



Permeability Barriers for Composites

Ageing Mechanisms and Surface Treatment of Polymeric Matrix
Composites under Cyclic Load

Henock Dandena Dibaba

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<p>Abstract:</p> <p>The thesis scope was to study and experiment the hydrolytic degradation of composite laminates under cyclic loads using different hydrolytic permeability barrier methods in short period of time. Four sample groups were prepared for the studies that were treated in two types of barrier methods; Aluminium foil reinforced and Durester CG spray paint. Experiments were performed on the Testometric testing machine for cyclic loading following placing samples in water container for hydrolytic mass up-taking inside an oven within a specified temperature. The process was repeatedly applied for a number of time and data's were collected accordingly. After completing the experiment, results were studied and compared according to their physical degradation, Testometric cyclic loading data, and water up-taking masses. In addition, the role of lamination methods and cost effectiveness of anti-corrosive methods were included for analysis. As a result saturation time for water up-taking was recorder at 127 Hours after the initial testing. The slope changes of the final and initial tests graphs, retrieved by sample laminates prepared using vacuum bagging and embedded with aluminium foils was 32% in slope decline and recorded as the highest . On the contrary, Durester CG shows 13 % of slope declination in the cyclic loading test graph which shows better mechanical performance. Moreover, Durester CG shows a 1.73 % increase in mass up-taking which was better result next to treated glass fibre laminates having 2.88%. As a summery , Durester CG treated glass fibre laminates shows a better coating or barrier method of all the rest laminates that were tested.</p>	
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Contents

1. INTRODUCTION	1
1.1 Background.....	1
1.2 Objective.....	2
2. LITERATURE REVIEW.....	2
2.1 Composite materials.....	2
2.2 Classification of Composites.....	5
2.3 The Aging of Polymeric Matrix Composites	6
2.4 Mechanics of Composites.....	8
2.5 Failure of Mechanisms in Fibre-Reinforced Composites.....	9
2.5.1 Failure Modes in Unidirectional Composite	10
2.6 Through-Thickness Compressive Properties of Laminate	11
2.7 Viscoelasticity	12
2.8 Ageing and Effective Time	13
2.9 Diffusion	14
2.9.1 Mathematics of Diffusion	15
2.9.2 Hydrolytic Degradation.....	16
2.9.3 Mass Up-taking Measurement Method	17
2.10 Factors Affecting Diffusion in Polymer Matrix	18
2.10.1 Polymer Chemistry	18
2.10.2 Porosity and Voids.....	18
2.10.3 Filler Particles	18
2.10.4 Mechanical Stress and Strains	20
2.10.5 Macro-Scale Interfaces.....	20
3. MATERIALS AND METHOD.....	21
3.1 Initial Material Preparation	22
3.1.1 Vinyl Ester	23
3.1.2 E-2 Glass Fibre	23
3.2 Protective Materials	23
3.2.1 Durester CG	24
3.2.2 Aluminium Foil Reinforcement.....	24
3.3 Sample Preparation Method	25
3.3.1 Vacuum Bag Lamination Methods	25

3.3.2 Compression Moulding.....	26
3.4 Cyclic Three-point Bending Test	26
4. RESULTS	27
4.1 Water up-taking and diffusion rate.....	27
4.2 Cyclic three-point bending test results.....	29
4.2.1 Cyclic three-point bending test result for Aluminium-Embedded Compression moulded samples	29
4.2.2 Cyclic three-point bending test result of glass fibre laminates	31
4.2.3 Cyclic three-point bending results of Durester CG treated laminates	33
4.2.4 Cyclic three-point bending test results of aluminium embedded Vacuum Bagged laminates	35
4 DISCUSSION	37
6 CONCLUTION	39
REFERENCE.....	41
APENDIX I.....	43
APENDIX II	44

