave pattern and the filament bundle amount are much smaller, therefore composite was build up by 16 layers of fabric. It will make difficulties spindle to penetrate through the composite. For CFP30, the woven is plain weave and the layers was 12 layers fiber in composite and the filament bundle amount is smaller than CFT and GFW, it will be easier to penetrate through composite. Table 4.7 were the depth, hardness and modulus of all type fibers composite at both temperature parameter. CFT30 and GFW30 both were in the middle result due to they were similar in the layers of composite and the thickness of fibers.

Table 4.7 The hardness, depth and modulus of nanoindention composite result

Composite/ Temperature	30ºC				30ºC+80ºC for 6hrs			
	CFP	CFT	GFW	GFF	CFP	CFT	GFW	GFF
Hardness (MPa)	219.49	334.13	531.06	473.96	425.09	918.37	557.91	430.58
Young Modulus (GPa)	9.73	10.85	15.34	15.92	9.41	15.37	14.89	17.11
Max Force (mN)	200	200	200	200	200	200	200	200
Max depth (μm)	6.41	5.50	5.90	4.40	5.52	4.20	4.21	4.90

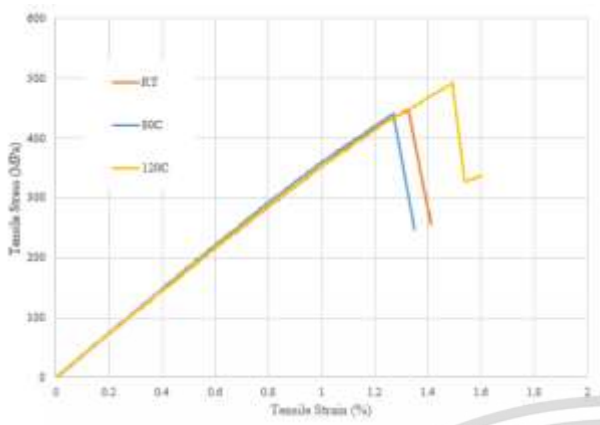
The composite result of 80ºC cured for 6hrs are shown in table 4.6 and figure 4.26 dot dash line graph. The graph show CFT80 and GFW80 result are almost similar and the displacement depth was decrease more than 1μm because the matrix (polymer) is increase crosslink at high temperature over the time passed. It will increase the polymer into high strength and become more brittle in composite. It makes the spindle more difficult to go through composite. Therefore, the depth is shorter than that of 30ºC cured specimens. Epoxy was increased in cross link that can make stronger epoxy while some epoxy loosen the cross link that may create porous or extend the existence porous size due to epoxies' shrinkage. For CFP80, the depth are decreased while GFF80 increase the depth with increase temperature. It can consider that the glass fiber composite are increase in ductility with the increase of curing temperature and time.

On the other hand, Cured at higher temperature make higher hardness and modulus in carbon fiber composite while glass fiber composite result does not change much with the increase of temperature. The main reason for higher hardness and modulus might be due to epoxy and cross linking make higher adhesive between carbon fiber and epoxy.

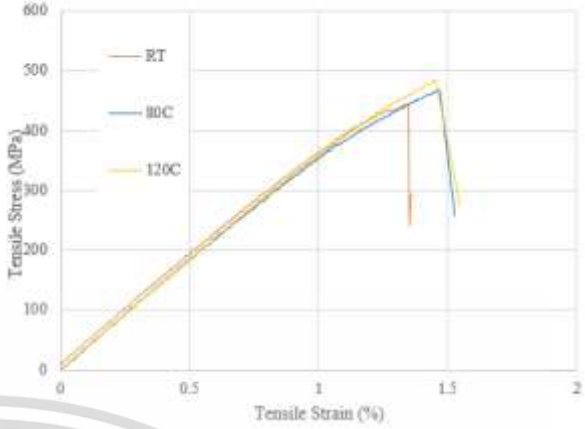
4.4 Tensile Properties of Composite

4.4.1 Tensile Properties of Normal Composite

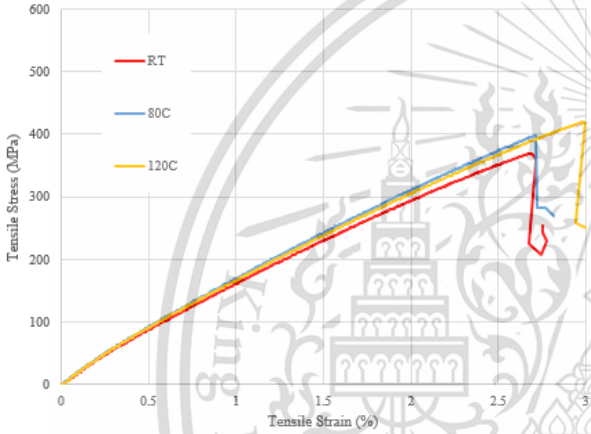
The composite were test cured at different temperature such ash 1) at 30ºC(RT) until it is dry, 2) 30ºC + 80ºC for 6hrs and 3) 30ºC + 120ºC for 3hrs. Six specimens are test in this research and excluded maximum and minimum. Figure 4.26 show all result of composite with three type of cured temperature. The result show that CFT and CFP is almost similar in 30ºC though the layers of composite are different as shown in chapter-3. CFT80 result are drop in strength while CFP80 and CFP120 result are increase rapidly. The result of CFP80 and CFP120 almost similar. Tensile strength of CFT120 is the highest result with around 500MPa in this graph. Tensile strain is increase with the increase of curing temperature. In carbon fiber, it can consider that CFT have the highest strength than CFP due to different woven pattern of fabric. For glass fiber, tensile strain are almost two times higher than that of carbon fiber composite tensile strain for both GFW and GFF. Higher tensile strain represent the higher ductility of composite that was found in glass fiber composite. It can assume that glass fiber composite have ductile behavior compare to carbon fiber.



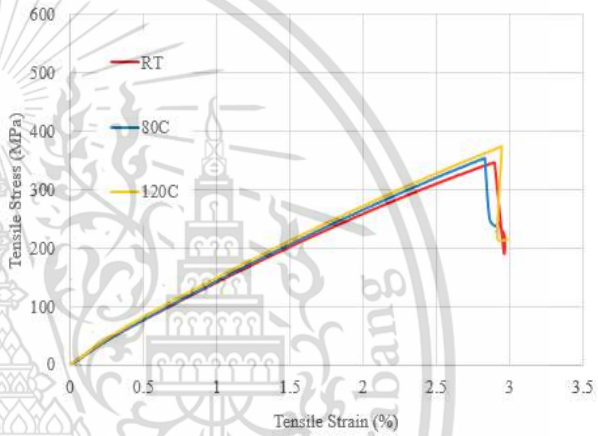
(a) CFT at all Temperature



(b) CFP at all Temperature

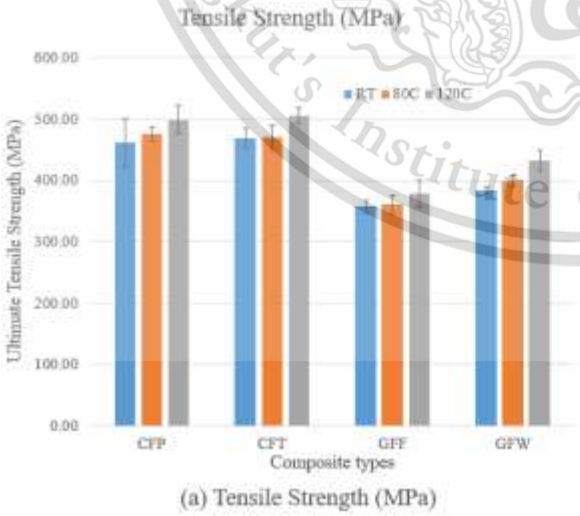


(b) GFW at all temperature

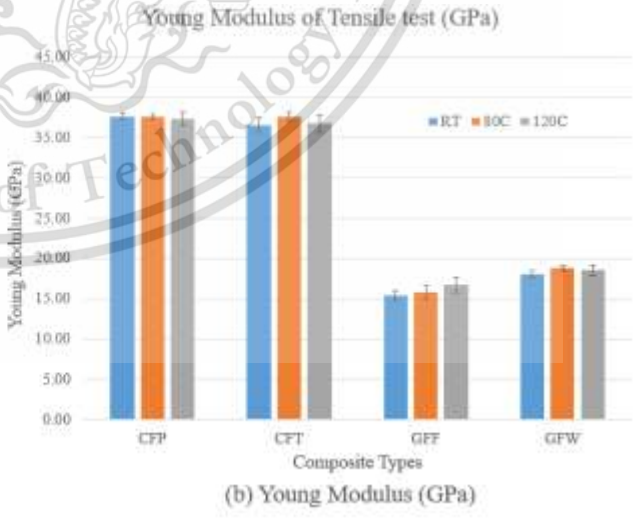


(d) GFF at all temperature

Figure 4.27 Tensile of composite at 30ºC, 80ºC and 120ºC for (a) CFT, (b) CFP, (c) GFW and (d) GFF



(a) Tensile Strength (MPa)



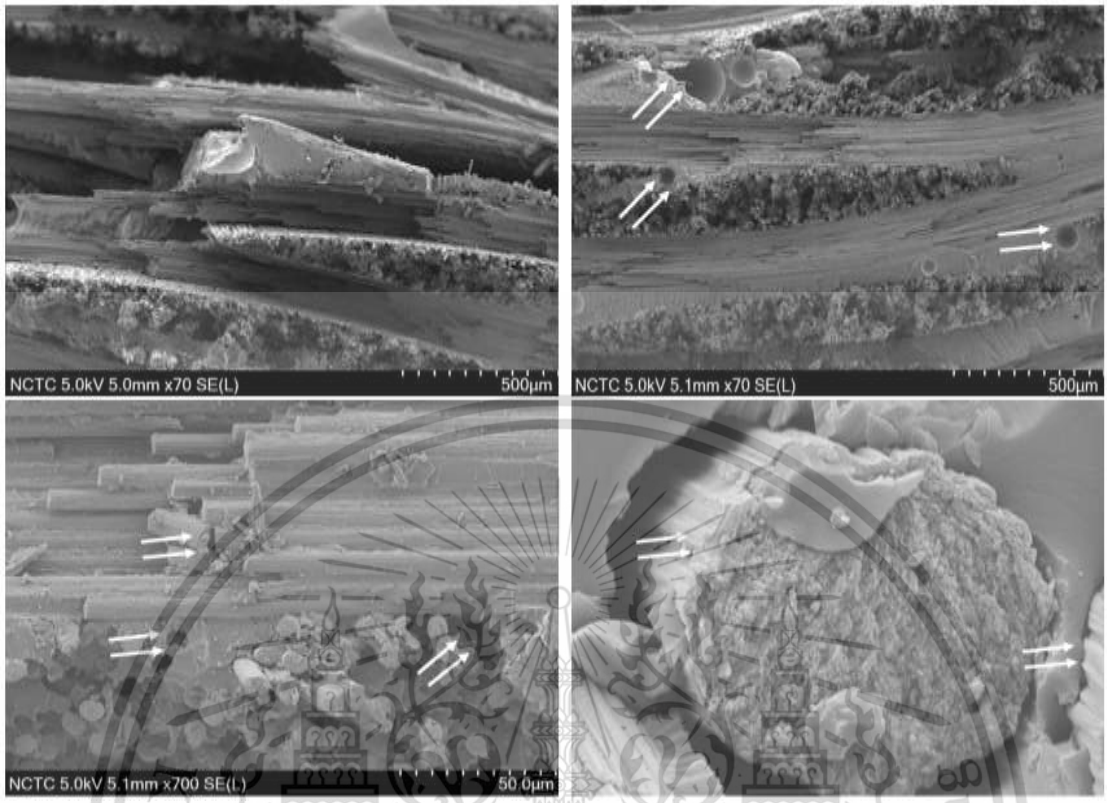
(b) Young Modulus (GPa)

Figure 4.28 Tensile strength and modulus of all of composite type at all temperature

4.4.2 Fracture Mechanism of Normal Composite

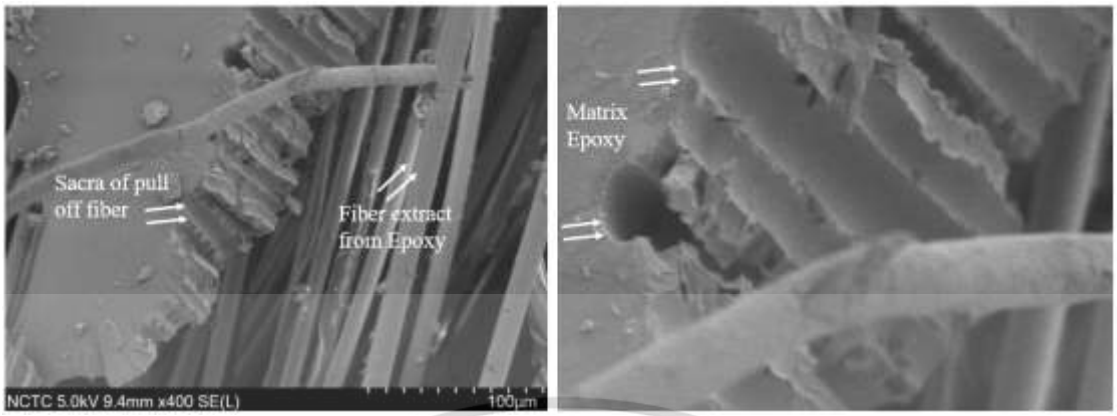
The fracture mechanism of carbon fiber and glass fiber composite were examine using SEM image analysis at 30ºC cured fracture as shown in figure 4.29 and 120ºC temperature cured fracture as shown in figure 4.29. The fracture mechanism of CFT show the fracture area are small but the matrix and fiber are break together to be small pieces especially vertical axis of tension force. The horizontal axis fiber are peel off after break of damage as shown in figure 4.29(a). For CFP, found that there are several porous trap between fibers it may help to start cracking when load are apply. In this composite, pull off fibers are found in vertical axis of tension load and peel off fiber bundle from horizontal axis of composite, those are similar fracture with CFT. Additionally, there is mechanical interlocking in the pitting area of fiber surface with matrix but very weak in chemical reaction between fiber and matrix. This make pull off fiber easily that are shown in figure 4.29(b). The roughness of fiber surface are still remain the same after pull of fiber, it represent the chemical bonding between fiber and matrix are very weak which cannot perform balancing load transferring to fiber. The fracture mechanism show that the wetting of fiber are very good that can cover the whole surface of fiber by matrix. But the weakness of attachment lead to pull out fiber easily from matrix.

(a) Carbon Fiber Twill Weave (CFT) of 30ºC cured Fracture



(b) Carbon fiber plain weave (CFP) of 30ºC Cured Fracture

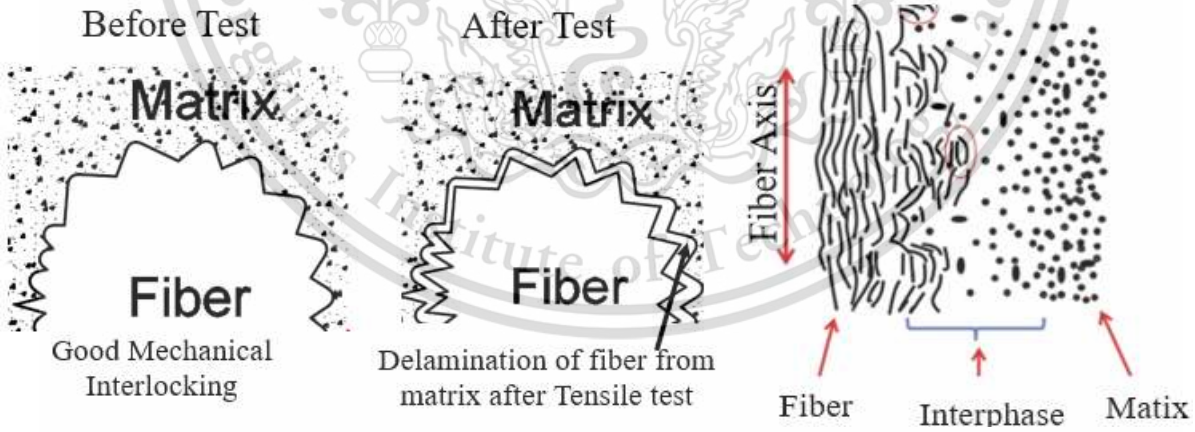
(c) Glass fiber woven (GFW) of 30ºC cured fracture



(d) Glass fiber fabric (GFF) of 30ºC cured fracture

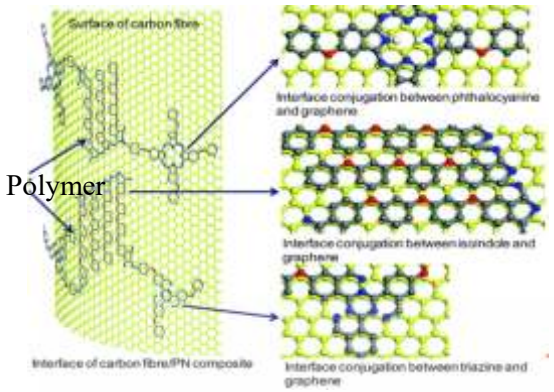
Figure 4.29 Fracture mechanism of 30ºC cured composite of (a) CFT, (b) CFP, (c) GFW and (d) GFF

For glass fiber, both GFF and GFW have the similar fracture mechanism of failure mode as their fracture area are quite large compare to carbon fiber composite. Most of failure mode are mainly extracted fibers from matrix. The voids from composite are one of the point to promote cracking of composite. Although fiber surface have several defects and crack on the cross section as shown in section 4.1.3 and 4.1.4, it cannot help to mechanical interlocking between fibers and matrix unlike carbon fiber. It make easily pull off fiber from matrix. Chemical reaction bonding of fiber and matrix are also not quite well. Therefore, the fiber pull area are remain clear and extracted fiber surface are also clear as fiber stand alone in fracture mechanism as shown in figure 4.29 (c) and (d). The fracture mechanism of glass fiber composite show that the fiber and epoxy are very weak in the composite, therefore the load transferring of external tensile load will not balance between fiber and matrix. The fracture prove that the chemical reaction of composite and mechanical interlocking are very weak in composite of glass fiber.