Figures

Figure 1: Lamina-Laminate Definitions.....	3
Figure 2: Specific strength as a function of time of use of materials. (Kaw, 2006).....	3
Figure 3 fibre laminate (stack of plies), illustrating the nomenclature system.....	8
Figure 4: Macroscopic (A, B) and microscopic (a, b) scales of observation in a unidirectional composite layer. (Daniel I. M., 2006).....	9
Figure 5: Distribution of interlaminar normal stress and interlaminar shear stress in [0/90], laminate under axial tension.	10
Figure 6: Different types of loading in a unidirectional laminate (Daniel I. M., 2006)	11
Figure 7: short-beam shear test set up (Daniel I. M., 2006)	12
Figure 8: Creep and recovery Stress and strain Vs time. Taken from: (Lakes, 2009)	13
Figure 9: an expression of diffusion through a membrane.....	15
Figure 10: example of concentration distance curve of a typical distribution at three successive time.	16
Figure 11: Diffusion in multi-layer systems (i) in parallel (i) Transversal direction. Taken from: (Duncan, Urquhart, & Roberts, 2005).....	21
Figure 12: (a) typical Vacuum bagging layup schematic. Taken from: (Craft, 2014) and (b) vacuum bagging setup.....	25
Figure 13: Compression moulding machine in Arcada UAS Production Laboratory.	26
Figure 14: Three-point bending setup. (solution, 2016).....	27
Figure 15: Percentile change in mass up-taking Vs time (at 90 °C , 1 bar)	28
Figure 16: Ideal fickian curve	28
Figure 17: Diffusion rate.....	29
Figure 18: (a) Initial cyclic results of laminates and (b) graph segment.....	30
Figure 19: (a) Final cyclic results of laminates and (b) graph segment.....	30
Figure 20: Physical changes. (a) Before testing and (b) after testing result	31
Figure 21: (a) Initial cyclic results of laminates and (b) graph segment.....	32
Figure 22: (a) Final cyclic results of laminates and (b) graph segment.....	32
Figure 23: Physical changes. (a) Before testing and (b) after testing result	33
Figure 24: (a) Initial cyclic results of laminates and (b) graph segment.....	33
Figure 25: Initial and final graph segments of the sample laminates.....	34
Figure 26: Physical changes. (a) Before testing and (b) after testing result	34
Figure 27: (a) Initial cyclic results of laminates and (b) graph segment.....	35
Figure 28: (a) Final cyclic results of laminates and (b) graph segment.....	36
Figure 29: Physical changes. (a) Before testing and (b) after testing result.....	37

Tables

Table 1: Specific Modulus and Specific Strength of Typical Fibres, Composites, and Bulk Metals. Taken from: (Kaw, 2006).....	4
Table 2: E- 2 glass fibre properties.	23
Table 3: General properties of Durester CG (Finnester Oy, 2016).....	24
Table 4: slope change of the initial and final results.....	30
Table 5: slope change of the initial and final results.....	32
Table 6: slope change of the initial and final results.....	34
Table 7: slope change of the initial and final results.....	36

Chart

Chart 1: Test procedure	22
Chart 2: Slope change summerye	36

Notation

b	Width of a beam
∂c	Concentration gradient
C_s	Saturation content
D	Diffusion constant
F	Flexural strength of a beam
h	Depth of a beam
J	Flux
K_s	Soret effect factor
K_p	Pressure stress factor
T_z	Absolute zero on temperature scale
$t^{1/2}$	Half life
L	Span of a beam
M	Amount of substance
M_r	Relative mass up-taking
M_s/M_∞	Mass up-taking
P	Load
R	Universal gas constant
$S(t)$	Compliance at a time
S_t	Solubility content
S_0	Initial compliance
V_f	Fibre Volume fraction
τ	Relaxation time

FOREWORD

I wish to express my sincere gratitude to Mr. Rene Herrmann for their outstanding supervision throughout this thesis His inspiration and technical guidance has been vital. In addition, I am also grateful for the support of the technical staff Mr. Erland Nyroth in technical supervision. Lastly, I would like to thank my family and friends for their continued support over the years.

1. INTRODUCTION

1.1 Background

Composite materials have been used for different structural applications in recent years more widely. Their high strength to weight ratios, chemical and corrosive resistance makes them in high demand for different industries and applications. However, despite their wider application and demand, their long term characters and properties when exposed to different loads, chemicals, and environmental conditions is not clearly illustrated. Even though so many experiments and solutions for specific composite types were established, futuristic characters that can affect the performance could not be drawn as whole. Therefore, this has led to the over designing of products and put in question of some applications of the composite materials in regard of durability and safety.

Aging for composites can occur in three ways: Mechanically, physically and chemically. These aging mechanisms are highly dependent on the material property and the environment condition. Mechanical aging refers to when a composite is exposed to different loading conditions in its structure. On the other hand, chemical aging refers to the irreversible change caused on the Polymer Matrix Composites (PMC) chains by mechanisms such as thermos-oxidation, hydrolytic and thermal aging. Physically, aging can occurs when rapid cooling of the material below glass transition temperature that consequentially effects the characters of the polymer such as volumetric property (free volume), enthalpy, and entropy ; which also lead to change in mechanical properties as well.

Permeability is a process of molecular mass transport through a surface by diffusion process. The mass up-taking transport can be established in polymers through combination of different methods. These are: absorption of molecules in the surface, diffusion process caused by concentration gradient and desorption (opposite of absorption) from the surface. In polymers, the sum of solubility and diffusion processes plays a vital role for permeability; in both processes, changes of state occur if energetically suitable. Reaching to equilibrium state is determined by the combination thermodynamic energy and gradient of diffusent species concentration.

Determination of through-thickness properties of laminates aids to determine and evaluate the three-dimensional state of stresses effects and delamination processes. Inter-laminar shear strength is a measure of the shear strength among matrix layers. Usually, inter-laminar shear strength is measure using a short-beam shear test using a three point bending test setup.

In general, Conducting, evaluate and predict composite long-term reaction using accelerated ageing testing is quite difficult; especially for high speed aircraft structures. To establish and evaluate long term ageing of composite structures, it is required to develop fundamental understanding of the physical phenomena of the structures. This includes mathematical model, experimental materials and methodology development, and computational algorithm models.

1.2 Objective

There are objectives which the thesis seeks to achieve;

- To study ageing factors affecting Polymer Matrix Composite (PMC) especially, delamination caused hydrolytic degradation under cyclic loads.
- To conduct permeability test at a set temperature value of 90 °c in sample laminates treated in different anti-corrosive methods and prepared by different lamination techniques.
- To obtain results for better anti-corrosive methods and laminating techniques.
- To establish mathematical relation for comparison of different physical and mechanical changes occurring on sample laminates.

2. LITERATURE REVIEW

2.1 Composite materials

Composites are materials made up of two or more different constituents that have different mechanical and chemical properties. These constituents assist each other with their better mechanical performance and counterbalance each other insufficiency to have a superior property than discretely. For instance, the lack of resistance in flexure and compressive deformation of a fibre or fibre assembly can be compensated by embedding it with a suitable material matrix which can provide the lacking properties. With that, greater properties in strength can be achieved. On the other hand, composite can also be made for enhancing a material property with introducing or incorporating with fibre or filler materials. Properties such as electrical conductivity, fire resistance and temperature resistance can be enhanced with placing of fibre or filler materials in the composite matrix. These can be exemplified by the incorporation of fibres with brittle materials such as ceramics and thermosetting resins to increase their physical toughness and produced economically. (Martine Buggy, 1996)