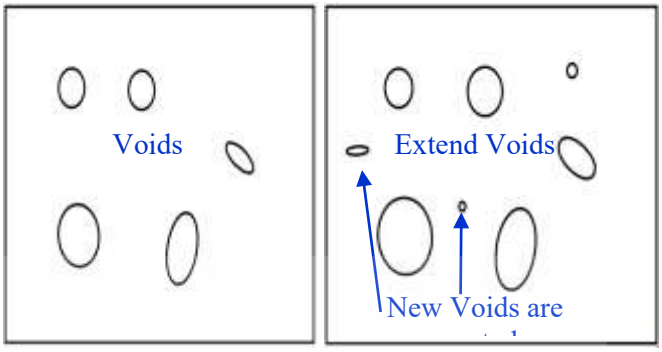


(a) Fracture before and after tensile

(b) Adhesive mechanism [54]



(c) Chemically bonding of fiber and matrix



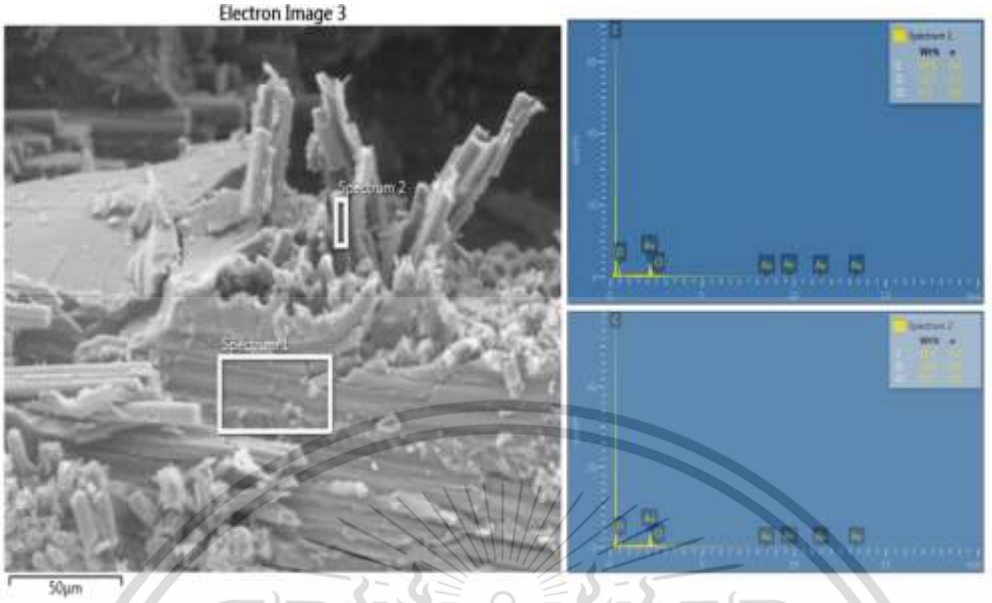
(d) Temperature curing mechanism

Figure 4.30 Mechanical interlocking between fiber and matrix, and temperature curing mechanism of composite

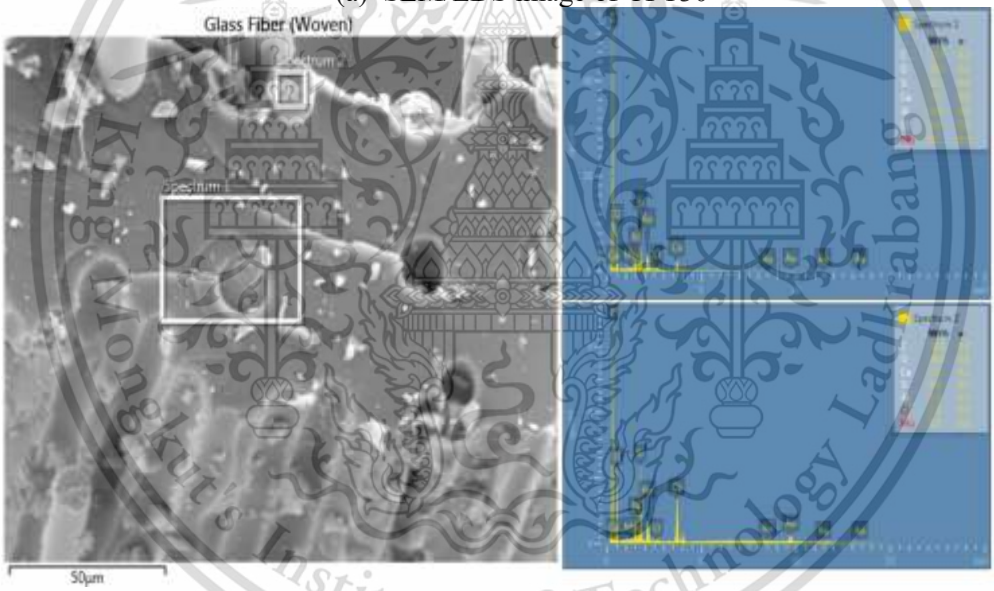
The mechanism of composite fracture are analysze by considering carbon fiber surface and epoxy matrix. Carbon fiber surface are pitting along fiber, it bring good mechanical interlocking between fiber and matrix. The fracture mechanism seem weak in chemical bonding between fiber and matrix. After tensile test are performed, the fibers are extract from matrix as shown in figure 4.29(b) and 4.30(a). It was agree with the previous researcher mechanism as shown in figure 4.30(b) and (c). The main interaction area of fiber and matrix area is valley pitting area that can make mechanical interlocking. Chemically bonding of fiber and matrix is very weak for carbon fiber with matrix epoxy. Temperature curing of composite shows the mechanism of changes while temperature post curing at higher temperature of composite. Normally hand lay up composite always trap micro porous inside of composite due to the moisture or bubbles of epoxy which is not removed completely. The existence porous are increase with the increase of curing temperature and new small porous are created while curing at higher temperature because of carbon fiber shrinkage, epoxy matrix shrinkage and crosslink increase at higher temperature. The diameter of carbon fiber filament from composite are reduce 9% for CFT120 compare to CFT30 while there is not shrinkage or reducing the glass fiber filament diameter.

4.4.3 SEM/EDS of Composite CFT and GFW at 30ºC

The composite of CFT and GFW of 30ºC cured were check in SEM/EDS to know the element composition of composite and found as shown in figure 4.31 (a) and (b). Two position are investigated as spectrum 1 at composite area and spectrum 2 at fiber alone in composite but it give different percentage of element containing. In CFT30, the chemical composite are carbon (87%), oxygen (11%) and chloride (0.3%). In carbon fiber alone, chemical composition are carbon and oxygen only, therefore we can consider that epoxy have three element such as carbon, oxygen and chloride. Glass fiber composite (GFW) show the element contain carbon (59-72%), oxygen (20-26%), silicone (3-5%), calcium (2-8%), aluminum (0.9-1.3%), chloride (0.3-0.4%) and magnesium (0.3%). The summary of composite is show in table 4.7.



(a) SEM/EDS image of CFT30



(b) SEM/EDS image of GFW30

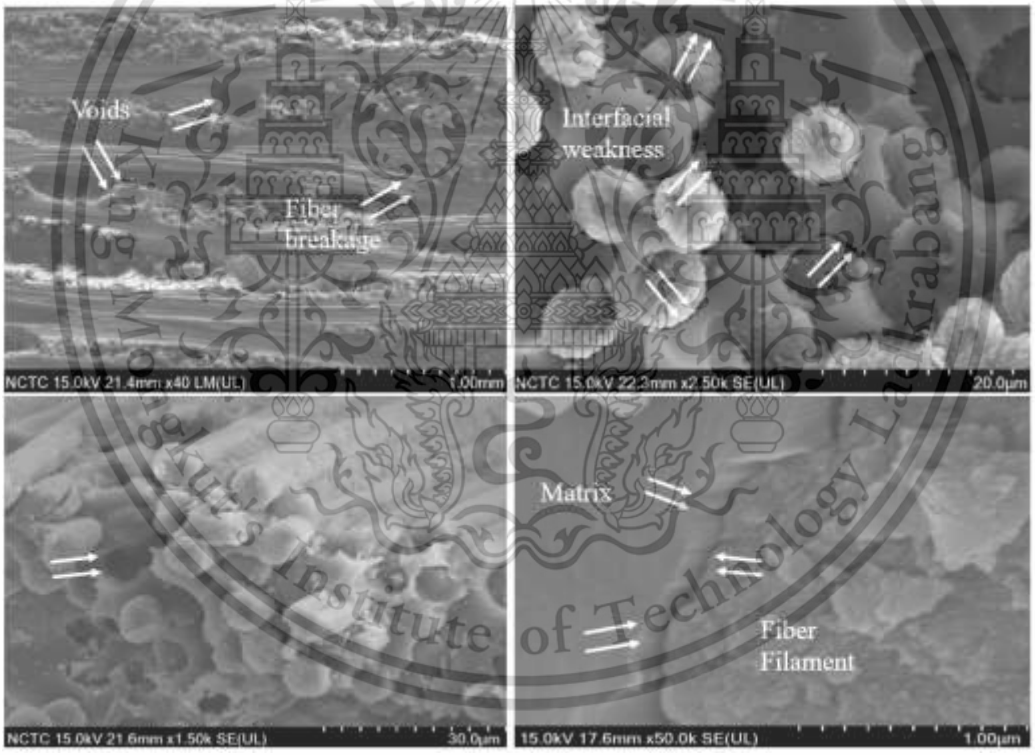
Figure 4.31 SEM/EDS image of (a) CFT and (b) GFW at 80ºC cured composite

Table 4.8 Element Containing Percentage in Composite of CFT and GFW

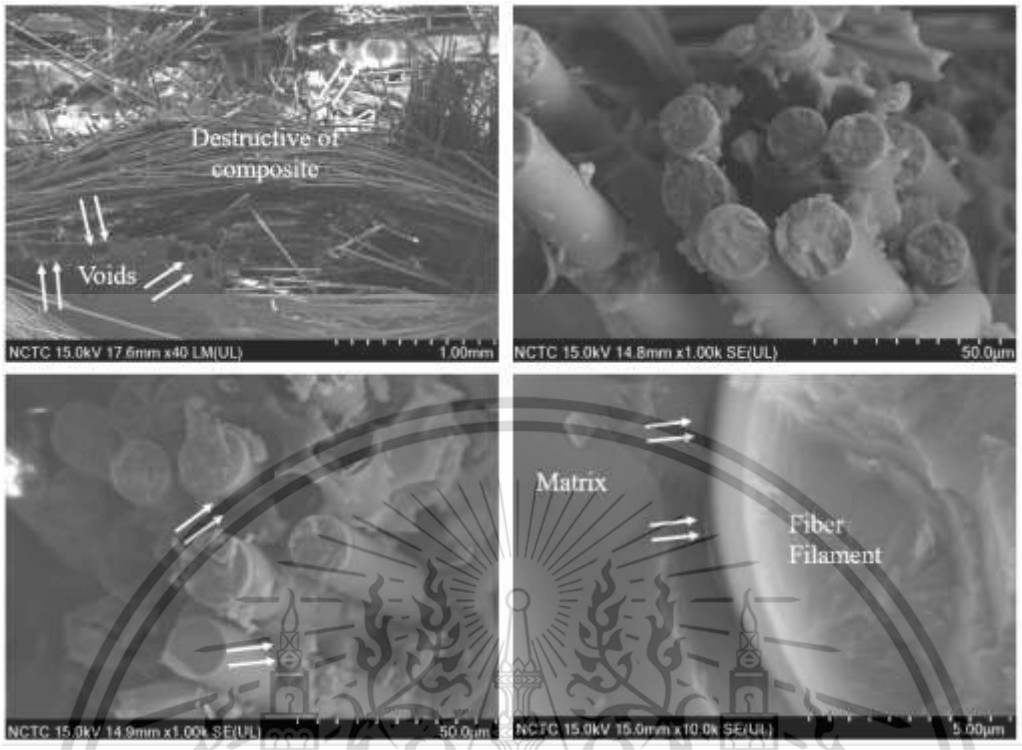
Element / Composite	Spectrum	Area	C (Wt. %)	O (Wt. %)	Si (Wt. %)	Ca (Wt. %)	Al (Wt. %)	Mg (Wt. %)	Cl (Wt. %)
CFT	1	Composite	87.9	11.7	-	-	-	-	0.3
CFT	2	Fiber alone	89.5	10.2	-	-	-	-	0.3
GFW	1	Composite	72.7	20.7	3.1	2.2	0.9	0.1	0.4
GFW	2	Fiber alone	59.2	26.6	4.8	7.7	1.3	0.2	0.3

4.4.4 Fracture Mechanism of CFT and GFW at 120ºC Cured Composite

High temperature cured at 30ºC, 80ºC and 120ºC were test in mechanical test. CFT30 cured composite fracture are mention above already. The fracture mechanism of CFT and GFW at 120ºC cured composite are examine by scanning electron microscope as shown in figure 4.32. In CFT120, the failure mode are not similar with CFT30, it is more compact than before and found well wetting fiber by matrix epoxy. More porous are found than before that can help initiating crack and propagation of cracking of composite. Matrix are increase to cross link each other at higher temperature and create voids in the place of pulling atom area. Horizontal axis of fiber bundle are peel off through tension force and mechanical interlocking on the fiber surface were seen but it was not sustain when tension load are applied. Interfacial bonding between fiber and matrix are very weak due to chemical bonding is not complete in the curing process. For glass fiber GFW120, it was also different failure mode from 30ºC cured composite failure. The fiber and matrix are holding tightly than 30ºC cured and very less pull off fiber in this fracture. The matrix hold fiber tightly not to pull off from external force because matrix work cross linking when the composite was cured at higher temperature for long time. It increase mechanical properties and increase brittle of composite



(a) Fracture mechanism of CFT at 120ºC cured composite

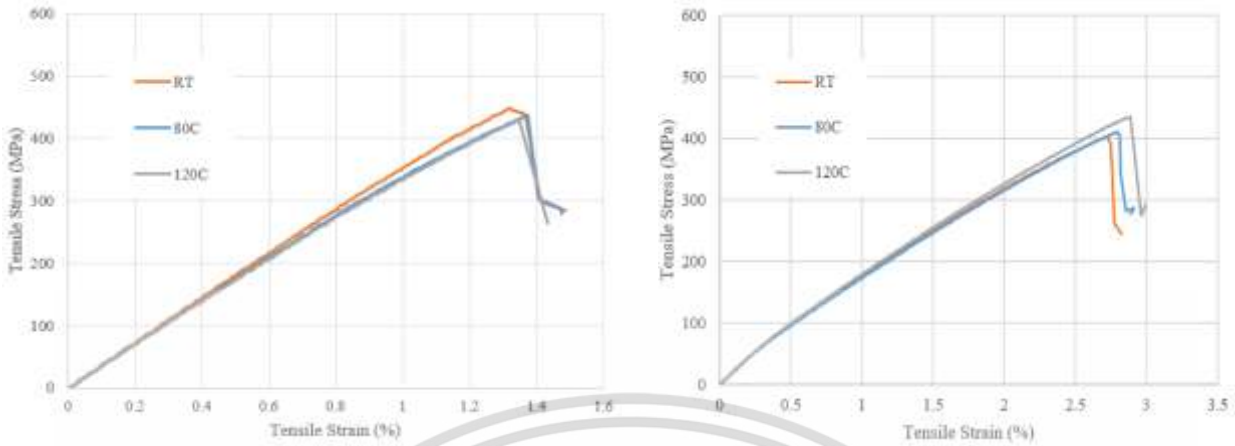


(b) Fracture mechanism of GFW at 120ºC cured composite

Figure 4.32 Fracture Mechanism of (a) CFT and (b) GFW at 120ºC cured composite

4.4.5 Tensile Properties of Treatment Fiber Composite

Carbon fiber twill weave pattern and glass fiber woven are choose among the previous four types of composite due to its higher mechanical properties. The fibers were treated to remove all of the particle on the fiber surface and fabricated composite with that treated fiber. The specimen for tensile test were prepared by the previous method describes in section 3.1.9. Six specimen were test and three result were used in this research. There was three condition for cured at room temperature and another cured at 80ºC for 6hrs and 120ºC for 3hrs after cut 30ºC cured specimens. Firstly the result at all temperature of TCFT and TGFW were shown in figure 4.33. TCFT are not increase with the increase of curing temperature base on the graph, cured at higher temperature was drop from 30ºC cured specimen. TCFT80 and TCFT120 was drop because fiber surface epoxy was weak to cross link with matrix epoxy due to it was removed by treatment method. For glass fiber, tensile strength was increase with the increase of cured temperature and it was opposite with carbon fiber. Not only strength, the elongation also increase with the increase of temperature.



(a) Treated carbon fiber composite (b) treated glass fiber composite

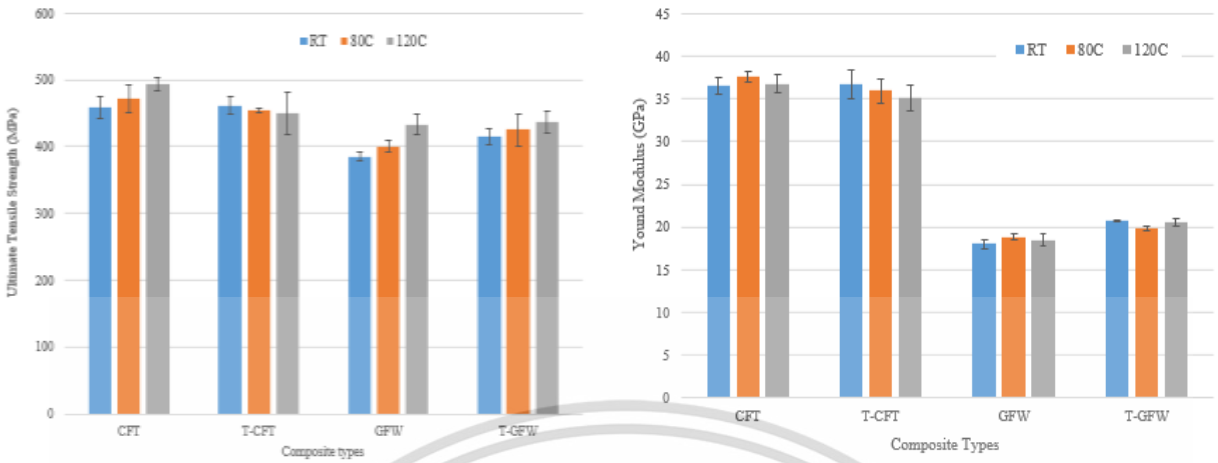
Figure 4.33 Treated composite result of (a) TCFT and (b) TGFW at 30ºC, 80ºC and 120ºC.

4.4.6 Comparison of Tensile Properties of Treated and Untreated Composite

Comparison to normal composite with treated composite at 30ºC, CFT is not increase between treated and untreated fiber. But for glass fiber, the strength was increase 7% in strength and 12% in modulus as shown in table 4.9. Figure 4.34 and 4.35 show tensile modulus and tensile strength of treated and untreated fiber composite at different temperature. From the table, the strength and the modulus for CFT and TCFT are not improve at all at 30ºC cured due to the de-sizing of the fiber surface are not effective much in the composite especially for carbon fiber in this research. Contrary, the strength and modulus of glass fiber are increase 7% and 13% respectively at 30ºC cured of GFW and TGFW. It make effective to the mechanical properties due to treatment of fiber in glass fiber.

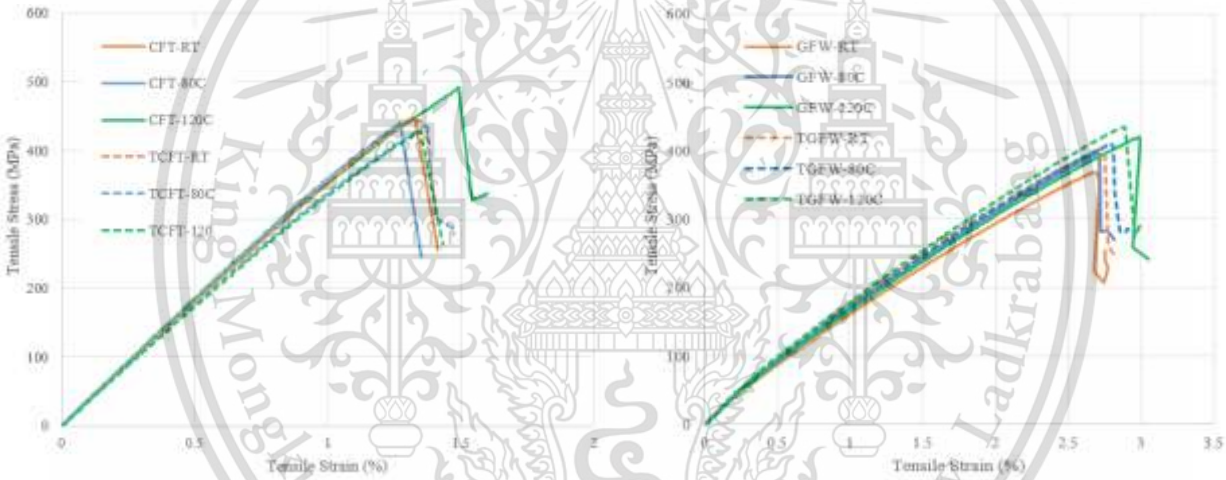
Table 4.9 Normal and Treated Fiber Composite at 30ºC

Composite	Max Load	Tensile Strength	Modulus	Elongation at Break	Increasing percent (%)	
	(N)	(MPa)	(MPa)	(%)	Strength	Modulus
CFT30	21470.85	458.22	36571.67	1.38		
TCFT30	21075.32	461.47	36699.67	1.37	0.71	0.35
GFW30	17600.28	385.35	18004.33	2.87		
TCGFW30	17802.41	415.16	20680.33	2.90	7.18	12.94



(a) Tensile strength (b) Modulus of composite

Figure 4.34 comparison of treat and untreated fiber composite at all temperature



(a) CFT tensile curve (b) GFW tensile curve

Figure 4.35 Tensile stress-strain curve of (a) CFT and (b) GFW at all temperature of treated and un-treated fiber composite

The graph and chart of both carbon fiber and glass fiber clearly show the improvement of fiber with the changing of temperature and with the treatment of fiber. For carbon fiber composite, untreated composite was increase a few of strength in the increase of temperature while the treated fiber was not increase the strength but it was drops. The modulus are increase with the increase of temperature but it was not slightly increase. The strength of glass fiber composite was increase step by steps with the increase of temperature and the treated fiber are improve with temperature changes. It may also change the morphology of composite and the fracture mechanism in composite.

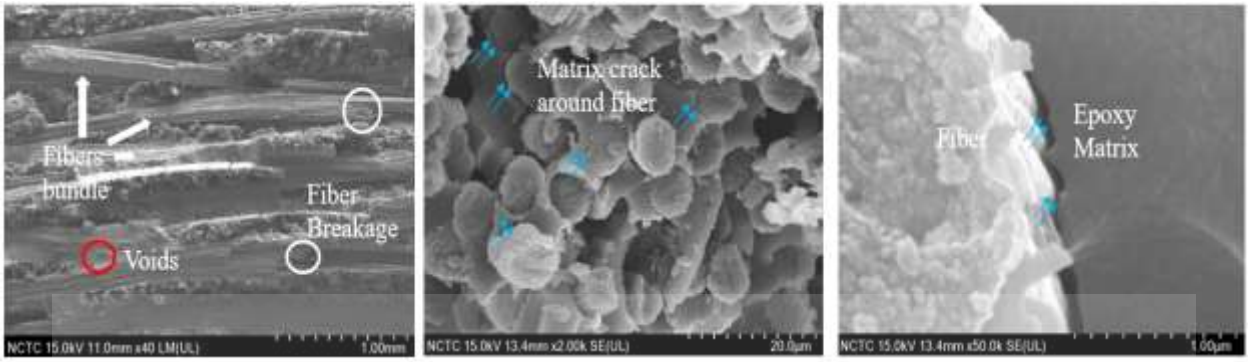
4.4.7 Fracture Mechanism of Treated Fiber Composite Cured at 30ºC and 120ºC

After the mechanical test are performed of the treated fiber composite, the fracture mechanism are determine by SEM and SEM/EDS. The whole fracture mechanism of composite are shown in figure 4.36. All of carbon fiber are break immediately while the specimen are testing, where as glass fiber are not break in all specimen but they are just bend. The fracture area of carbon fiber composite is much smaller than that of glass fiber composite. The breakage area of TCFT are at the smallest area near the grip due to load transferring area is the weakest area of composite. The load are gather at the smallest cross sectional area of the composite from all direction and it starts to break at matrix and crack propagate to the nearest neighbors fiber. Then the breakage are distribute to the whole area of cross sectional area. For glass fiber, the white color area is where the load concentration has applied except grip area. Some specimen show two area of load concentration and break at the place where is more weaker. The white color area is come from the damage of matrix epoxy and propagation of damage. The breakage area are very large and complex for glass fiber composite because crack are propagate. Micro scale of fracture mechanism are investigated to explain more detail of the fracture mechanism.

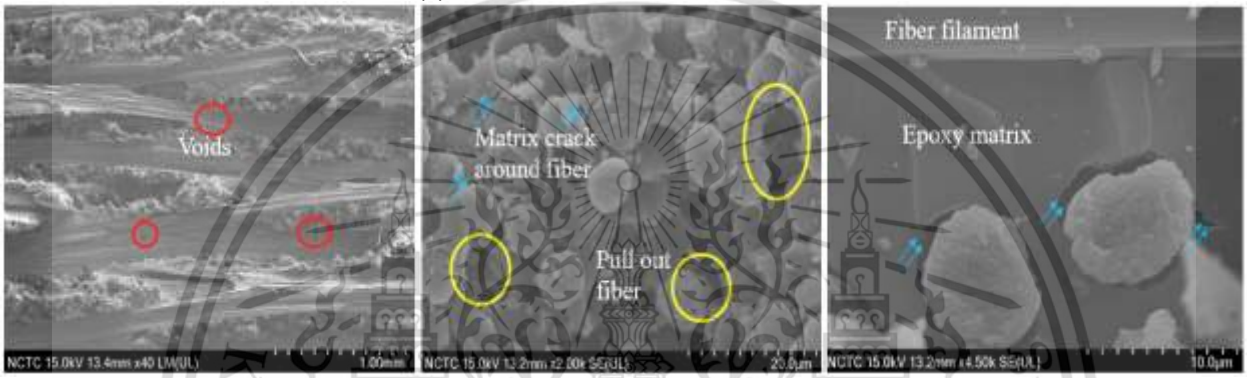
(a) (b)

Figure 4.36 The fracture specimen of treated (a) carbon fiber and (b) glass fiber composite at 30ºC and 120ºC cured

Fracture mechanism of 30ºC and 120ºC cured carbon fiber composite are shown in figure 4.37. The fracture mechanism of TCFT30 are quite different from the composite fracture of the CFT30. The fracture mechanism show clearly about the fracture in fiber bundle after the matrix are damage in the composite. Some porous or voids are found in the composite, it may help to initiate crack of composite. When the external tension force are applied, the crack will propagate along the direction of matrix epoxy. Horizontal fiber bundle are separated from the main composite while the vertical axis of fiber bundle are pull out from the composite through it the vertical tension load. Fracture mechanism show that fiber are well wet by matrix epoxy. The main fracture mechanism is found that the fiber are well attach each other in both temperature in mechanical interlocking. But it was become pull out fiber mechanism later after the mechanical test because chemical bonding are very weak in composite. The main needed for this mechanism is the chemical bonding between fiber and matrix, it will sustain the attachment of fiber and matrix epoxy. On the other hand, more holes and voids are found in the fracture surface at 120ºC cured composite and the matrix of composite more compatible around fiber. Therefore, the holes are form at the place between two layers of fabrics. It can initiate crack in the composite.

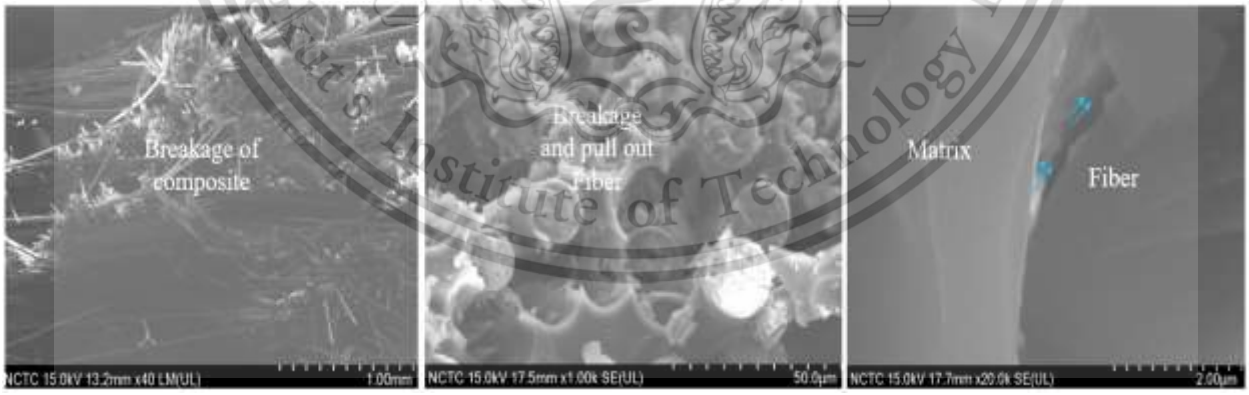


(a) Fracture mechanism of TCFT-30ºC

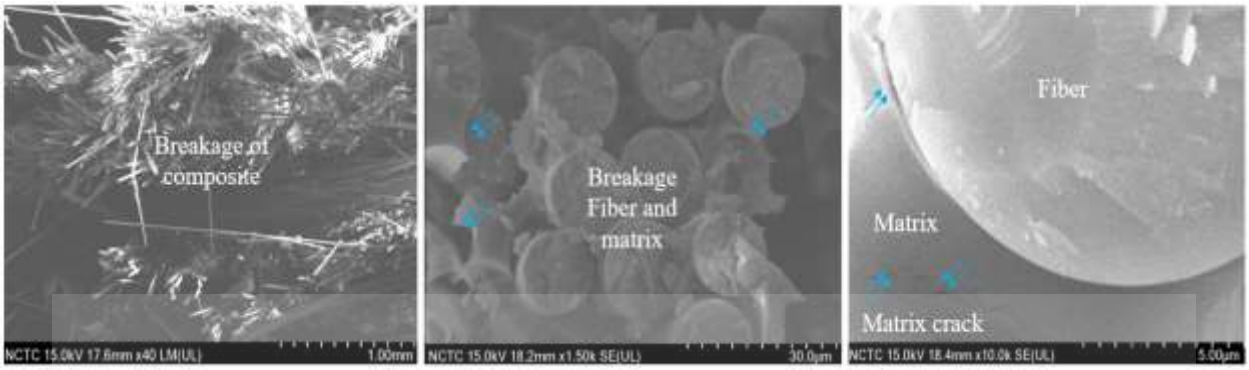


(b) Fracture Mechanism of TCFT-120ºC

Figure 4.37 Fracture Mechanism of TCFT at different temperature curing of (a) 30ºC and (b) 120ºC



(a) Fracture mechanism of TGFW-30ºC



(b) Fracture mechanism of TCFT-30ºC

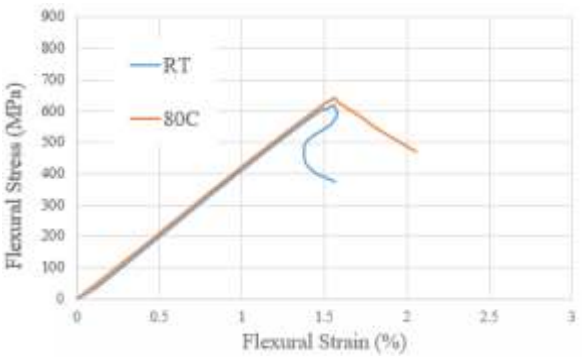
Figure 4.38 Fracture Mechanism of TCFT at different temperature curing of (a) 30ºC and (b) 120ºC

The fracture mechanism of treated glass fiber are quite different from the untreated fiber failure mode. In untreated composite, it show well wetting in fiber with matrix but failure surface are mainly pull out fiber from the matrix and the surface of fiber are very clear without sticking any of matrix in failure composite. But in treated fiber composite failure mode, the fiber are tightly attach with epoxy and fiber surface are full of epoxy matrix. At 30ºC cured, the fiber and matrix are well wetting and the main failure mode is pull out. But it was found that well chemical bonding in the surface of fiber. Therefore, the treatment of fiber is very effective in the glass fiber composite that can make tensile strength increase. The breakage area are large that was similar with untreated composite. At 120ºC cured composite, the fracture composite are obviously different from 30ºC composite, it show well wetting and good attachment between fiber and matrix. Fiber and matrix are break together in this composite and pull out fiber are lesser than 30ºC composite. The main failure mechanism is come from matrix due to it cracks in the composite that can not conduct well in load transferring to the fiber. High adhesion are found between fiber and matrix because cross link are increase with the increase of temperature. It can make high adhesion of fiber and matrix in the interfacial bonding to create high mechanical properties in the composite. Literature [43] also show that high bonding of composite are found at moderate temperature and time.

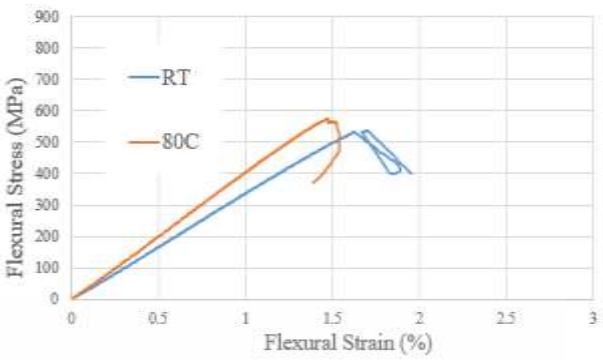
4.5 Flexural Properties of Composite

4.5.1 Flexural Properties of Normal Composite

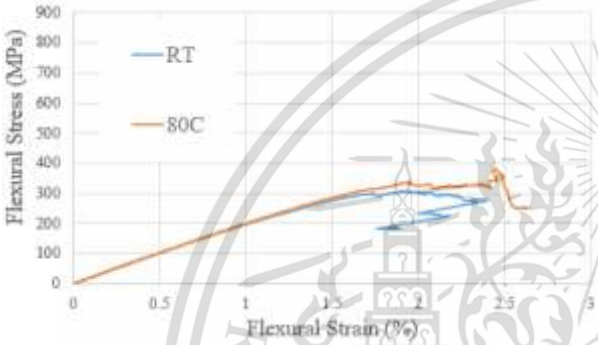
The mechanical properties of all composite are test at flexural strength at 30ºC and 30ºC+80ºC for 3hrs. Different fiber types are CFT, CFP, GFW and GFF type with epoxy matrix. The testing load vs elongation are used as a loading parameter. Flexural strength and flexural strain are calculated by the machine automatically and flexural modulus are calculated by the ratio of stress and strain. In this composite, all of the test are perform with the strain rate 5.2mm/min and all of the test result were shown in figure 4.39. In carbon fiber composite, the composite are suddenly break at deflection loading point at the outer surface of composite, it indicate the brittle behavior composite. Among two type of carbon fiber composite, CFT have higher flexural strength almost 15% is higher than CFP, different pattern of carbon fiber produce different strength. On the other hand, higher cured temperature composite are not improve much in carbon fiber composite, only a few percent are increase in flexural strength it was agree with tensile test result at high temperature.



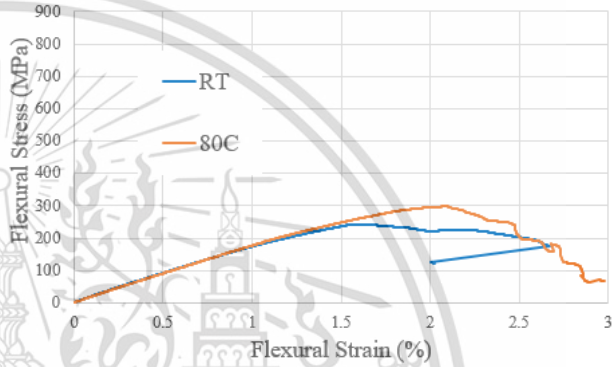
(a) CFT curve at 30ºC and 80ºC



(b) CFT curve at 30ºC and 80ºC



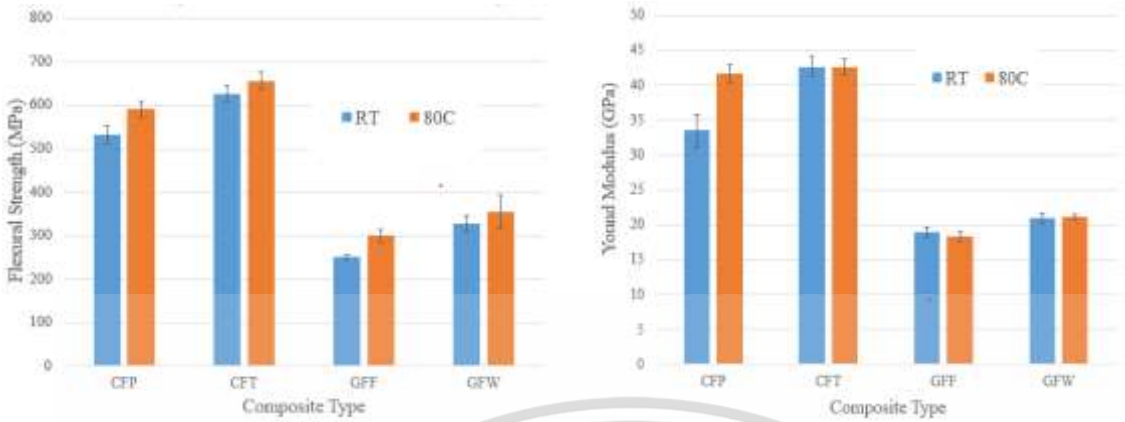
(c) GFW curve at 30ºC and 80ºC



(d) GFF curve at 30ºC and 80ºC

Figure 4.39 Flexural stress and strain of (a) CFT, (b) CFP, (c) GFW and (d) GFF at 30ºC and 80ºC

In the case of glass fiber composite, the fracture mechanism are quite different because glass fiber composite are break gradually at load bending point and break at the outer surface of composite. It represent the ductile behavior of glass fiber composite with large fracture area. The flexural strength 300MPa for GFW and 250MPa for GFF at 30ºC whereas elongation rate are almost similar. At 80ºC high temperature cured, the elongation are almost similar and flexural strength increasing rate are 350MPa for GFW80 and 300MPa for GFF80 respectively as shown in figure 4.39(c) and (d). The overall composite flexural strength and modulus are as shown in figure 4.40. All of composite are increase in the same increasing rate with the increase of curing temperature at 80ºC for 6 hrs. Therefore, we can consider that the increase of temperature in both carbon fiber and glass fiber composite bring the better flexural mechanical properties especially in flexural strength while the modulus of flexural are not effected by temperature curing.

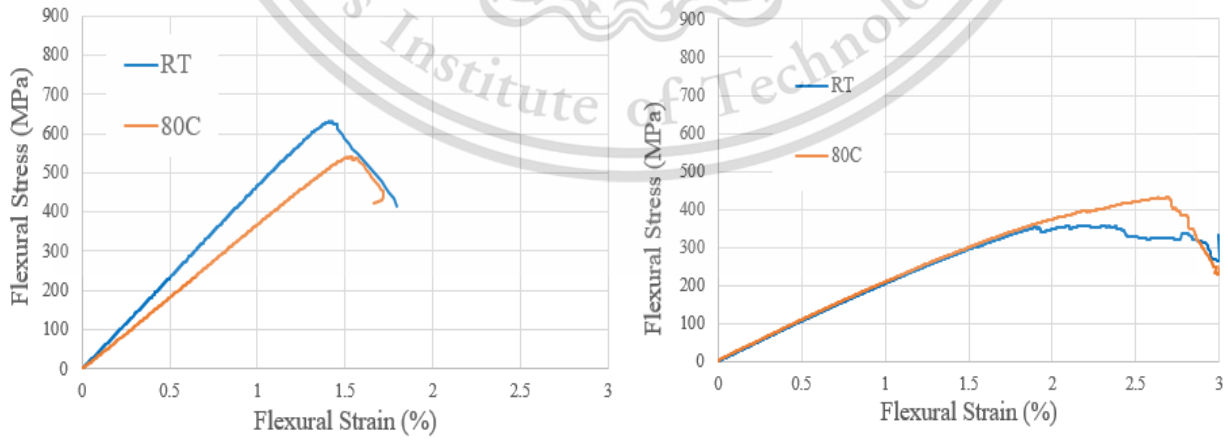


(a) Flexural strength of all composite (b) Flexural Modulus of all composite

Figure 4.40 Flexural strength of and modulus of all composite at 30ºC and 80ºC

4.5.2 Flexural Properties of Treatment Fiber Composite and their Fracture Mechanism

After all of the composite are tested at flexural and tensile, the highest mechanical properties of carbon fiber (CFT) and glass fiber (GFW) are choose to make a treatment fiber and fabricated composite to investigate the mechanical behavior and interface bonding of fiber and matrix. Treated fiber composite are cured as the previous two temperature at 30ºC and 80ºC. Firstly, Treated carbon fiber is at around 550MPa at 30ºC and 630MPa at 80ºC. Curing temperature effect to the flexural mechanical properties increasing 15% and the elongation of composite is also increase with the increase of cured temperature. The fracture mechanism of carbon fiber is break immediately after the maximum load is applied. It represent the brittle behavior of composite and elastic material. In the case of glass fiber, the fracture mechanism are break gradually and some of the specimens are not break but some are break only at the outer surface of composite in bending point are. It indicate the ductile behavior of composite with high elongation. The flexural strength are increase linearly from 30ºC composite result with the increase of curing temperature at 80ºC for 6hrs and elongation are almost similar. And the mechanical properties of glass fiber are increase around 8% increase in strength and 11% increase in elongation at normal cured specimens.