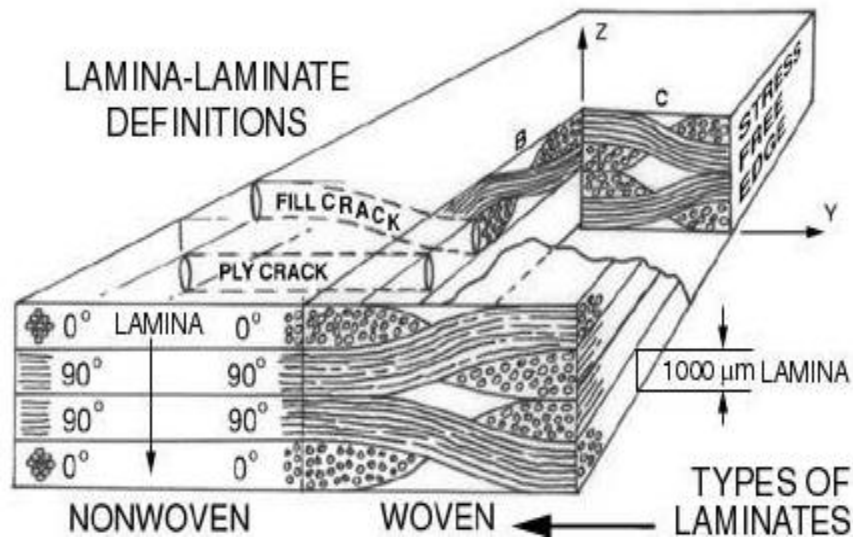


Figure 1: Lamina-Laminate Definitions

Historically, composite materials were used through human civilization along with the early tools such as bones, horn and wood. In Egyptian civilization mummy castings were made using plywood and linen-plaster composites. Moreover, human beings used to prepare a shrinkage resistance bricks made from mix of clay with straw for buildings and household items. (Martine Buggy, 1996) Composite were even used around 1500 BC to design bow for military purpose. The bow was made in Piling up of layer of materials that have different reaction under tension and compression. With that, it gives quite an advantage at the time by introducing a higher result. (Gascoigne, from 2001)

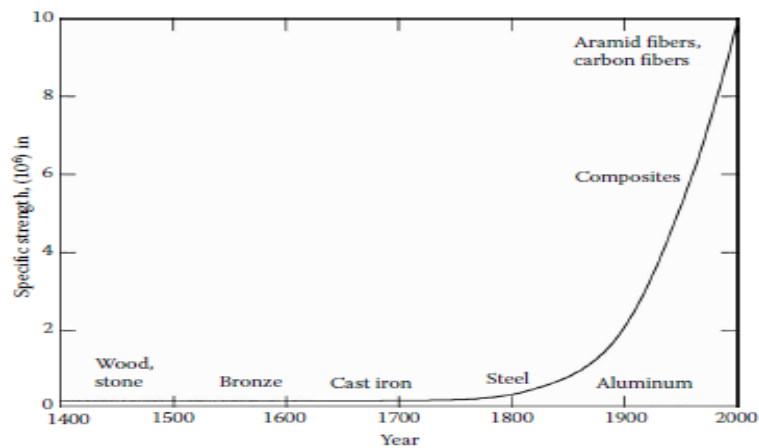


Figure 2: Specific strength as a function of time of use of materials. (Kaw, 2006)

Composites are mainly used because they can deliver properties that are difficult to achieve at once from conventional materials. For instance, designing a structural material for an air craft require in decreasing the over whole mass and achieving a better strength and fatiguing resistance. Therefore,

the use of composite came in handy when designing structure and component that requires multiple physical properties. Though the cost of composite are much higher than metal alloys, the mass reduction will enable to save fuel cost which leads to higher profit margin for the airlines. (Kaw, 2006)

The mechanical advantage of composites can be seen by comparing specific modules (E/ρ) and specific strength (σ_{ult}/ρ) with other materials. This conception was emphasized and exemplified on (Kaw, 2006) stating that: “the strength of a graphite/epoxy unidirectional composite could be the same as steel, but the specific strength is three times that of steel. What does this mean to a designer? Take the simple case of a rod designed to take a fixed axial load. The rod’s cross section of graphite/epoxy would be same as that of the steel, but the mass of graphite/epoxy rod would be one third of the steel rod. This reduction in mass translates to reduced material and energy costs.

Table 1: Specific Modulus and Specific Strength of Typical Fibres, Composites, and Bulk Metals. Taken from: (Kaw, 2006)

Material Units	Specific Gravity	Young's modulus (Gpa)	Ultimate Strength (Mpa)	Specific modules (Gpa-m3/Kg)	Specific Strength (Mpa-m3/Kg)
Graphite fibre	1.8	230.00	2067	0.1278	1.148
Aramid fibre	1.4	124.00	1379	0.08857	0.9850
Glass fibre	2.5	85.00	1550	0.0340	0.6200
Unidirectional graphite/epoxy	1.6	181.00	1500	0.1131	0.9377
Unidirectional glass/epoxy	1.8	38.60	1062	0.02144	0.5900
Cross-ply graphite/epoxy	1.6	95.98	370	0.06000	0.2331
Cross-ply glass/epoxy	1.8	23.58	88.25	0.01310	0.0490
Quasi-isotropic graphite /epoxy	1.6	69.64	276.48	0.04353	0.1728
Quasi-isotropic glass /epoxy	1.8	18.96	73.08	0.01053	0.0406
Steel	7.8	206.84	648.1	0.02653	0.08309
Aluminium	2.6	68.95	275.8	0.02652	0.1061

Currently, the range of combination variations is higher because of the growth plastic industries and availability of fibre materials in wider range. With that, production processes these days considers multi-functional factor such as, performance required from a composite material, economical manufacturing routes and methods of manufacturing. (Martine Buggy, 1996)

2.2 Classification of Composites

Classification of composite can be based on the geometry of the reinforcement or by the type of the matrix element. Geometrically, composites reinforcement can be particulate, flakes or fibre type whereas, the matrixes can be constructed as such from ceramic, metal and carbon/graphite.