(a) Treated fiber of TCFT (b) Treated Fiber of TGFW

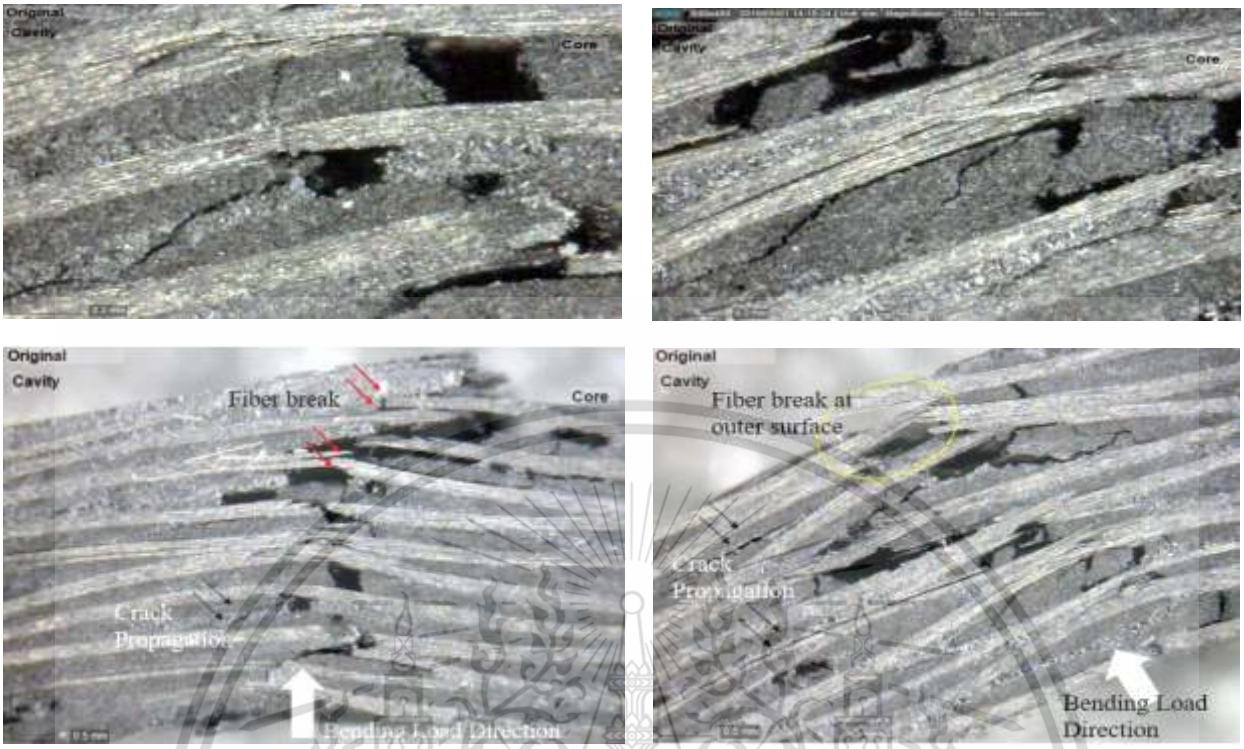
Figure 4.41 Flexural strength-strain curve of (a) TCFT and (b) TGFW at 30ºC and 80ºC

The fracture mechanism of flexural specimen are as shown in figure 4.43. The failure mechanism of carbon fiber and glass fiber composite are quite different. Almost half of carbon fiber composite are break immediately while the test are perform while glass fiber composite specimen are not break in any specimen. The touching area of flexural testing nose are define as internal and the opposite side was define as external side. For carbon fiber composite at different temperature cured are almost similar in failure mode of specimen, the external side of composite are starts to break when the maximum load are applied to the composite when internal side are bending. The matrix are start to break first at external side of composite and the load are transfer to fiber and propagate the crack but some specimen are still holding in the middle of composite which are break. The breakage behavior of carbon fiber composite represent the fracture mechanism of composite.

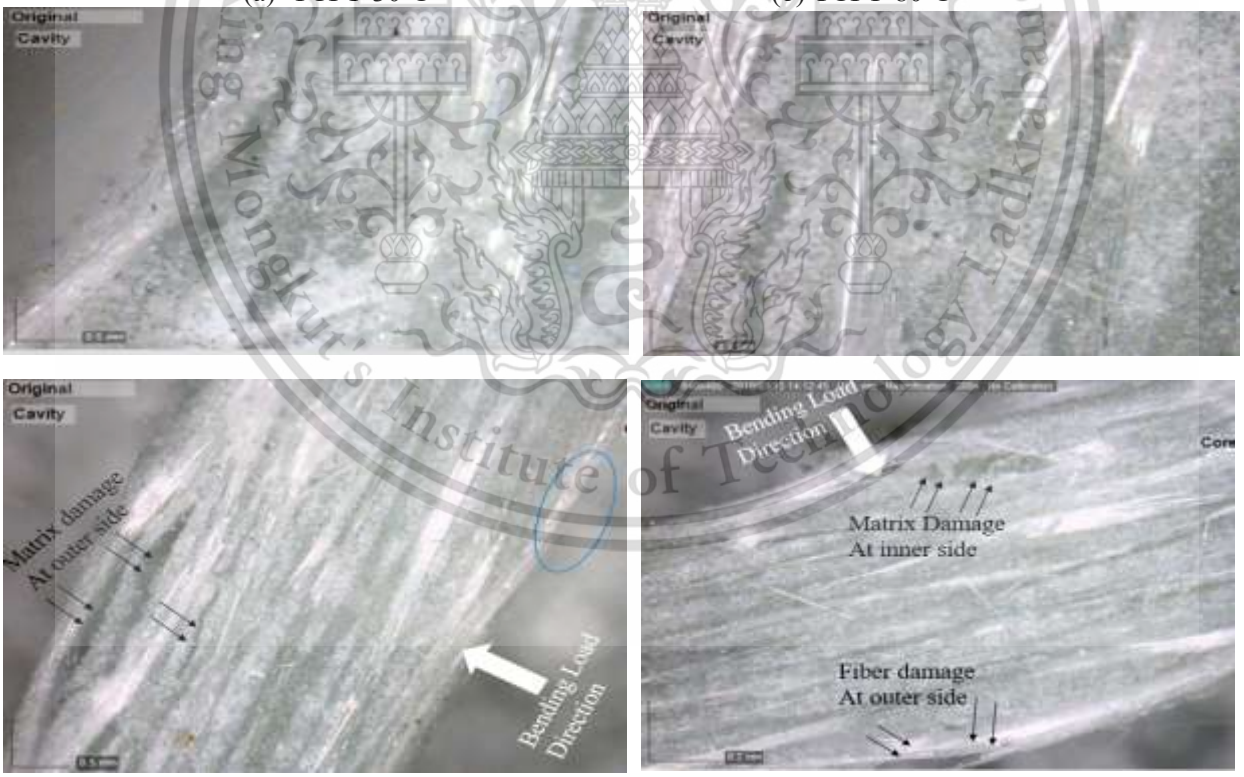
Glass fiber composite in flexural test specimen are not break in two pieces at all in all specimen. Slightly changes of composite give the information of different cured temperature due to 80ºC for 6hrs cured specimen are a bit yellowish compare to 30ºC cured. And the fracture mechanism of two temperature are a bit different because the deflection area of TGFW80 are much smaller than 30ºC cured and deflection area are turn to be white. The external composite side of bending area are tension and internal side are become compression and some fiber bundle lead to collapse between damage matrix. Even the maximum load was applied, the fiber are not break and it does not return to the original point. The graph of glass fiber composite are show that the load are continuous applied although the maximum load hits already. And the fracture mechanism of glass fiber composite represent brittle mechanical behavior of composite as shown in figure 4.43 (b).

(a) (b)

Figure 4.43 Flexural specimen fracture mechanism of treated (a) carbon fiber and (b) glass fiber composite at 30ºC and 80ºC cured sample



(a) TCFT-30ºC (b) TCFT-80ºC



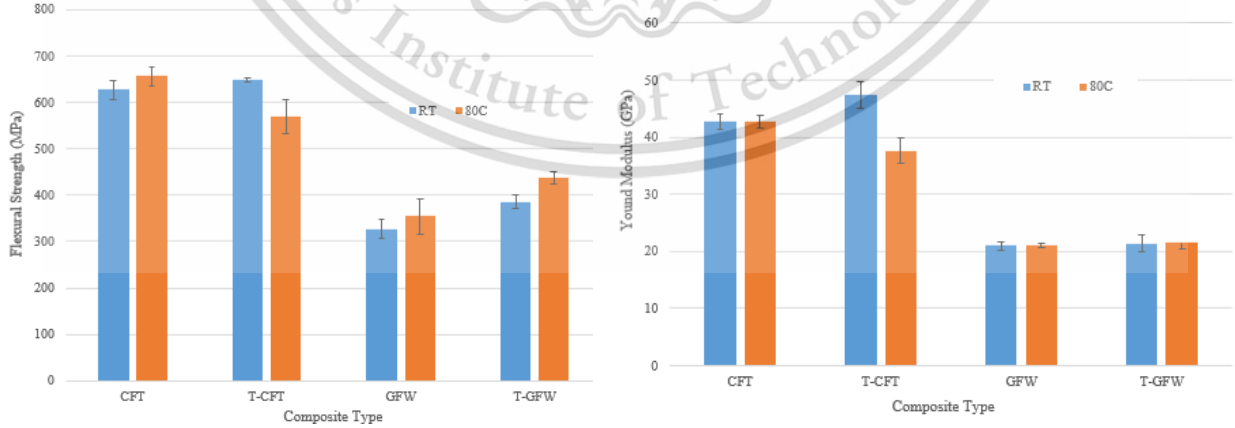
(c) TGFW-30ºC (d) TGFW-80ºC

Figure 4.44 Flexural Fracture of carbon fiber at (a) 30ºC & (b) 80ºC and Glass fiber at (c) 30ºC & (d) 80ºC

The fracture mechanism of flexural strength were examine by using optimal microscope. For carbon fiber composite, the matrix at the outer surface of were damage and the matrix crack are propagate along matrix axis. The fiber from the outer part of the composite are mostly break and the middle part fiber near the edge of composite are also break of room temperature cured specimen. For high temperature cured fracture of carbon fiber, the matrix are compact more or shrink matrix due to cross link of epoxy while curing at high temperature and it create some voids in the place of attracted element area. Therefore, more porous or voids are created in composite and it lead to crack easily at voids area as shown in figure 4.44 (a) and (b). The composite can consider as a brittle material due to the behavior of breakage and the mechanism of Matrix between fibers layer are become lesser and lesser due to matrix that are gather near fiber and the fiber pull the matrix to their side while temperature is increasing in curing. The fiber itself might not change due to high temperature curing. The fiber are breaks immediately due to its brittle properties. For glass fiber, fiber of 30ºC cured composite are break at the outer side of composite, opposite side to the flexural nose and the inner side of the composite are become fiber folding or collapse after damage of matrix as shown in figure 4.44(c). Oppositely, cured at higher temperature composite are different fracture with 30ºC cured composite. The matrix are damage first at outer side of composite at 80ºC cured and the touching area of bending nose are break in some fiber. Porous can not detected very well in glass fiber due to its color.

4.5.3 Comparison of Flexural Properties of Treated and Untreated Composite

(a) Treatment fiber composite of CFT (b) Treatment fiber composite of GFW



(c) Tensile strength of all treated fiber composite (d) Young Modulus of treated fiber composite

Figure 4.45 Flexural strength and modulus of treated fiber composite

The composite of all treated fiber were tested as the same condition as the previous specimens. In carbon fiber composite, the strength of TCFT30 are higher 3% than CFT30 but it was not significant changes and the strength are not increase with increasing temperature of TCFT80. Young Modulus of both TCFT and CFT are not significant change but slightly increase as shown in figure 4.45 (a) and (c). The elongation of carbon fiber composite are also the same or shorter, therefore, can consider the heating at oven for 6hrs with 80ºC release all of the moisture and evaporate. It make the brittleness if composite. For glass fiber, the strength and elongation are obviously increase with the increase of temperature and also increase on treatment fiber composite compare to normal composite as shown in figure 4.45 (b). Between two of GFW30 and TGFW30, 15% of strength are increase and 11% of elongation are increase, it indicated the treatment fiber on glass fiber are effective to the strength and elongation of GFRP composite. The strength was increase 12% and 17% increase in elongation of composite when the composite are cured at 80ºC for 6hrs on treatment fiber composite TGFW80. The result of TGFW30 and TGFW80 indicate, the treatment is effective to glass fiber composite and also heat treatment make the significant changes in composite mechanical properties especially in flexural strength.

Table 4.10 summary of mechanical properties in different testing method at elevated temperature

Name & condition			Tensile Fabric			Indentation Test			Tensile Test			Flexural Test		
NO.	Condition	Name	σ (MPa)	E (GPa)	ε (%)	Hardness (MPa)	E (GPa)	ε (%)	σ (MPa)	E (GPa)	Strain (%)	σ (MPa)	E (GPa)	ε (%)
1	Normal	CFP30	69.6	12.7	1.1	219.5	9.7	6.4	463.3	37.6	1.5	533.3	33.5	1.7
2		CFP80				425.1	9.4	5.5	476.9	37.5	1.5	591.1	41.6	1.6
3		CFP120							499.9	37.3	1.5			
4		CFT	133.6	22.4	1.1	334.1	10.9	5.5	469.5	36.6	1.4	626.9	43.1	1.6
5		CFT80				918.4	15.4	4.2	471.4	37.6	1.4	656.3	42.6	1.6
6		CFT120							506.3	36.7	1.6			
7	Treatment Fiber (Sizing)	TCFT30							461.5	36.7	1.4	648.8	47.4	1.5
8		TCFT80							454.3	35.9	1.5	570.1	37.6	1.7
9		TCFT120							450.3	35.1	1.4			
10	Normal	GFW	123.6	8.5	2.5	531.1	15.3	5.9	385.3	18.0	2.9	327.6	20.9	2.0
11		GFW80				557.9	14.9	4.2	400.6	18.8	2.8	354.9	21.1	2.3
12		GFW120							433.3	18.5	3.1			
16		GFF	123.1	8.1	2.8	474.0	15.9	4.4	357.8	15.4	3.0	250.5	18.9	1.7
17		GFF80				430.6	17.1	4.9	361.8	15.8	2.9	299.6	18.3	2.1
18		GFF120							379.1	16.8	3.0			
19	Treatment Fiber (Sizing)	TGFW30							415.2	20.7	2.9	385.0	21.3	2.3
20		TGFW80							425.4	19.8	3.0	438.3	21.4	2.7
21		TGFW120							437.0	20.5	2.9			

4.6 Thermo gravimetric analysis (TGA) on Composite at Different Temperature Cured

All condition of different temperature samples of carbon fiber and glass were tested in TGA for thermo analysis, to observe the different burning rate of sample and to confirm fiber volume fraction of different cured composite. Non-isothermal condition was used in all tested. Only one condition are tested for epoxy, raw carbon fiber and glass fiber filament, and then composite of carbon and glass fiber which was cured at 30ºC, 80ºC for 6hrs and 120ºC for 3hrs. In carbon and glass fabric, heating range is at 30ºC to 900ºC and air flow environment with 50ml/min. Carbon fiber filament weight was reduce until it was 0 % left in the crucible pan while glass fiber weight reduce only a few 1.23% in weight, it assume only moisture are remove from glass fiber. The tested result was shown in figure 4.46. The result confirm that carbon fiber reduce with three steps and at 800ºC all of the fiber are oxidized. The first step remove the moisture with 6% at 160-180ºC, second step reduce the coating epoxy around 27% at 350ºC and the rest are carbon oxidation with oxygen from 530ºC to 800ºC. And glass fiber reduce only 1.23% for moisture at 180C and the rest are constant continuously until 900ºC due to unburnable oxides are exist in glass fiber such as SiO2, MgO and CaO, these oxide cannot burn but it can melt inside of the furnace and it turns to solid state when the temperature cool down. For epoxy, most of the chemical composition are C, O and H, therefore, they are compatible materials and the weight are reducing in two steps. The moisture 15% are reduce in the first stage at 170ºC and the rest polymer are reduce at 350ºC. Therefore, the moisture containing is the highest one in epoxy matrix which may give advantage or disadvantage on the composite.

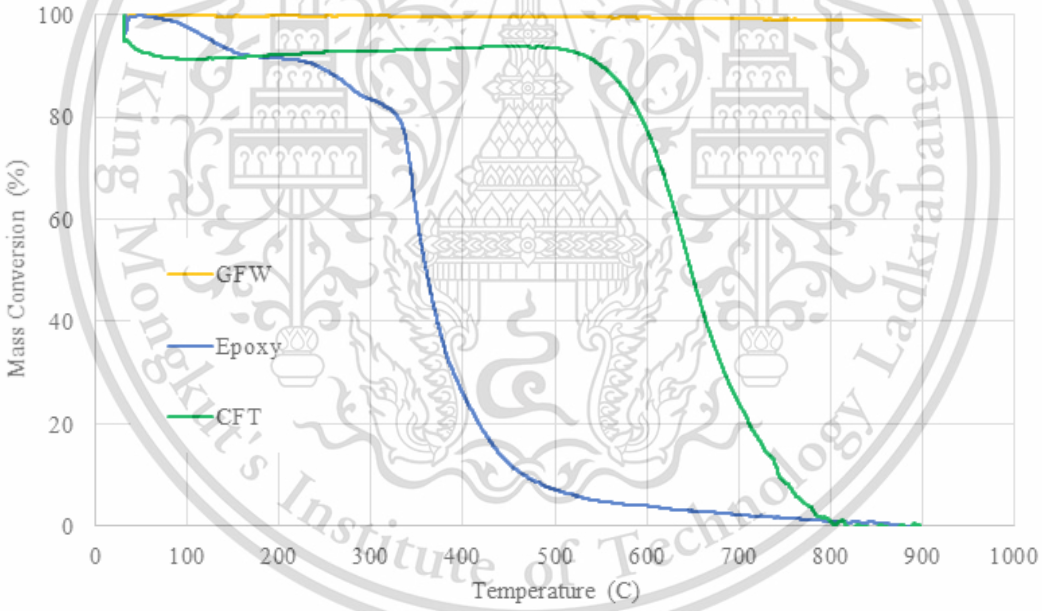
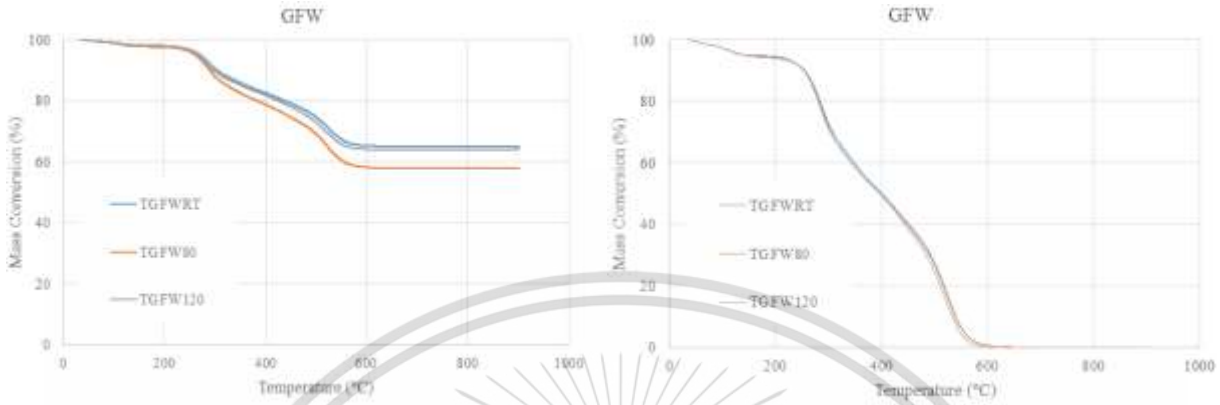


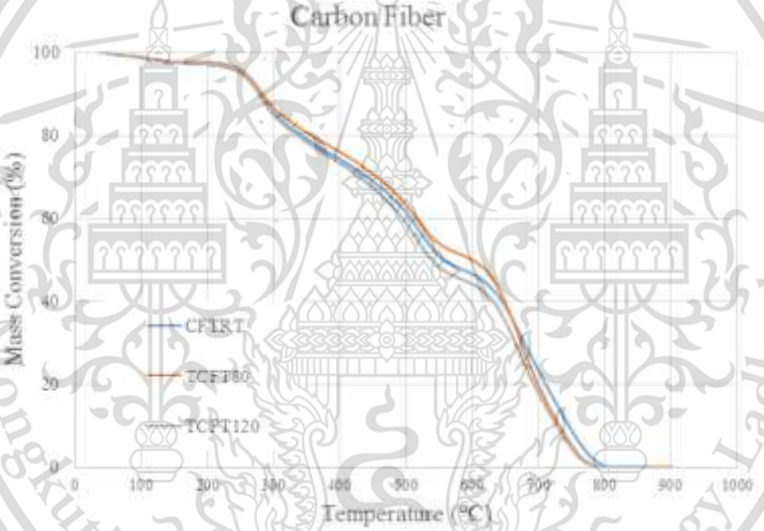
Figure 4.46 TGA result of carbon fiber and glass fiber filament

For glass fiber composite at different curing temperature, the result are also different based on the curing temperature condition with carbon fiber composite, it might another changes in glass fiber composite while the specimen was curing at higher temperature for limited time frame. For all curing parameter of glass fiber composite have different remaining part. Weight reducing steps are divided into three part such as moisture, epoxy matrix and fiber of composite as shown in figure 4.47 (a) and (b). TGFW80 burning rate was faster and weight release percentage was higher than GFW30 and GFW120. It can consider that, different effect in curing was given in composite especially chemical changes. Fiber and matric adhesion was higher at higher temperature that lead to better mechanical properties. GFW80 got the highest weight reducing at the first step one. It show that the good curing condition is at 120ºC for 3hrs which have the

highest cross link and it is not break bonding energy easily at high temperature before degradation of matrix epoxy. All test result was summarize in table 4.9 and it show burning rate in each steps.



(a) TGA of GFW (b) Combustible range of GFW



(c) TGA of CFT

Figure 4.47 TGA result of composite (a) glass fiber original result and (b) combustible process of glass fiber composite and (c) carbon fiber composite at 30ºC, 80ºC and 120ºC

The composite weight reduce at different temperature give different result. For carbon fiber composite, all of composite are reduce the weight until it was oxidized all with oxygen to be gas phase of CO2 or CO. The curing condition of composite at different temperature and their curing duration are much effect on the composite not only in mechanical strength but also in thermal stability. There are four steps TGA for carbon fiber composite as shown in table 4.11. For 120C curing composite CFT120, it show only 2.55% at 170ºC are reduce and it can assume curing temperature remove all of the moisture while CFT30 cure have 2.69%. The moisture containing in CFT30 is higher 16% compare to CFT120. The gap between each temperature are around 7% weight reducing at different range from 30ºC to 80ºC and 120ºC because the curing temperature effect to the composite, therefore, different temperature curing give different result. For 30ºC cured, it does not have additional crosslink increase in composite, that make the composite burning rate is slower than other 80 and 120C. CFT80 was increased in crosslink more in 6hrs curing time, that

make higher bonding energy especially in epoxy ring, therefore it was difficult to release attaching energy to break. It make the same effect to CFT120 sample to oxidize all of the matrix in composite.

Table 4.11 TGA burning step and remaining percentage of fabric and composite of carbon and glass fiber at different temperature cured condition.

Material	Step one		Step two		Step three		Step Four		Total Weight Red	Remain Weight (%)
	P. Temp	Wt. Red (%)	Peak Temp	Wt. Red (%)	Peak Temp	Wt. Red (%)	Peak Temp	Wt. Red (%)		
CF	200ºC	6.34	350ºC	27.17	650			66.26	99.8	0.23
GF	200ºC	1.23	-	-	-	-	-	-	1.2	98.8
Epoxy	170ºC	15.61	350ºC	80.89	-	-	-	-	96.5	3.5
CFTRT	173ºC	2.69	282ºC	21.88	522.21ºC	27.74	676ºC	47.65	100.0	0.04
CFT80	175ºC	2.24	281.5C	20.26	699ºC	25.81	669ºC	51.65	100.0	0.05
CFT120	170ºC	2.55	281ºC	22.22	515ºC	29.66	669ºC	45.47	99.9	0.09
GFWRT	178ºC	1.93	286ºC	14.97	-	-	521ºC	17.96	34.9	65.14
GFW80	178ºC	2.24	285ºC	18.12	-	-	521ºC	21.69	42.1	57.94
GFW120	175ºC	1.77	311ºC	15.36	-	-	521ºC	18.65	35.8	64.2

CHAPTER 5

CONCLUSION

In this research, Characteristic and morphology of carbon fiber and glass fiber are investigated in micro and nano structure using image processing method. And the composite of all fiber are fabricated using hand lay up process to observe the mechanical properties of carbon fiber and glass fiber composite. The fracture mechanism of carbon and glass fiber were investigated by using optical Microscopy and scanning electron microscopy. The current research will be divided into four parts such as characteristic of fiber, mechanical properties of composite, mechanical properties of treatment fiber composite and fracture mechanism of composite.

The investigate fiber was found that the commercial carbon fiber are full of pitting valley on the surface of fiber and chemical composition show 92-99% of C, 2-7% O_2 contains. On the other hand, glass fiber is full with defects along the fiber surface and micro porous at cross section of glass fiber. The chemical composition of glass fiber are Si, Ca, O, C and Mg. Transmission electron microscopy, X-ray diffraction and Raman spectroscopy are used to observe the nano structure of both carbon and glass fiber. The structure of carbon fiber are mixing graphite crystallite and carbon cluster or amorphous. solid. For properties of fabrics, CFT have the highest mechanical properties in tensile test among carbon fiber properties and GFW have higher mechanical properties compare to other glass fabric.

The composite were cured at three parameter of temperature such as 1) cured at room temperature (30ºC) until it is cured, 2) 30ºC cured + cured at 80ºC for 6hrs and 3) 30ºC cured + cured at 120ºC for 3hrs. The mechanical properties of all composite are tested in nanoindentation test, tensile test and flexural test. Nanoindentation of carbon fiber composite shows the increase of hardness and modulus with the increase of curing temperature. In tensile test, carbon fiber composite are improve gradually in tensile strength with the increase of temperature while glass fiber composite are increase significantly in tensile strength with the increase of curing temperature. For Flexural test, both glass fiber and carbon fiber composite are slightly increase with the increase of temperature in flexural strength. But it's not effect to the modulus of flexural strength. Therefore, higher temperature curing make effect to all composite especially for glass fiber, increase 11% at 80ºC in tensile strength and 18% in tensile strain. For carbon fiber, 4% increase at 80ºC compare to 30ºC cured sample. In indentation test, CFT80 increase hardness 63% compare to CFT30 while GFW80 increase 5% hardness. For Flexural test of composite, CFT increase 5% and GFW increase 8% at 80ºC temperature.

The highest mechanical properties of composite for GFW and CFT are choose to make a treatment in desizing solution for making better interfacial adhesion composite. The treated fiber composite are also tested in tensile test and flexural mechanical test. The treated composite of carbon fiber are not increase in tensile strength. Flexural test of treated composite are slightly increase compare to untreated fiber composite of carbon fiber. On the other hand, treated glass fiber composite are increase in both tensile and flexural strength. Both of tensile and flexural strain also increase in treated fiber composite and strain also increase especially with the increase of curing temperature. Glass fiber treatment make higher improvement around 15% and carbon fiber treatment increase only 3% in composite.

The fracture mechanism of composite are investigated by using SEM for all of fracture composite. 30ºC of cured Carbon fiber composite are crushing into small pieces at the same time for both matrix and fiber. But the fracture mechanism of 30ºC and 120C cured sample are different. The fiber are well wetting in matrix and well adhere between fiber-matrix mechanical interlocking at 120C cured sample. The main failure mode is pull out fiber due to it was not well interact in chemical bonding between fiber and matrix.

At higher temperature cured, the composite become brittle due to increase cross linking in matrix but it can create holes or porous. Therefore, even though brittleness make higher strength, the hole or voids are also the good point to initiate the crack in composite.

For glass fiber, the damage are is very large and pull off fiber surface is very clear due to the weakness of bonding and mechanical properties. On the other hand, 120ºC cured composite of glass fiber found that more compatible than 30ºC cured, it lead to the better adhesive between fiber and matrix. The most important of glass fiber is to bond chemically due to glass fiber surface do not have the roughness surface instead of mechanical interlocking. The composite have a good chemical bonding at high temperature curing that lead to increase strength in both tensile and flexural test especially for glass fiber composite. Treated glass fiber composite is better compare to normal composite. After removing coupling agent from the fiber surface, it was better to adhere in matrix and fiber, therefore it improve the mechanical properties. The failure mode of treated fiber is crack matrix at higher temperature cured composite.

Future Work

There are several plan to do more investigation for further improvement in composite field. Mainly I have two plan, one is to measure the roughness of fiber surface before and after a treatment because the mechanical properties of composite are much depend on the interfacial bonding or mechanical interlocking between fiber and matrix. High roughness surface give higher mechanical properties due to good mechanical interlocking between fiber and matrix. On the other hand, low roughness surface give low mechanical interlocking that can make the weakness of interfacial bonding. Another future work is to control the porosity of composite by using vacuum or other special tool while the composite are fabricating because porosity make effects to the mechanical properties and it might be one of the reason to start crack initiation while mechanical test are apply. If we use special tool or infusion method to make a composite, the composite might be looking good at both side, get rid of porosity and more compatible. Therefore, I would like to make a further research or suggest to make this kind of method for further improvement in composite field.

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APPENDIX

APPENDIX A

Abbreviation

CFT	-	Carbon Fiber twill weave
CFT30	-	CFT cured at Room temperature
CFT80	-	CFT Cured at 80ºC for 6hrs after cured at 30ºC until it was dried
CFT120	-	CFT Cured at 120ºC for 3hrs after Cured at 30ºC until it was dried
CFP	-	Carbon Fiber plain
CFP30	-	CFT cured at Room temperature
CFP80	-	CFT Cured at 80ºC for 6hrs after cured at 30ºC until it was dried
CFP120	-	CFT Cured at 120ºC for 3hrs after Cured at 30ºC until it was dried
GFW	-	Glass Fiber woven
GFW30	-	GFW cured at Room temperature
GFW80	-	CFT Cured at 80ºC for 6hrs after cured at 30ºC until it was dried
GFW120	-	CFT Cured at 120ºC for 3hrs after Cured at 30ºC until it was dried
GFF	-	Glass Fiber fabrics
GFF30	-	CFT cured at Room temperature
GFF80	-	CFT Cured at 80ºC for 6hrs after cured at 30ºC until it was dried
GFF120	-	CFT Cured at 120ºC for 3hrs after Cured at 30ºC until it was dried
TEM	-	Transmission Electron Microscopy
SEM	-	Scanning Electron Microscopy
XRD	-	X-ray Diffraction
RS	-	Raman Spectroscopy
FT-IR	-	Fourier Transform Infrared
TGA	-	Thermo gravimetric Analysis

APPENDIX B

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Mechanical property and fracture mechanism of glass fiber reinforced polymer and carbon fiber reinforced polymer

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Abstract. In the automotive field, most of the manufacturers are looking to replace the material steel, aluminum with lightweight material like carbon fiber or glass fiber composite. The purpose are due to their relatively high strength, higher chemical resistance, flexible usage temperature and higher stiffness than steel. In this study, mechanical properties of carbon fiber and glass fiber reinforced polymer were investigate. It was find that the tensile strength of carbon fiber composite is approximately 11% higher than that of glass fiber, almost twice in Young's modulus than that of glass fiber. Carbon fiber is two times higher than glass fiber in both flexural stress and young modulus of flexural three points bending test. Image analysis of fracture and damage were detect by field emission scanning electron microscopy (FESEM) in microstructure scale to observe the fracture mechanism. Observed different failure mode in fiber and resin. Chemical composition of composite and fibers were investigated by using electron dispersive x-ray (EDX) spectroscopy that gave out in 88 w.t % of carbon and 12 w.t % in carbon fiber twill (CFT) composite. On the other hand, glass fiber woven (GFW) composite contained 72.7 w.t % of C, 20.7 w.t % of O_2, and the rest contained Si, Ca, Al, Mg and Cl.

Keywords: Carbon fiber, Glass Fiber, Fracture Mechanism, Mechanical Properties

1. Introduction

Nowadays, most of the automotive manufacturer deeply invested to make research on carbon fiber and glass fiber due to their lightweight properties. The purpose of lightweight vehicles is for future automotive fields [1]. Vanishing of CO_2 footprint and preventing the particles matters (PM) emission from vehicles in our environment are also one of the most important point. Carbon fiber and glass fiber are the most popular materials not only in the automotive field but also in the field of marine, aircraft, military vehicles, sports, rockets and etc [2]. The main properties are lightweight, high stiffness, high

temperature resistance and high strength compare to the same weight [3,4]. In composites, there are two main functions those are fiber and matrix. Fiber is the main product in the composite and matrix is one of the material that can create bonding strength between fibers [5]. Polymer matrix is the most widely used in the composite industry. There are two main matrix resins such as thermosetting and thermoplastic resins; those are the most useful resin in the composite field for laid up process laminar. Thermosetting resin is liquid form and it is irreversible resin [6]. After curing thermoset resin, the resin cannot turn back to the original liquid form and the resin can cured by chemical reaction at room temperature. Among several resin types in thermoset, epoxy is the most useful matrix because the matrix can lead composite to the best material of high bonding strength [7]. The composite need higher strength to resist the force and epoxy have to transfer stresses to fibers. Most of the composite starts to crack from matrix de-bonding, fibers delamination and pull out fibers from the matrix failure because matrix is not wet enough to bond one to another fiber. Then matrix cannot distribute the load uniformly to the fibers [8]. Dany Arnoldo Hernandeza [9] observed the main fracture of the composite is come from the interface zone (area between resin and fiber) which is affected by the tensile stress concentration on resin. It makes a crack initiation at the resin area, and then the force and crack are distribute to the fibers. Therefore, fiber is pull out then leads to break fibers. Issa A. Hakim [10] studied the porosity of the composite according to different vacuum poor, moderate and high vacuum. As the result, the poor vacuum composite has the highest quantity of porous that can make lower strength with faster crack then the toughness of fibers are decreasing. Energy absorptions are drop compare to moderate and high vacuum. From the view of an image, it is quite important to investigate the porous inside fiber and the structure of each fiber that can affect mechanical properties and fracture of composite. P. Karin [11] observed particle matters (PMs), average agglomerated particle diameter sizes are in the range of 50-500 nm size and average nanoparticle diameter size are in the range of 20-50 nm respectively. He used (TEM) transmission electron microscopy to investigate the size of PM. P. Karin [12] used TEM to observe particle matters size from biodiesel engine and diesel engine with different speed RPM. He investigated the average of nanoparticles size in approximately 48, 34 and 32 nm for carbon black for biodiesel and diesel engine's PMs respectively. P. Karin [13] has investigated carbon black by using electron microscopy analysis. Therefore, electron microscopy is one of the most important tool to study fracture mechanism. However, in case of composite, micro and nanostructures are not understand well, it induces to investigate the properties and fracture mechanism of composite with different type of base material.

2. Materials and Methodology

2.1. Materials

In this research, two types of materials were used with epoxy resin matrix; carbon fiber (twill weave and plain weave) based on polyacrylonitride (PAN) [8, 9] and E-glass fiber (woven roving and fabrics). Epoxy YD 582 and hardener TH7253 are use as the matrix in this composite. Listed materials in table 1 are supply by concrete composite.

Table 1. Materials type

No.	Material	Prod name	Composites name in this paper	Type
1	Carbon fiber twill	C135T	CFT	
2	Carbon fiber plain	PC6951500	CFP	Carbon
3	Glass fiber	Woven roving N323	GFW	
4	Glass fabrics	Glass cloth EW200	GFF	Glass
5	Epoxy (part: A)	YD 582		
6	Hardener (part: B)	TH 7253		Resin

2.2. Sample preparation

Orientation for all of the materials are bidirectional 0/90 degree. The fabrication process are wet lay up process based on the weave fibers pattern from manufacture. All of the fibers are lay up in the same orientation. In epoxy mixing ratio, there are two parts, part A: epoxy and part B: hardener are mix for 3:1 ratio based on fiber ratio and equations (1-3). The thickness of composite were control based on the fabric thickness of each layer. The same method is use for all composites with wet lay-up process. All of laid up conditions can see in table 2.

$$Hardener\ weight\ (g) = \frac{Weight\ of\ Fibers\ (g)}{4} \quad (1)$$

$$Epoxy\ weight\ (g) = weight\ of\ hardener\ (g)\ x\ 3 \quad (2)$$

$$Epoxy\ and\ hardener\ ratio = 3:1 \quad (3)$$

Composite was cut by computer numerical control (CNC) machine Mazak FJV-20 with the situation of spindle speed 150 mm/min and feed speed 90-100mm/min. Samples are prepared according to ASTM 638 for tensile test and ASTM 790 for flexural test specimens. Fabricated composite information of composites.

Table 2. Material ply and lay up status

No.	Material	Fabric layers	Fabric thickness (mm)	Fibers w.t (g)	Composite size (mm)	Comp w.t (g)
1	CF twill	10	0.31	261.1	320x320x3.2	512
2	CF plain	9	0.3	343.29	240x280x2.9	429
3	GF woven	10	0.3	408.1	320x320x3.2	636.06
4	GF fabrics	16	0.2	343.62	320x320x3.2	574.167

2.3. Mechanical testing

The specimens were test on a tensile test machine INSTRON 8801 with load cell 95KN, test speed 5mm/min for all composite with the same testing function at temperature 24 degrees Celsius with 47% RH. Another mechanical flexural test (three-points bending) is perform at 24C temperature and 49% RH with INSTRON 55R4502 machine. Testing rate speed is 5.2mm/min with cell load 10KN.

3. Result and Discussion

3.1. Tensile test results with different materials CFT, CFP, GFW and GFF

Figure 1 shows the average value of each composite. CFT/CFP has the highest tensile strength than that of GFW/GFF. The tensile strain of glass fiber is two times longer than carbon fiber elongation. CFT is the peak tensile stress around 450MPa while CFP is around 420MPa at the same tensile strain 1.3%. Maximum strength of GFW and GFF are decrease particularly compare to CFT /CFP. The elongation at break point are 1.37% for CFT and 1.31% for CFP respectively, while GFW and GFF elongation are 2.85% and 2.91% respectively. Young modulus is the main important point in mechanical test and properties. Therefore, the highest young modulus is around 37 GPa from CFP and it is almost similar to CFT composite with the value of 36 GPa. The value of CFT and CFP are almost two times higher than that of GFW and GFF's young modulus.

Tested materials data are summarize and compare to each sample properties in table 3. The specimens are not the same thickness, but the thickness will not affect to young modulus of materials

because material properties are not depends on the thickness of samples. Unlike steel or alumina, the samples are sensitive and break at several points. Therefore, strain gauge was not use in this experiment. The elongations are calculate from sample crosshead displacement because it was collect at maximum strength of sample.

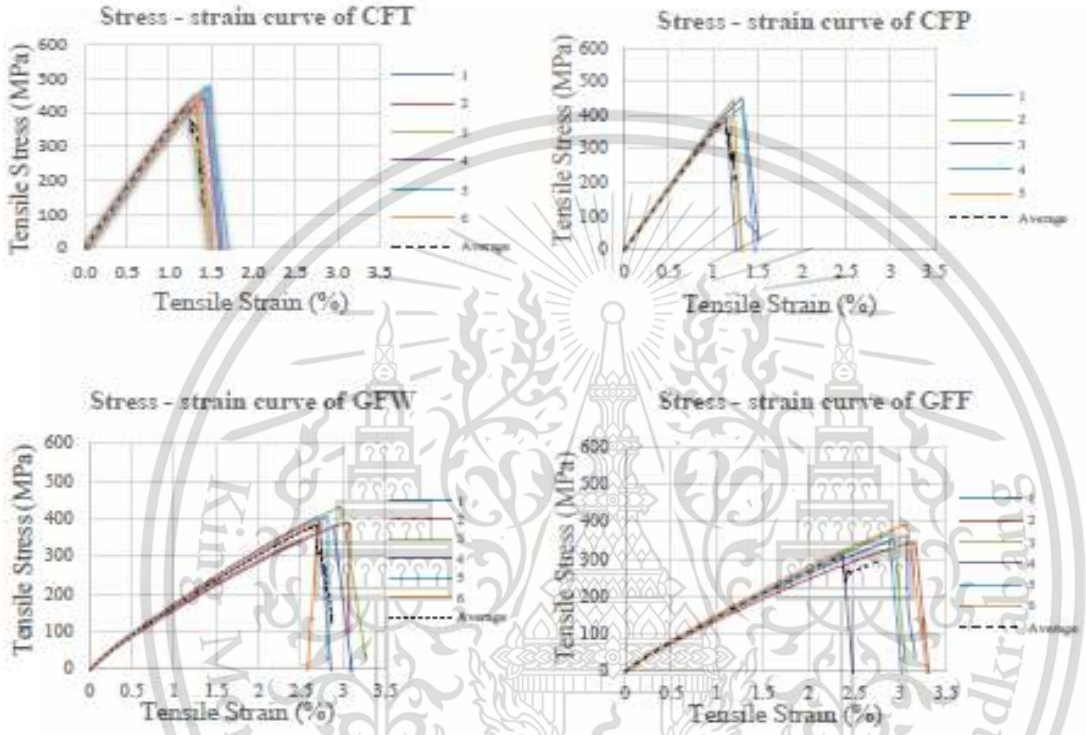


Figure 1. Stress-strain relationship of tensile test data of CFT, CFP, GFW and GFF

The specimens were disintegrate to be several pieces immediately after the peak points of tensile stress for CFT. It leads to brittle behaviour, but higher stiffness. Composite breakage of CFT and CFP are almost linear. Figure 1 also shows that stresses are immediate drop. For glass fibers, the nature of glass are flexible and higher elongation. The fracture behaviour of GFW and GFF are tougher comparable to CFT and CFP behaviour. After the maximum load are applied, the specimens break immediately in the weakest point of the sample where the fillet of specimens are located for all types of specimens. The failure mode of composite are as shown in figure 2.