Fibres can be found in both natural and advanced types. Naturally, fibres can be extracted from animals in a form of wool, silk, hair and others; on the other hand, from plants, in forms of cotton, jute, stem, leaf, etc. Fibre can also be extracted from minerals in forms such as glass wool, mineral wool, basalt and asbestos. Advanced fibres are fibres which has a high specific stiffness and strength. Advanced fibres are made from materials such as of carbon fibre, glass fibre, Alumina, Aramid, Silicon Carbide and Sapphire. Most of these material elements are found in the top of the periodic table where their masses are quite lighter than the conventional metals such as iron, chromium, nickel. (Mohite, 2012)

The matrix material of a composite can be made from polymers, metals, ceramics and carbon or graphite.

Thermoplastic -Matrixes have features low cost processing, high toughness, working temperature up to 225⁰c. on the other hand, thermosets- matrixes include polyesters, epoxies and polyimides. General character such as requiring heating for decomposition, easy failure with low strains, difficulty for re-processing and repair and limited shelf life can be considered to be some of the few characters that are considered to be drawbacks. (Mohite, 2012)

Metal-Matrix Materials have preferable feature because their working temperature range up to 300°C and in some matrixes such as titanium matrixes it can reach up to 800°C. These matrix materials show advantageous features such as higher transfer strength and toughness, unavailable moisture on the material and high thermal conductivity. However, heavier weight, corrosion and vulnerable degradation can be taken as their disadvantage. (Mohite, 2012)

Ceramic-Matrix Composites are matrixes such as silicon carbide fibres fixed in a matrix made from a borosilicate glass. Ceramic-matrix materials are suitable specifically for use in lightweight, high-temperature components, such as parts for airplane jet engines. (Mohite, 2012) Advantages of ceramic matrix composites include high strength, hardness, and high service temperature range for ceramics, chemical resistance, and low density. Nevertheless, ceramics by nature have low fracture

toughness .with that, under tensile or impact loading, they are brittle. By reinforcing ceramics with fibres, such as silicon carbide or carbon, it is possible to increases their fracture toughness and other mechanical properties. In addition, since the mechanical failure happens gradually the service period will increase as well. (Kaw, 2006)

Carbon and Graphite fibre matrixes are mainly used in high-modulus and high-strength applications such as aircraft components. There advantages consist of high specific strength and modulus, low coefficient of thermal expansion, and high fatigue strength. Composite materials those having carbon fibre as the reinforcement plus as matrix material are known to be carbon –carbon composite. These materials have a working temperature range up to 3000°C.however, their low impact resistance, high electrical conductivity and high cost for the fabrication complex processing can be considered as a downside of their properties. (Kaw, 2006)

2.3 The Aging of Polymeric Matrix Composites

Composite aging is a broader term that involves a change of physical properties through a period of time. Depending on the material properties and ageing environment, the mechanism of ageing of composite can be categorized in to three basic ways: Physical, Mechanical and Chemical. These mechanisms can be either additives or subtractive depending on factors such as mechanical loading, the environment and material types. (Rod Martin , 2008)

In the detail study of the concept of ageing, additional important terms needs consideration. These can be: environmental degradation factors, critical degradation mechanisms and accelerated aging. Environmental degradation factors are the environmental factors that are found in the surrounding in general such as heat, moisture, and chemicals, mechanical loading, etc. on the other hand, critical environmental degradation factors are those which specifically degrade and alter physical changes within the confined environment. (Rod Martin , 2008)

Accelerated ageing is the process of speeding up a specific degradation method to achieve the real-time ageing with a shorter period of time; this helps to save cost and time on the study of needed on the composites. In addition, accelerate ageing can help the in determining the service-time of materials and improve the material selection for specific process. These method is vital for selection a way of composing material that are durable in an extreme environmental condition such as change of temperature, moisture, oxygen and mechanical loadings. (Rod Martin , 2008)

Moreover, numerous studies shows that accelerate ageing tests can be difficult in obtaining accurate results; especially for high speed aircraft structures. To inaugurate and evaluate ageing of composite structures, it is vital to develop fundamental understanding of the physical phenomena of the structures with mathematical and analytical models, and experimental methodology development.