Table 3. Comparison of composite material and their properties of tensile test

Samples	Thickness (mm)	Width (mm)	Grip distant (mm)	Maximum load (N)	Tensile strength (MPa)	Young modulus (MPa)	Elongation at break (%)
CFT	3.5	13.35	115	21,332.87	452.98	36,204	1.37
CFP	2.85	13.6	115	16,328.60	430.73	36,604	1.31
GFW	3.3	13.6	115	18,182.37	399.85	18,620	2.85
GFF	3.2	13.7	115	15,582.11	358.99	15,860	2.91

Figure 2. Fracture sample of tensile test a) CFT, b) CFP, c) GFW and d) GFF

3.2. Flexural Test

Three points bending flexural test condition are as mentioned; six pieces of each type are tested. The average flexural strength of all specimens are as shown in figure 3. Each type of samples are much different not only in flexure stress at maximum load but also in young modulus. The summary and properties of all flexural tested conditions are show in table 4. CFT and CFP have a big gap in maximum stress but almost the same flexure strain in elongation. GFW/GFF in this flexural test are drop gradually without breaking immediately. After the maximum load are applied, the fibers breaks unstably and tends to more elongation. In young modulus, CFT is the best one with the highest value of 47 GPa and the next follow by CFP with 42 GPa. Young modulus for GFW and GFF are lesser more than compare to CFT. On the other hand, glass fiber are very good in elongation that lead to the good toughness behaviour. The failure behaviour are also quite different in each type as shown in figure 4. Tension side of surface starts to crack first due to tension load and compression side are crack.

Table 4. Flexural strength and modulus of composites

Name	Maximum flexure load (N)	Flexure Stress at max load (MPa)	Modulus (automatic young's) MPa	Flexure strains at max flexure stress (%)	Thickness (mm)	Width (mm)
CFT	655.69	675.37	46,975	1.55	3.35	13.34
CFP	425.35	598.26	41,945	1.53	2.88	13.63
GFW	329.04	327.15	20,720	2.21	3.32	13.67
GFF	229.33	246.18	18,603	1.72	3.2	13.66

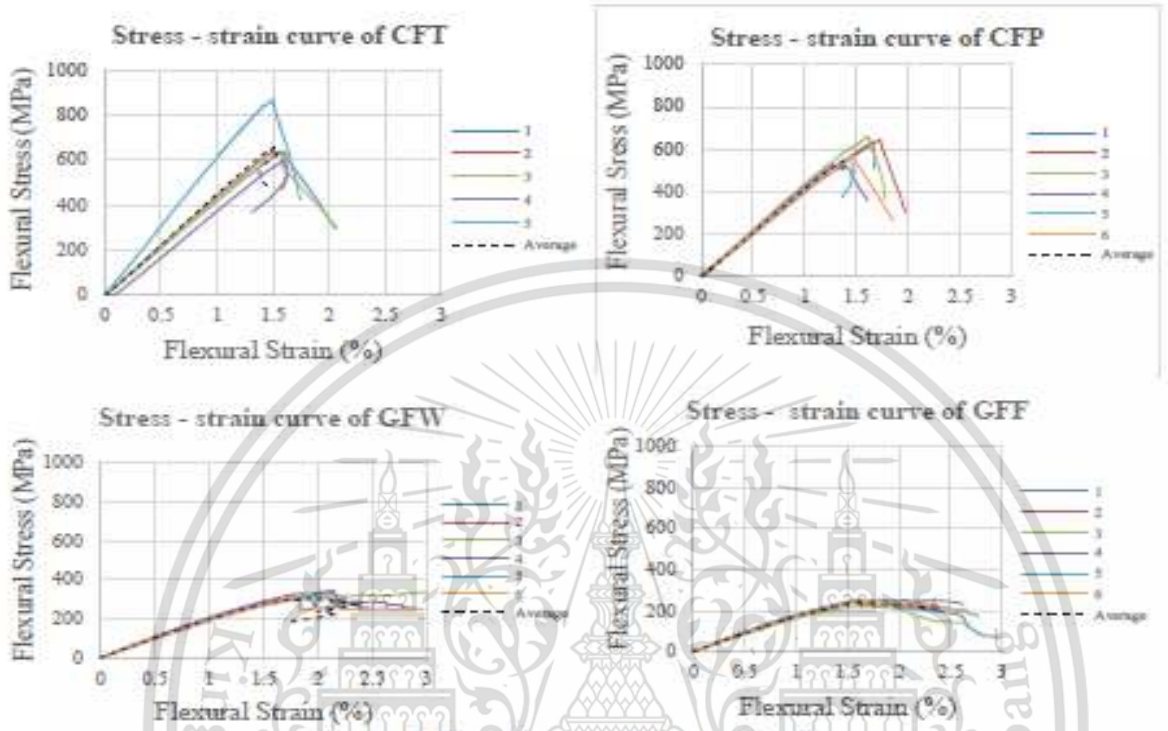


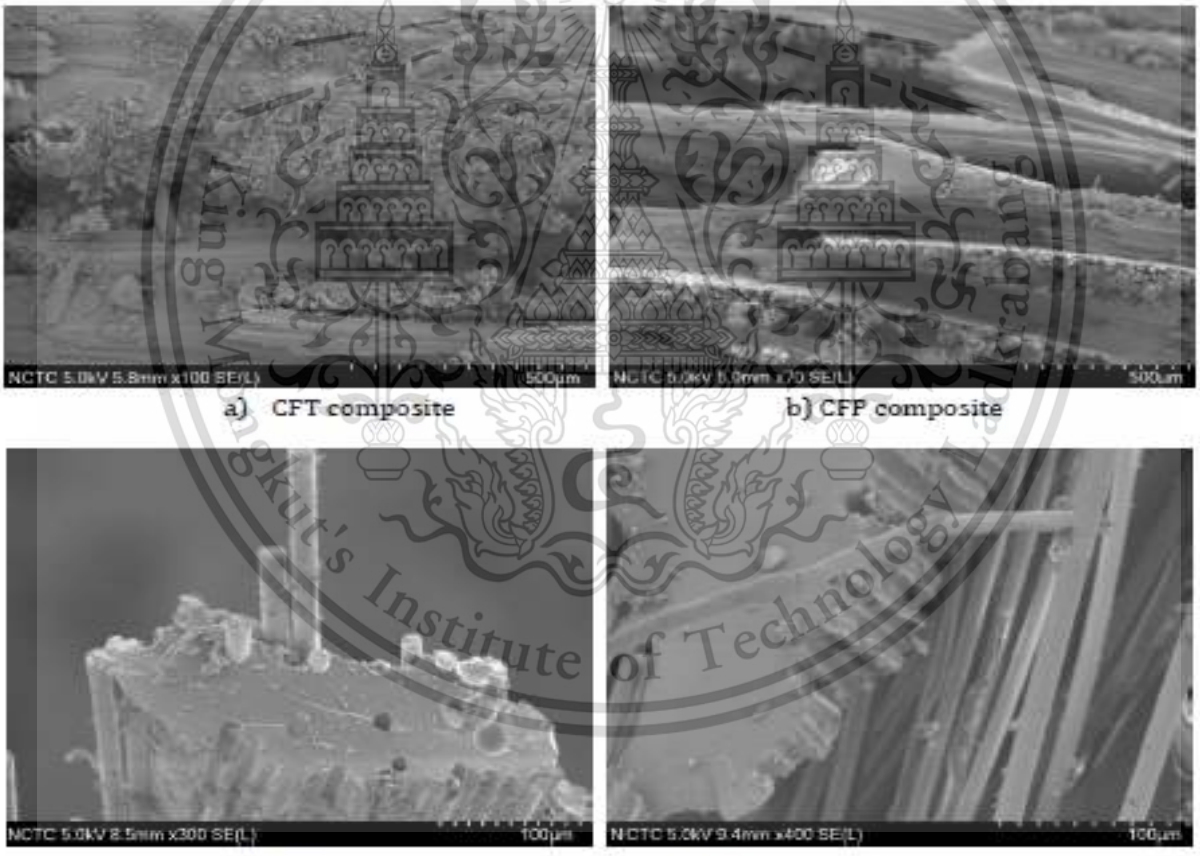
Figure 3. Flexural stress-strain curve for all composites

Figure 4. a) CFT, b) CFP, c) GFW and d) GFF specimens after the flexural test

3.3. FESEM image Analysis

Fracture mechanism of materials is very important role to improve the property or strength because it is extremely depend on the microstructure of interface. In this paper, the microstructure is describe based on tensile test specimens for all four types of composite. Firstly, the main fracture of all samples are come from the smallest and the weakest area of the specimens. The fracture mechanism of glass and carbon are quite different in crack area. Small area for CFT/CFF and quite a large area for

GFW/GFF with complex breakage. CFP fracture breakage shows 0 degree (the same direction with tension load) laid fiber are break almost linearly while 90 degrees across lay up fiber are break by each strand delamination in figure 5 a) and b). The main tension force of CFT and CFP are come from vertical direction laid up fibers, it lead to break resin and fiber. Longitudinal tension force pulls the fiber yarn to separate each filament that make each fiber delamination from bonding. As shown in figure 5 a), CFT is disintegrating into several pieces in the middle, but still hold together matrix and fiber on both edge sides. GFW/GFF is difficult to detect the breakage area because of breakage filament are complicated and breakage area of fibers is longer than that of carbon fibers. The main fracture behavior of GFW/GFF is delamination and pullout the fibers from the bonding matrix as shown in figure 5 c) and d). The matrix bond tightly, but the fibers are delaminate and pull out due to external from through fibers. Delamination of fiber will bring to stand alone each filament, therefore each filament are very easy to break. The middle filament are break first and the breakage are distribute to the both side of fibers, it is the main breakage of fiber in GFW and GFF. Additionally, voids can see in matrix area that comes from the fabricated process through air bubbles that didn't remove very well while lay up process are performed, it becomes voids in composite. Those voids are also one of the weak points to initiate crack in the matrix.



a) CFT composite b) CFP composite

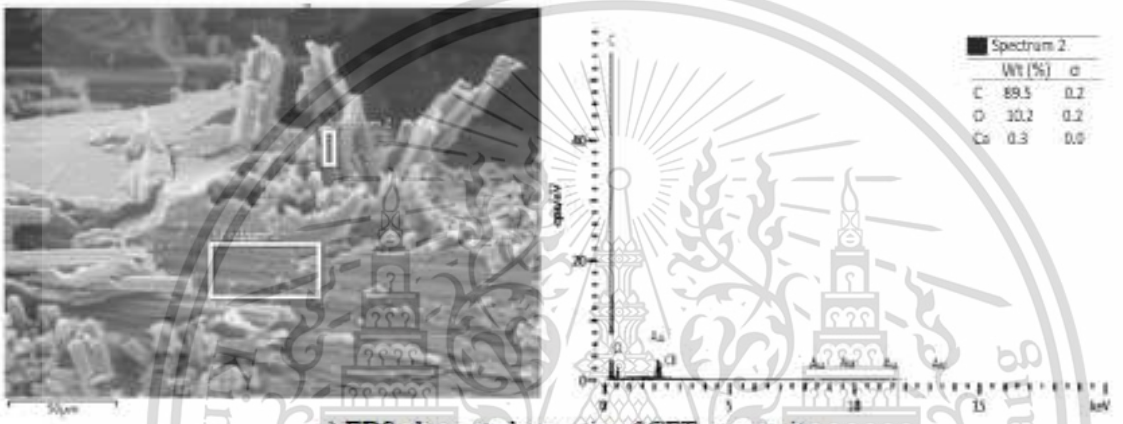
c) GFW d) GFF

Figure 5. FESEM image a) CFW, b) CFP, c) GFW and d) GFF

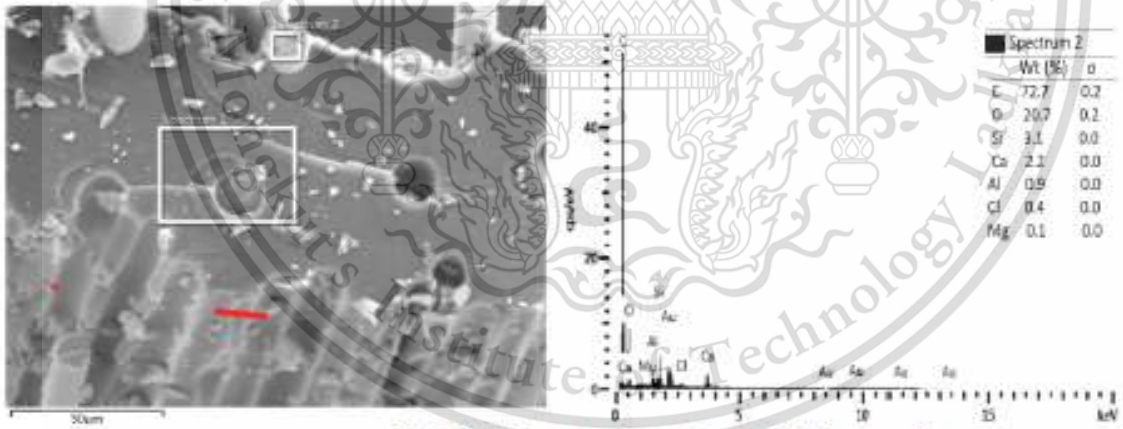
3.4. EDS Analysis

FE-SEM machine with EDS system observed the element composition on the composite CFT and GFW with 60 CPS in figure 6. a) and b). CFT composite spectrum 1 proved that 88% w.t of

carbon, 11.7% of O_2 and 0.3% of calcium containing in composite. Therefore, especially carbon and oxygen are the most important element in this material to play at the main role of carbon composite. GFW has observed 72.7% w.t of C, 20.7% of O_2, 3.1% of Si, 2.2% of Ca, and a few of AL, Cl and Mg respectively in spectrum 1. Thus, element composition of epoxy can be assumed as C, O_2, Ca and Cl. Table 5 is the summary of EDS chemical composite in each GFT and GFW. On the other hand, breakage of CFT prove that it have brittle behaviour due to tension force to the fibers as shown in figure 5. Au containing percent is due to gold spray on sample for electron conductive purpose. Therefore, Au data was ignore in figure 6. A) and b). Al (Alumina) also EDS scanner may count from the stub, which use to stick the sample.



a) EDS element observatio of CFT composite



b) EDS element observation of GFW composite

Figure 6. Element composition of a) CFT and b) GFW composite by using electron dispersive x-ray spectroscopy (FESEM-EDS)

Table 5. EDS result of CFT/GFW composite and each filament

Element/ Composite or Fiber		Element (Weight %)						
		Carbon	Oxygen	Silicone	Ca	Al	Cl	Mg
CFT	Spect:1	87.9	11.7	0.3	-	-	-	-
GFW	Spect:1	72.7	20.7	3.2	2.2	0.9	0.4	0.1

4. Conclusion

The composite of carbon fiber and glass fiber reinforced polymer with an epoxy matrix were test in tensile testing machine, the results show carbon fiber composite (CFT/CFP) was the highest modulus and tensile strength. Approximately 11% of tensile strength are higher than that of GFW/GFF and almost double modulus are achieve in CFT and CFP with 36, 204MPa and 36,604MPa respectively.

Flexural test shows higher modulus than a tensile test with value 46,975 MPa and 41,945MPa for CFT and CFP respectively, meanwhile GFW and GFF are lower than CFT and CFP almost half.

Fracture surface shows, the main character of CFT/CFP are peel off fiber by each bundles and break therefore the breakage area are almost linear in each bundles. This experiments show that CFT/CFP have brittle behaviour. As a contrast, GFW and GFF are delaminate in each filament then each filament are crack after matrix transfer the load. Then the whole fibers are leads to failure. GFW/GFF has a lesser brittle deformation behaviour than CFT/CFP.

Acknowledgement

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Characterization of Carbon Fiber and Glass Fiber's Micro and Nanostructure Using Electron Microscopy, Raman Spectroscopy and XRD Analysis

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Abstract

Nowadays, most manufacturers are looking for the improvement of lightweight parts and other components in the automobile field. Carbon fiber and glass fiber are the most effective materials for their requirement to reduce the weight in vehicles due to their light weight and high tensile strength. The diameter of carbon fiber is 6 µm while glass fiber diameter is 17 µm. The mechanical tensile force of carbon fiber and glass fiber are 430 N and 290 N respectively on fiber alone without matrix. Carbon fibers are gradually smaller in each filament due to tensile force. Approximately 5 mm are elongated for both carbon fiber and glass fiber in tensile test report. In current research, characteristic and tensile force of carbon fiber and glass fiber were investigated by using electron microscopy, Raman spectroscopy and XRD.

Introduction

There are several different materials of carbon fiber such as reconstructed cellulose (Rayon), polyacrylonitrile (PAN) and mesophase pitch. Bascorr [1] investigated that PAN are made from precursor of polymer, Rayon and Pitch are made from oil or lubricant oil. PAN based precursor is commonly produced by wet spinning or air-gap spinning with the diameter range between 5 and 15 µm. There are three steps which can provide the highest carbon fraction of precursor such as, oxidation, carbonization and graphitization. Oxidation is curing process of precursor in atmospheric air over the range of 200°C-300°C. Carbonization temperature is around 1000°C-1900°C. Graphitization temperature is at more than 2000°C. All these steps have limited time to cure them to get higher carbon contain in carbon fiber. More than 98 wt. % of carbon contains carbon fiber was achieved by Kazim et al. [2]. In this research, PAN based carbon fiber was used to investigate the structure of fiber and its morphology. David et al. [3] investigated x-ray diffraction of atom of fiber. And he calculated crystal size (*Lc*) by using Sherrer equation and spacing (d_{002}) by Bragg equation with the angle of 2θ scattering. And also found graphitization for graphite crystal structure of carbon fiber. The main tools to observe fiber morphology are TEM and Raman spectroscopy. Carla et al. [4] studied about the structure by Raman spectroscopy and graphene layers of fiber and the crystal of fiber filament by TEM. Jaleel et al. [5] studied about treatment material in two methods to make a treatment on the fiber such as epoxy on the fiber surface are release out by de-sizing process and by special solutions process. Therefore, the composite bonding between fiber and resin are 20% higher than normal composite in the properties of flexural mechanical test. Jing et al. [6] studied about sizing and material treatment. As the result, treatment material improve interfacial bonding and also the mechanical properties. P. Karin et al. [7] observed particle matters (PMs), average agglomerated particle diameter sizes of diesel and primary particle of biodiesel are 50-500 nm size and 20-50 nm, respectively. Transmission electron microscopy (TEM) was used to investigate the size of PM. P. Karin et al. [8] used TEM to observe particle matters size from biodiesel engine and diesel engine with different speed RPM. And investigated that the average of primary nanoparticles size are approximately 34 nm for biodiesel and 32 nm for diesel engine's PMs respectively. P. Karin et al.[9], investigated soot contamination in oil was tested in four-ball tribology with friction and wear resistance test. As a result, appropriate particle size are 20-80 nm primary particles and 80-300 nm of agglomerated particles. Very big particles will jump out and very small particle will not effect on wear compare to oil film thickness. Therefore, electron microscopy is one of the

most important tool to study morphology and their behavior. Chawla [10] investigated that glass fiber is also one of useful material in lightweight component due to its extreme flexible characteristic. Glass fiber are produced based on Silica, however, varieties of different chemical composition glass fiber are available in common. Most of glass fibers are based on the highest amount of silica including small amount of calcium, sodium, aluminum and iron. There are several types of glass fiber based on different chemical composition such as E-glass, S-glass and C-glass with different properties. B. Karmakar et al. [11] investigated the main component element in glass fiber by using Raman spectroscopy (RS) and Fourier-transform infrared spectroscopy (FT-IR). In Raman spectroscopy, element bonding are shown by vibration mode base on wavelength of atom activation. It describe the structure element and atom bonding link. IR peak defines the element group that holds together. However, in case of fibers and composite, micro and nanostructures are not understood well, it induces to investigate the properties and fracture mechanism of composite with different type of base material. In current paper, the main focus point is to study morphology of neat carbon fiber and glass fiber.

Experimental Setup

In this paper, two types of carbon fiber and two types of glass fiber with different patterns were used and tested mechanical test in tensile machine. Figure 1 show the fiber type and table 1 shows the fiber information with their respective properties. And the same sample materials of fiber are used to make a treatment and deposited diamond like carbon (DLC) by using plasma assistance chemical vapor deposition (PACVD) to investigate mechanical properties and surface morphology. Two types of fiber CFT and GFW were deposited DLC.

FIGURE 1 Naked fibers of (a) CFT, (b) CFP, (c) GFW and (d) GFF.

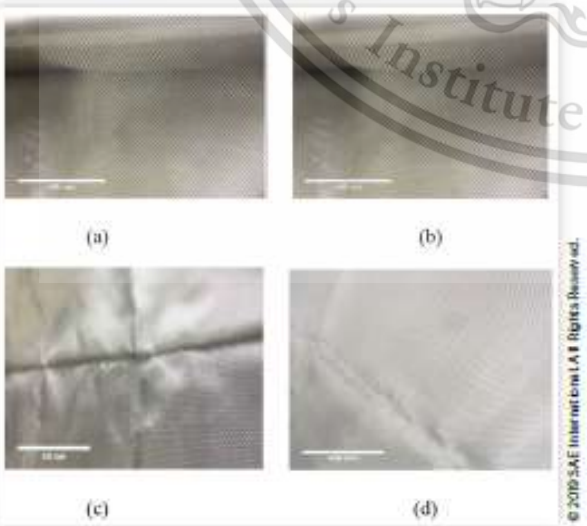


TABLE 1 Materials properties.

No.	Material and product name	Weight (g/m^2)	Thickness (mm)	Name in this paper
1	Carbon Fiber Twill	240	0.31	CFT
2	Carbon Fiber Plain	200	0.3	CFP
3	Glass Fiber	400	0.3	GFW
4	Glass Fabrics	200	0.2	GFF

Deposited fibers were tested in tensile machine to know their properties.

Fibers are cut to be the shape of rectangle with 127 mm length and 12.7 mm width. The thickness of samples are based on supplier's material thickness as shown in table 1. Current research used tape to cover the fabric not to freak out from the pattern at both side. The prepared specimens are tested in tensile machine INSTRON 8801. The tested parameter are load cell 20 KN and test speed 50 mm/min for all materials with the same operation function at room temperature 24°C, humidity 47%. Two layers tape was prepared the same size as specimens and tested in the same machine and parameter. The average tape result of 6 specimens are used to subtract from fiber result that stick with tape. DLC coated materials are tested with the same situation named as naked fiber. Six specimens were tested in each type of material as listed 1-6 by different colour in figure 3.

The same fabric pattern of fiber are prepared by cutting with scissor and covered by tape not to freak out the pattern of fibers while treatment are performing. Firstly, the cut fabrics are put in the bowl and applied ethanol to sock out unclean particles from the surface of fiber for 30 mins and clean with distilled water for 10 minutes. Then cleaned fabric are put into oven to cure at 80° C for one and half hours. After cured, fibers are fit to the holder of the sample. Cleaned with Argon (Ar) for 10 mins with flow rate 20 cm^3/min, pressure 3 Pa and voltage 3.5 kV in PACVD machine. After cleaned with argon, applied Acetylene (C_2H_2) for 30 minutes with flow rate of 20 cm^3/min, 3Pa pressure with 3.5kV voltage. Raman spectroscopy (JASCO machine) and x-ray diffraction machine use for analyze the structure of carbon fiber and glass fiber. SEM/

FIGURE 2 Appearance of the Fracture of fiber after tensile test (a) GFF while tensile test, (b) CFP while tensile test and (c) GFW & CFT after tensile test.

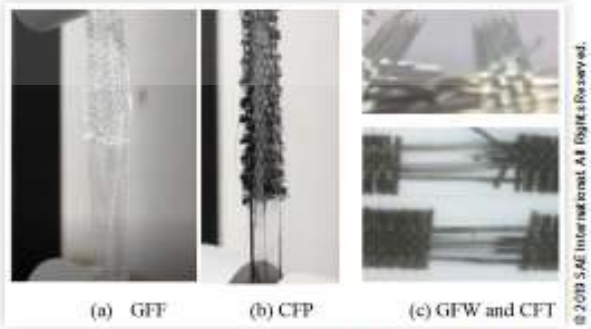
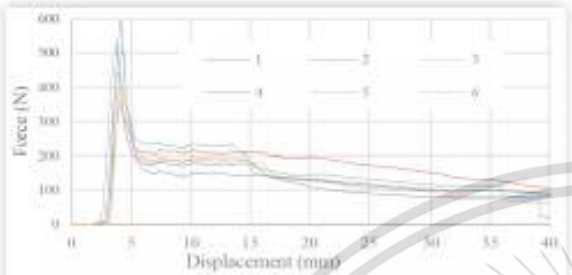
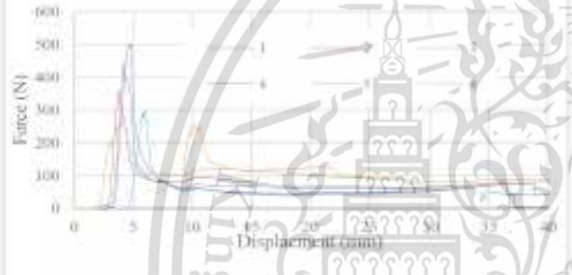


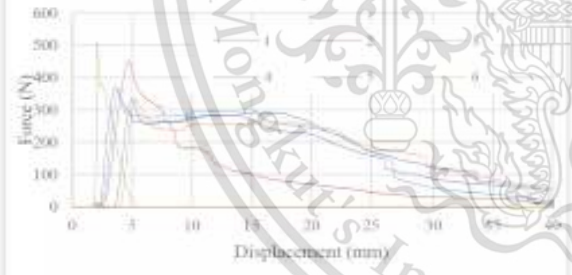
FIGURE 3 Tensile test results for all naked fiber Force (N) and displacement (mm) curves of (a) CFT, (b) CFP, (c) GFW and (d) GFF each specimen in each color.



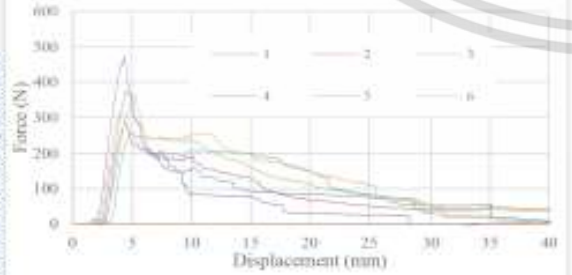
(a) Force - Displacement curve of CFT



(b) Force - Displacement curve of CFP

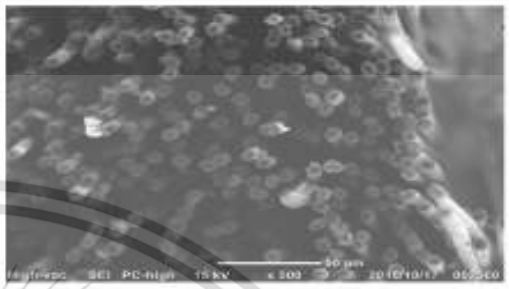


(c) Force - Displacement Curve for GFW

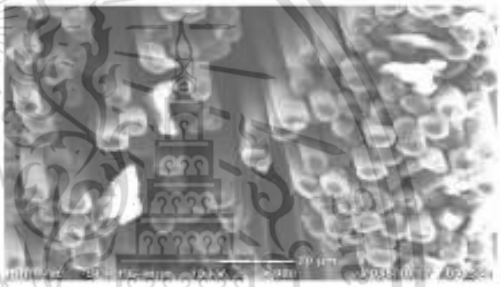


(d) Force - Displacement Curve of GFF

FIGURE 4 SEM image of (a) CFT, (b) CFP, (c) GFW and (d) GFF filaments.

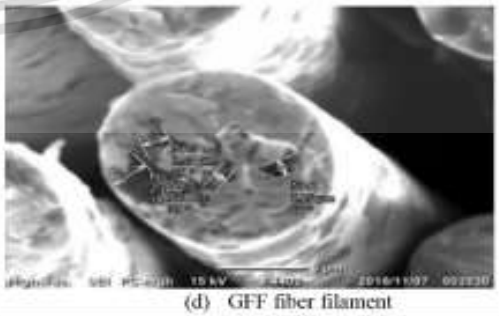


(a) CFT carbon fiber filament



(b) CFP carbon fiber filament

(c) GFW fiber filament



(d) GFF fiber filament

EDX (JEOL - JCM 6000) with 15 kV is used and TEM observed the behavior of fibers and their chemical composition.

Result and Discussion

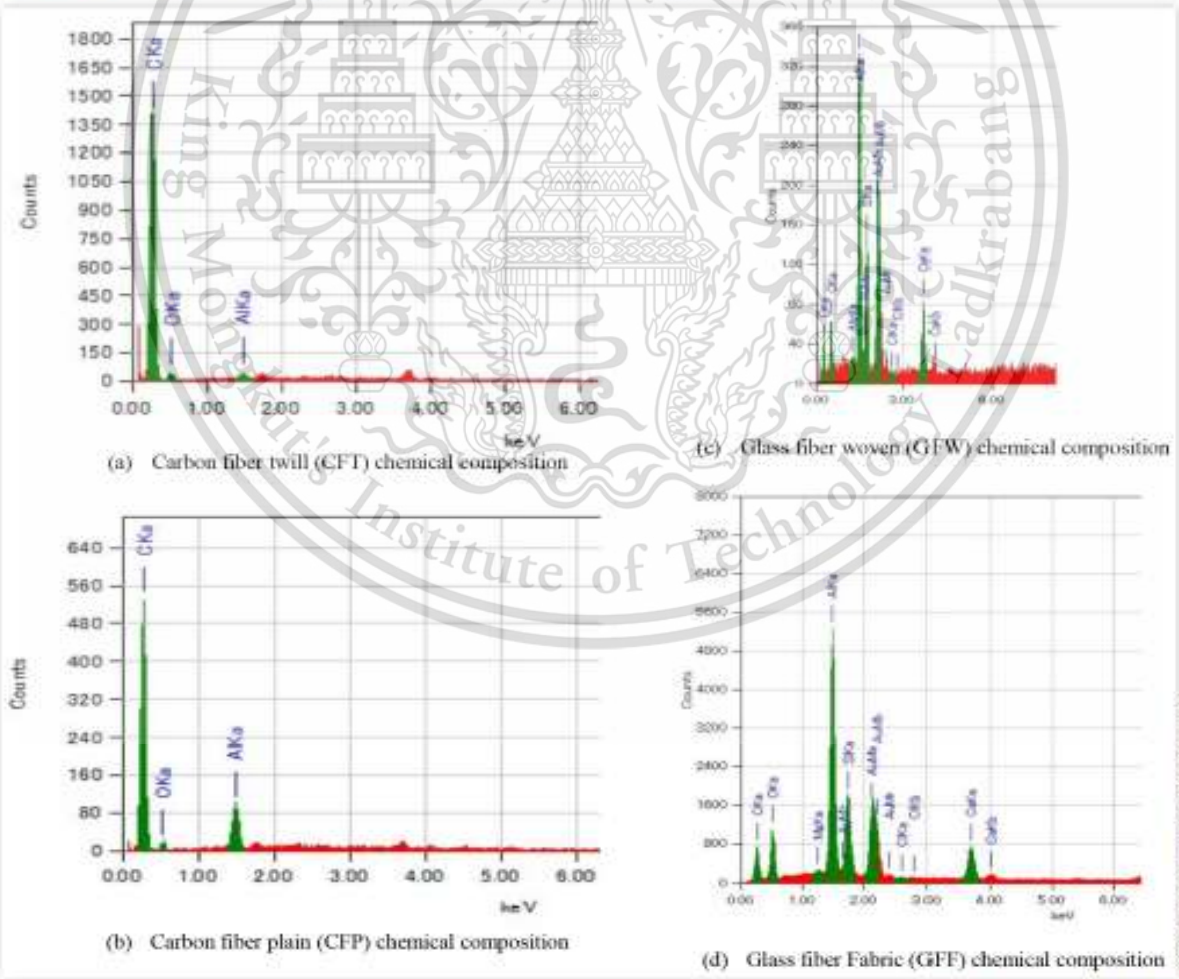
Mechanical Property

The mechanical properties of each fiber are as shown in figures 3. Graph shows CFT have the highest force with around 430 N in average from six specimens while CFP is around 200 N. Different patterns much affect to the properties of material. More than half of force is lower in CFP compare to CFT. Therefore, twill weave pattern of fabric leads to the better properties. In glass fiber, the results are not much different between two fibers. GFW is almost 300 N force while GFF is around 310 N. GFF has higher force because of the fiber bundles of GFF are smaller than GFW. Therefore, more fiber bundles were placed in the same size specimen, it brought to the higher tensile force. In the case of elongation displacement, all of materials are almost the same displacement around 5 mm elongation. Figure 2 shows the breakage of fiber while tensile test are performing. After break the tape, diameter of verticals axis carbon fiber are reduce that bring to break fiber. The main direction that can resist external tension force is vertical axis fiber. With the help of tape, horizontal laid fabrics are stick together with vertical axis fiber.

Electron Microscopy

The fiber surface and cross section area are observed by scanning electron microscopy (SEM) scanning electron microscopy. The surface of CFT and CFP have some defects on the surface, but in the cross section area, defects area cannot observe by SEM. One of the reason might be due to

FIGURE 5 SEM-EDX chemical analysis of (a) CFT, (b) CFP, (c) GFW and (d) GFF.



(a) Carbon fiber twill (CFT) chemical composition

(b) Carbon fiber plain (CFP) chemical composition

(c) Glass fiber woven (GFW) chemical composition

(d) Glass fiber Fabric (GFF) chemical composition

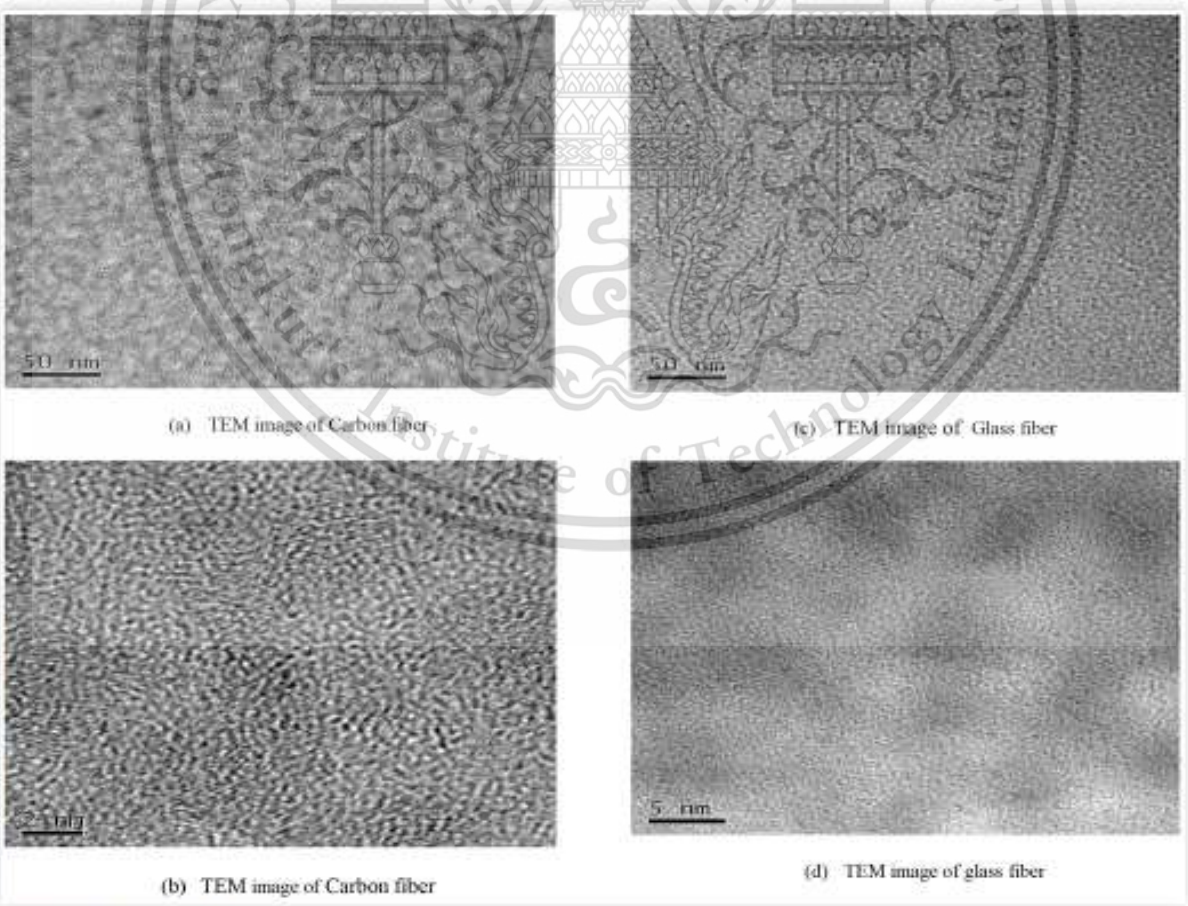
very small porous that cannot detect by SEM. In the case of glass fiber, the defective surface can see easily and several porous in the cross section area also observed by SEM. The porous sizes are between 0.8 to 2.2 µm base on SEM information as shown in figure 4 (d). This is one of the main reason to make the mechanical properties drop in glass fiber. Also because of its natural softness and flexible properties. SEM-EDS shows the chemical composition of all materials. In both CFT and CFP carbon fiber, the highest percentage of chemical elements are carbon with 92-93% percent and another element contain in this fiber is oxygen with 3-4%. In glass fiber, both of glass fiber are also same. Silica (Si) is the highest percentage containing element. Oxygen (O_2) also one of the highest containing element after Si. C, Mg and Ca element also contain in both glass fiber type.

Transmission electron microscopy (TEM) reveals the structure of carbon fiber and glass fiber as shown in figure 6. To investigate the structure of carbon fiber and glass fiber, focused ion beam (FIB) cutting method was used to cut the material in the size of more than 100 nm square meter from the center of fiber filament. TEM detects the behavior of carbon fiber and glass fiber. The first two pictures are carbon fiber with low (50 nm) and high (2 nm) magnification as shown in figure 6 (a) and (b). In figure 6 (b), graphene layers of carbon hexagonal chain and there are several disorder fibers with random grapheme layers from defected crystal that create disorder band (D-band) in Raman spectroscopy. Graphene are laid with the same gap between each layers in some parts of fiber filament. There was no signs or structure are shown for glass fiber both in low and high magnification as shown in figure 6 (c) and (d).

Raman Spectroscopy

Raman spectroscopy is one the main tool that can give the structure information of carbon fiber and glass fiber. Figure 7 shows Raman shift of carbon fiber two type. This two peaks are fit using origin 6.0 peak fitting to observe the exact value of each peak, full width at half maximum (FWHM) and I_D/I_G. In Raman graph, there are two peaks at 1350 cm^{-1} (D-band) and another one is at 1590 cm^{-1} (G-band). D band came from defected crystal without symmetry line and it indicates the disorder or foreign material are bonding in the place of carbon

FIGURE 6 TEM images of fiber (a) carbon fiber, (b) glass fiber, (c) glass fiber and (d) glass fiber.