These was emphasized in (National materials Advisory Board; Commission on Engineering and Technical; National Reserch Council , 1996) ; where An engineering committee studied the long-term ageing of materials and structures using accelerated testing methods for high speed aircraft structures; In their summery they stated: *“In most cases, service failures due to inaccurate characterization of aging responses result in potentially costly repair or premature component replacement. However, in safety-critical applications such as nuclear reactors and commercial aircraft the cost of inaccurate characterizations could be significantly greater. Simulated service testing to characterize materials aging is fraught with deficiencies that stem from the inability to reproduce complex service conditions in the laboratory. Important aspects of the environmental conditions encountered in service cannot be accurately simulated. These environments are influenced by changing loads, temperature, humidity, radiation, and other effects that are interactive and cannot be consistently recreated and controlled. Moreover, critical degradation mechanisms and failure modes are not well known and understood prior to testing. While most service simulation testing has provided insights concerning degradation mechanisms and potential failure modes, it has yielded little engineering quality data useful in the systematic characterization of materials aging. Thus, alternative approaches are required to develop such data.”*

Adding to that, the committee lay out fundamental approach to portray the ageing regardless of materials property and application; with that, five basic steps were recommended. These are:

- Outlining the service atmosphere,
- Recognizing possible degradation or failure mechanisms,
- Illustrating the materials aging reactions using accelerated ageing methods and mathematical models,
- After obtaining results, examine and understand aging in structural components.
- Confirming expectations.

2.4 Mechanics of Composites

The design and analysis for of the composite material is different form the conventional materials because of the different properties of the constituents. Approach for the analysis of mechanical behaviours can be determined by either micromechanics or macro mechanics of the lamina.

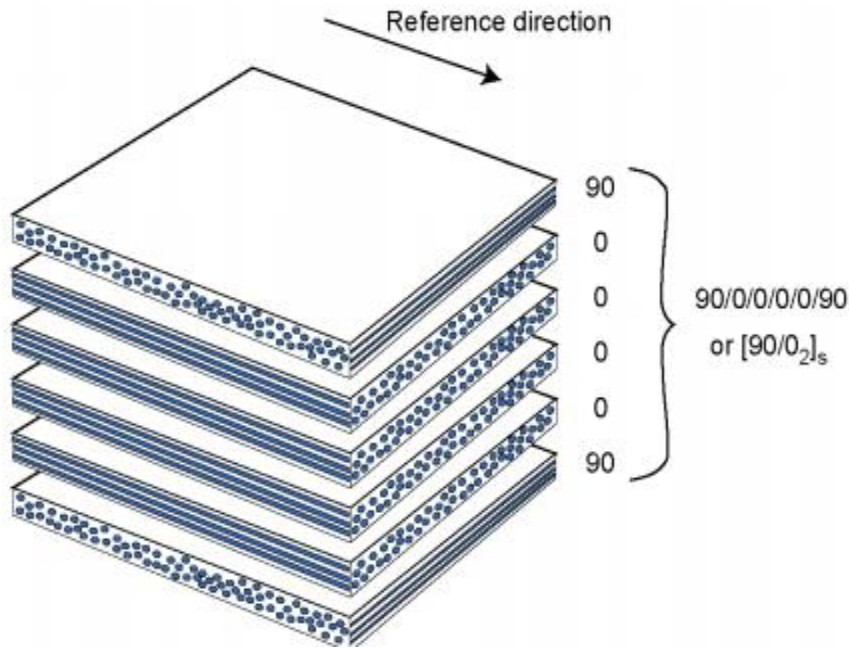


Figure 3 fibre laminate (stack of plies), illustrating the nomenclature system

Micromechanics of lamina studies properties of individual constituents and considering the ply as a homogenous lamina. Theoretically, properties of a lamina can be predicted from the known properties of the fibre and matrix. By considering parameters of the lamina constituents such as stiffness, strength, coefficient of thermal and moisture expansions, relation can be derived which can help to select constituents for a specific design of composite lamina.

Micromechanics Analysis uses values of volume and mass fractions, density and void contents to find theoretical formulas for stiffness, strength and hydrothermal properties as function of fibre volume fraction. In addition, experimental determination of fibre volumes and void fractions inquire the density values of the composite. Void contents in the composite can have a direct effect in the mechanical properties such as, shear stiffness and strength, compressive strength, transversal tensile strength, etc. The increases of void content with in 1 % can lead to 2-10 % of decrease in the preceding matrix dominated properties as a whole. (Kaw, 2006)

Macro-Mechanical Analysis concerns with the study of laminate which consists of more than one lamina. The mechanical strength and stiffness can depend each lamina's elastic moduli, Stacking position, thickness, angle of orientation, coefficients of thermal and moisture expansion. The stiffness and the strength of a lamina can differs from point to point since it is compiled of the fibre, the matrix material (both being homogenous and isotropic) and the cross-linking (interface) area ;these makes it difficult to mechanically model and for these reason macro-mechanical analysis are based on average mechanical properties of the fibre and matrix values. (Kaw, 2006)

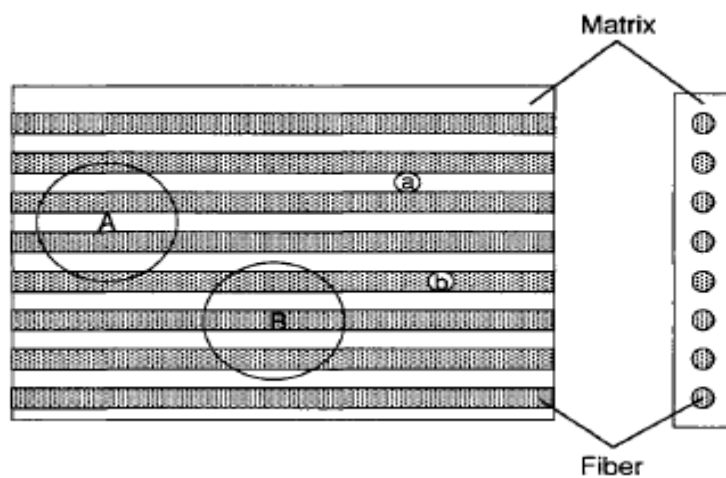


Figure 4: Macroscopic (A, B) and microscopic (a, b) scales of observation in a unidirectional composite layer. (Daniel I. M., 2006)

2.5 Failure of Mechanisms in Fibre-Reinforced Composites

Failure of composite materials can be a function of different constituent factors such as geometric lamina properties, environmental condition, lamination geometry, and state stress. These failures can be seen for microscopic level and macroscopic level. In the microscopic level, where each lamina are considered as homogenous layers and anisotropic, failure can occur and may propagate to other layers causing interplay failure.

2.5.1 Failure Modes in Unidirectional Composite

In unidirectional composites the fibre strain plays a major role in the strength of the over whole lamina. Therefore, failure most expected to propagate by the failure of fibre strands. These failure can be initiated mostly when fibre brakes which increases interfacial shear stress in the area of the brakeage, then passes to the surrounding matrix. When the longitudinal tension rises, the failure area will remain relatively the same. However, the number of broken fibre increase throughout the matrix plane and causing progressive weakness.

The three basic failures modes in the lamina scale can be identified as: brittle; brittle with filament pull-out; and staggered failures. These Failure modes are more likely to be seen in different fibre volume-ratio of composites lamina. Hence, for fibre volume ratio less than 0.4 shows most likely brittle failure, then for values between 0.4 and 0.65, shows brittle with filament pull-out and staggered failure above 0.65. (Daniel I. , 1978)

Compressive load of unidirectional composites exhibits different modes of failure than the tensile failure modes. These are: filament micro-buckling with matrix elastic and fully bounded, matrix yielding, panel micro buckling, shear failure, ply separation by transverse tension. (Daniel I. , 1978)