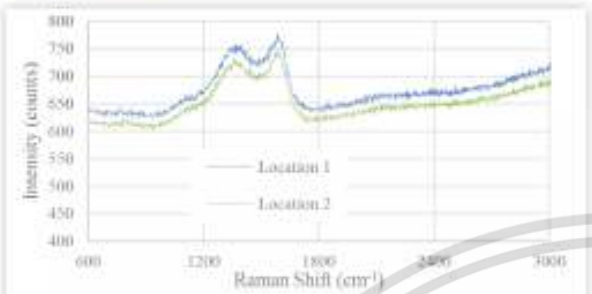
(a) TEM image of Carbon fiber

(c) TEM image of Glass fiber

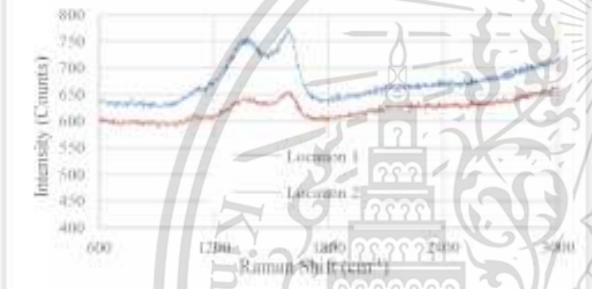
(b) TEM image of Carbon fiber

(d) TEM image of glass fiber

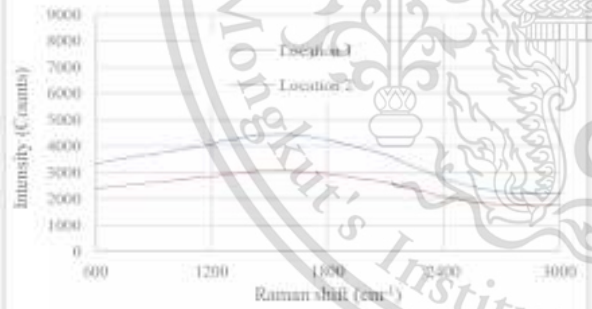
FIGURE 7 Raman spectra graph of (a) CFT, (b) CFP, (c) GFW and (d) GFF.



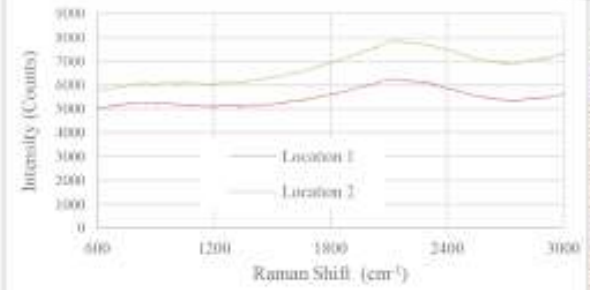
(a) Raman Shift of Raw Carbon Fiber Twill (CFT)



(b) Raman shift of raw carbon fiber plain (CFP)



(c) Raman Shift of raw glass fiber woven (GFW)



(d) Raman Shift of raw glass fiber fabric (GFF)

TABLE 2 Raman Spectroscopy analysis from origin peak fit.

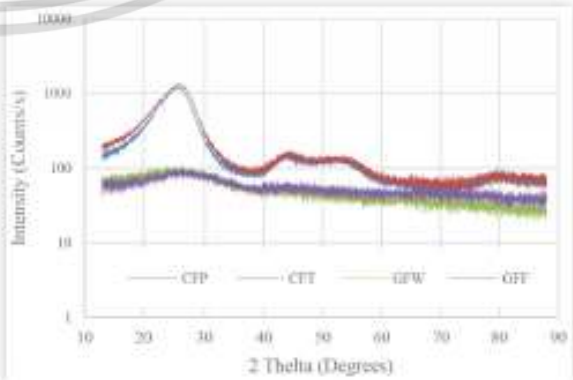
Material	Peak	Center of Peak(cm^{-1})	FWHM (cm^{-1})	I_D/I_G
CFT1	D - Band	1382.43	308.69	0.868472
	G - Band	1591.79	103.03	
CFT2	D - Band	1390.46	260.58	0.873726
	G - Band	1591.41	108.56	
CFP1	D - Band	1376.04	243.40	0.869422
	G - Band	1582.71	110.02	
CFP2	D - Band	1338.56	440.77	0.851065
	G - Band	1572.82	91.46	

atom bonding. And G band peak indicates carbon to carbon bonding (C-C) structure in graphene layer with the first order of consonance frequency based on the previous researcher. The results reflected to the behavior of carbon fiber crystal. In D-band, peak is around 1338-1390 cm^{-1} in Raman spectra and around 1571-1591 cm^{-1} Raman shift for G-band peak. In CFT, the highest Raman is around 1591 cm^{-1} at G-band while 1572-1582 cm^{-1} for CFP shown in table 2. FWHM of carbon fiber have smaller value for G- band with around 100 cm^{-1}, it is almost 70% lesser than D-band. In the case of glass fiber, there are no specific peaks in the Raman spectroscopy. Figure 7, c and d shows glass fiber structure in the graph. There is no carbon bonding in this graph and it indicate amorphous structure in the graph of glass fiber.

X-Ray Diffraction Spectroscopy

X-ray diffraction is also one of the tool to define the material type and to find the spacing between the grapheme and crystal size base on the material structure. In this research all of naked materials are checked in XRD with chi integration type, CuKα X ray of 1.54056 Å and frame angle is 2 theta. In the graph of figure 8, the narrow peak in carbon fiber twill and plain both at 25 degree with 2 theta of angles. According to

FIGURE 8 XRD pattern for all carbon fiber and glass fiber.



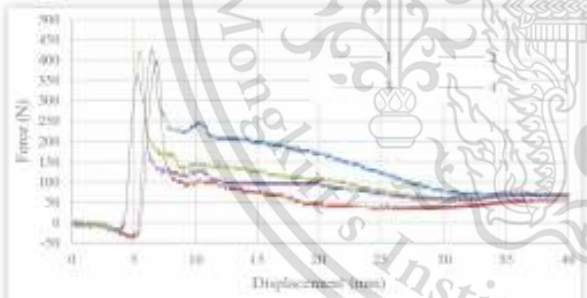
ICDD, XRD pattern of fiber exhibited peaks 26.5°, 44.3° and 54.5° can be assigned to planes (002), (101) and (004) in graphite structure respectively. It reveals the structure of carbon crystal are there in carbon fiber filament with graphite structure arrangement of carbon. For glass fiber, there are no peak in the graph.

Diamond Like Carbon

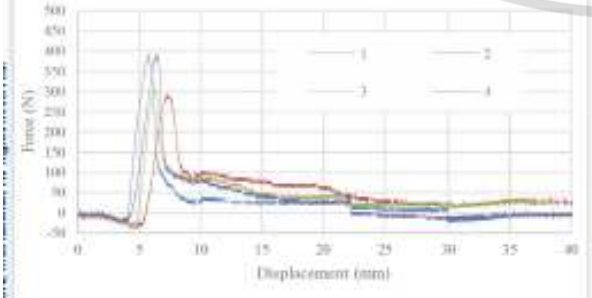
Coated fiber are test in mechanical test and compare to the naked fiber properties. The mechanical properties of carbon fiber and glass fiber woven are shown in figure 9. Four specimens of each carbon and glass fiber were tested in this paper. In Carbon fiber, average maximum force is around 300 N with approximately 5 mm elongation while glass fiber woven are 265 N maximum force with around 5 mm elongation. The fiber failure mode are same as before naked fabric. The diameter of filament become smaller and smaller that bring to the weakness fiber until break fiber. One of the weakness point of deposition of fiber is the whole filament diameter cannot wet well by the plasma coating DLC. Inside of the fabric pattern are not wet well with DLC. That cannot make the fiber to reinforce more than original naked fiber.

Figure 10 is the summary result of carbon fiber twill weave and glass fiber woven in naked and DLC coated. Based on the result, DLC coated fabric maximum forces are less than naked fiber while elongation are almost same in both carbon

FIGURE 9 Tensile test force and displacement of carbon fiber twill weave and glass fiber woven coated with DLC.



(a) DLC coated carbon fiber



(b) DLC coated glass fiber

FIGURE 10 Tensile test summary of naked fabric and DLC Coated fabric.

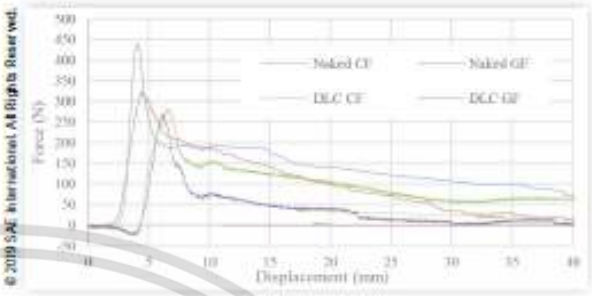


TABLE 3 Force and displacement of naked and DLC coated fiber.

	Force (N)	Displacement (mm)
CF	440.87	4.05
GF	271.11	5.03
DLC - CF	280.78	6.5
DLC - GF	261.72	6.27

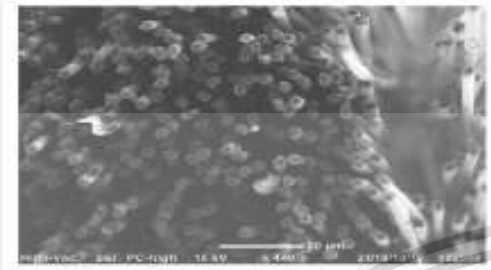
fiber and glass fiber. In carbon fiber twill weave, the maximum force of DLC coated fiber are dramatically decrease to be around 36% from naked material tensile test result. And around 3.5% maximum force are decrease in glass fiber woven. The main failure mode of coated fiber are due to the treatment process. In treatment process, the fibers are put into ethanol for 30 mins to sock all of the defective and sizing material. Therefore the diameter of carbon fiber are decrease and all of foreign element on fiber surface are remove from filament surface. That bring weakness in fabric test. But it might enhance bonding behavior between fibers and matrixes. The summary of tensile force and displacement are shown in table 3.

Both naked and coated fiber of carbon and glass fiber woven were investigated by using SEM image analysis to observe the difference of coated and uncoated DLC. Figure 11 shows uncoated naked fiber have several defects on the surface of fiber. In contrast, coated fiber are smoother and reduce defective on the surface, but coated DLC become small particle on the surface of fiber as shown in figure 11 (b) and (d). The graph shows negative value because the tape were collapsed, therefore, tensile load try to make straight to the collapse tape, it make negative value in the graph.

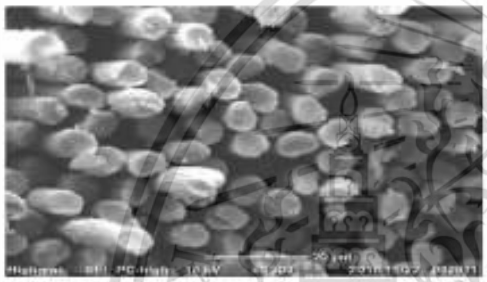
Summary

SEM images show the morphology of each fiber on the surface with several defectives material on glass fiber than carbon fiber that make the lower properties compare to carbon fiber in tensile test. Maximum force of glass fiber are lower 32% than that of carbon fiber maximum force. TEM detects the

FIGURE 11 SEM images of glass fiber (a) naked CF, (b) DLC coated CF, (c) naked GF and (d) DLC coated GF.

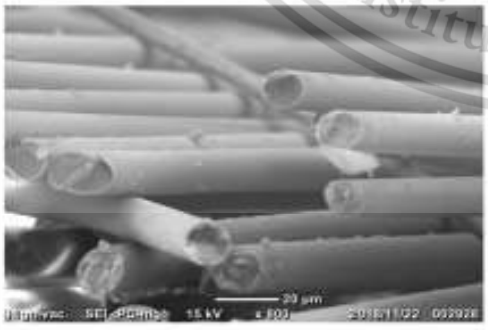


(a) Carbon fiber before coated DLC



(b) Carbon fiber after deposited DLC

(c) Glass fiber woven before coated



(d) Glass fiber woven after deposited DLC

structure of carbon fiber and glass fiber grapheme arrangement which is reflect to Raman spectroscopy. Mechanical properties of carbon fiber is higher than glass fiber in naked fabric and also in DLC coated fiber. Raman spectroscopy shows the structure of material D-band with around 1380 cm^{-1} and G-band with 1581 cm^{-1}. The graphite structure of carbon bonding are constructed in this carbon fiber. Treatment fiber is one of the tool that can de-sizing the material from the surface. DLC will helps to improve mechanical properties if DLC coating wet the whole filament surface. SEM finalize coated and un-coted DLC, un-coated fiber have several defective and coated DLC fiber have smooth surface with less defective surface compare to uncoated fiber.

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APPENDIX C

Thesis Defense Question and Answer

1. Professor Dr. Naoto Ohtake
   1. Why the Raman spectroscopy graph shows slightly different slope magnitude between CFP and CFT at the range of 2000-3000 (cm^{-1}) Raman shift?

   ⇨ The slope magnitude are slightly different between CFP and CFT at 2000-3000 cm^{-1} Raman shift because sizing material containing percent are different between the two materials CFP and CFT. Most of the fiber manufacturer used water base epoxy to coat the fiber surface with the purpose of preventing fiber surface or for easy handling purpose in further process such as oxidation, carbonization and graphitization. Ferrari and Robertson [51] also show their investigation of polymeric C-H bonding at 2100-2200cm^{-1} and CH_x stretching at 2920cm^{-1} from Raman spectroscopy.

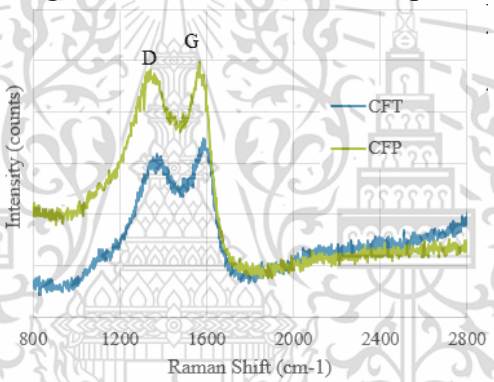

Figure (1) Raman Spectroscopy (page 15 from My thesis presentation)

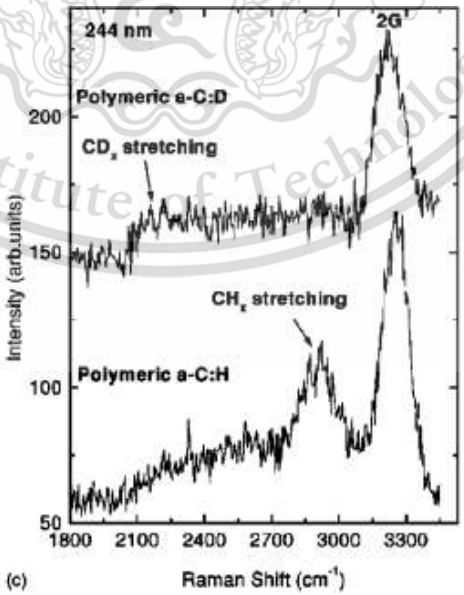

Figure (2) Raman spectroscopy [51]

Remark: Add at Chapter (4) Result and Discussion, page no. 46, 47.

2. What kind of material do you used for de-sizing fiber or fiber treatment and what kind of the chemical species like oxygen or OH is there on the fiber surface.

⇨ Acetone are used to make a treatment or de-sizing epoxy coating from fiber surface. The chemical composition of carbon fiber are carbon (92-99% in wt.) and oxygen (2-7%) The fiber surface are not analyze by FTIR for functional group, therefore the main functional group are not determine after treatment fiber. But there are some FTIR result of untreated fiber which shown the functional group of O-H as shown in figure (3). This functional group might be remove by acetone while treatment process, that can make the weakness of bonding between fiber and matrix. This might be one of the reason for dropping the mechanical properties of composite after treatment fiber.

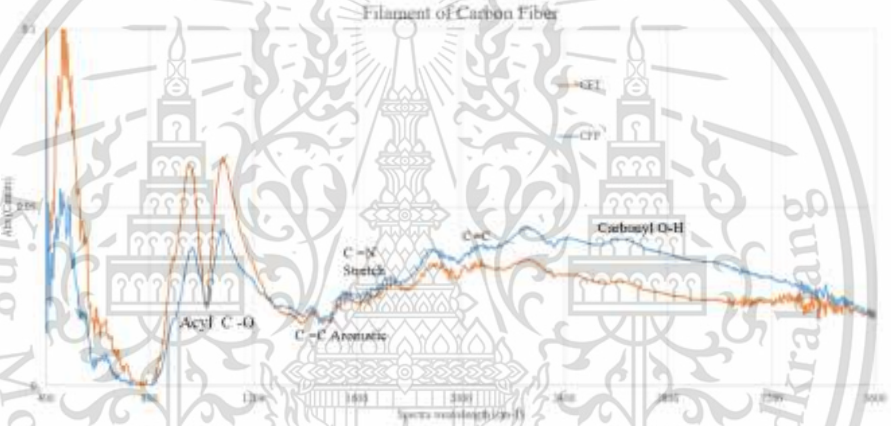

Figure (3) FTIR result of carbon fiber filament.

Remark: Add in chapter (4) Result and Discussion page no. 48 and 56

3. Is it possible to measure the surface roughness each fiber of carbon fiber and glass fiber after treatment or is there any method to measure the roughness?

⇨ Yes, we have several method to measure the surface roughness of fibers. It is very good to know the roughness of fiber that can affect mechanical interlocking between fiber and matrix, lead to mechanical properties of composite. The main effective tool is atomic force microscope (AFM) which can measure the surface roughness. But in this research, the surface roughness are not measure.

Remark: Add in chapter (4) Result and Discussion page no. 56,

4. What is your future work on this topic?

⇨ There are several plan to do more investigation for improvement in composite field. Mainly I have two plan, one is to measure the roughness of fiber surface before and

after a treatment because the mechanical properties of composite are much depend on the interfacial bonding or mechanical interlocking between fiber and matrix. High roughness surface give higher mechanical properties due to good mechanical interlocking between fiber and matrix. On the other hand, low roughness surface give lower mechanical interlocking that can make the weakness of interfacial bonding. Another future work is to control the porosity of composite by using vacuum or other special tool while the composite are fabricating because porosity make effects to the mechanical properties and it might be one of the reason to start crack initiation while mechanical test are apply. If we use special tool or infusion method to make a composite, the composite might be looking good at both side, get rid of porosity and more compatible. Therefore, I would like to make a further research or suggest to make this kind of method for further improvement in composite field.

Remark: Add in chapter (5) Conclusion, page 88

2. Asst.Prof. Dr. Chinda Charoenphonphanich

You have compare three kind of temperature parameter RT, 80ºC and 120ºC, what do you means room temperature (RT) or how many degree Celsius? To avoid the confusion between this thesis reader of different location and different whether?

⇨ Room temperature is base on Thailand temperature and it might be around 30ºC. RT are replace with 30ºC in the whole thesis book.

Remark: Change all of the RT word to be 30ºC in the whole thesis

3. Dr. Sompong Srimanosaowapak
   1. Please explain the reason behind why you choose this material or do you have any grade consideration and did you get the specification from the company. Are the mechanical properties match with your result from tensile test. Do you think glass fiber is the best mechanical properties in the market?

⇨ Most of researcher requests special material from company to make a research deeply but for me I would like to study the fiber that available in Thailand market which is commercial material. Therefore, I bought all of my material from Concrete Composite Company. Most of the specification are receive from company and glass fiber properties result are similar to the supplier specification while carbon fiber mechanical properties did not show in specification. Glass fiber is not the best one, the best mechanical properties are come from carbon fiber due to its stiffness and brittle. Compare to carbon fiber, tensile strength and modulus of glass fiber are quite low about half or more than half. But some researcher make hybrid composite which are mix with

carbon fiber and glass fiber in a composite, which make the higher mechanical properties.

Remark: Add in chapter (3) Methodology, section 3.1, page no. 23

2. Do you think you have a good preparation of composite from hand layup or do you think your composite are perfect. Which kind of tool do you used to inspect the composite to know whether the composite are good enough or not like ultrasonic scan or x-ray scan?

⇨ For better quality control, the composite are need to check deeply not only in visual by eye but also by scanning machine which can investigate the defects in the composite. In current research, only human eyes are used to check the defects or the imperfection of composite. But in the future, composite material usage are increase widely across the whole world in many field, therefore better or advance tool such as ultrasonic scan are need to use to control the quality. Some of researcher also used Ultrosonic sensor or scan to investigate, the porosity of composite, microcrack, defects area, delaminations area and impact damage[52,53]. These are the tool that will use to inspect the composite in the near future.

Remark: Add in chapter (3) Methodology, section 3.2, page no. 35

Another comment

- Change Thesis abstract
  ⇨ Yes, The content of thesis abstract was changed as shown in page IV.

- Need correction for Grammar
  ⇨ Most of grammar are change as much as possible