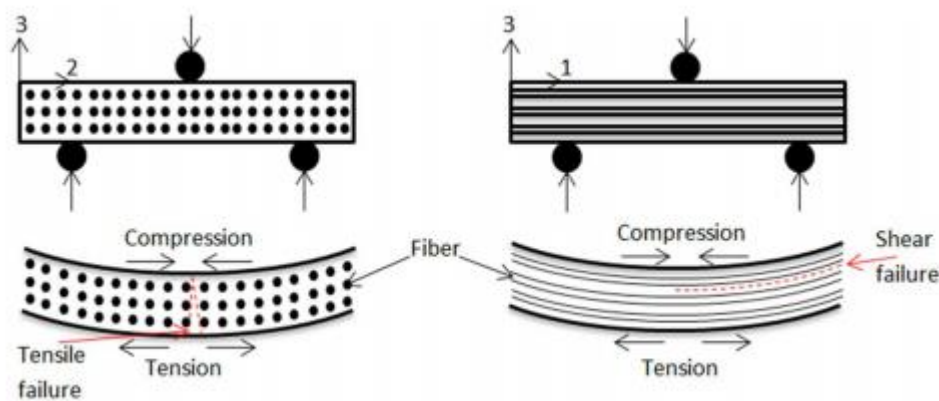


Figure 5: Distribution of interlineal normal stress and interlineal shear stress in [0/90], laminate under axial tension.

Other loads such as in-plane shear, transverse compression and biaxial loadings can have failure modes on the lamina. Failures such as, matrix shearing, compressive failure and de-bonding or combination of all failures can occur. On the other hand, composites having discontinuities such as

holes and cracks, exhibits numerous failure modes which have been theoretically unpredictable. (Daniel I. , 1978)

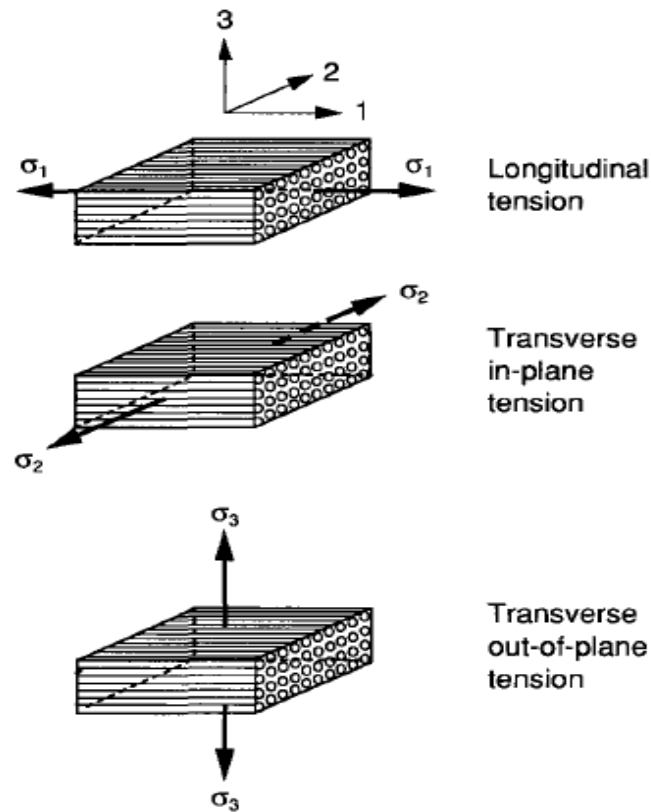


Figure 6: Different types of loading in a unidirectional laminate (Daniel I. M., 2006)

2.6 Through-Thickness Compressive Properties of Laminate

The thick composite section of a laminate behaviour can be establish by elaborating a through-thickness studies. Through thickness compressive properties can be studied using a rectangular or square specimen on a three-point bending set up (a short-beam shear test). The test helps to determine the inter-laminar shear strength which is a measure of the shear strength between matrix layer layers. However, the method does not give the exact values of the properties. The short-beam shear (SBS) testing method is used mostly for quality control of lamination processes. The inter-laminar shear strength can be calculated from classic beam theory. Which is given by: (Daniel I. M., 2006)

$$F_{31} = \frac{3P}{4bh} \dots\dots\dots [\text{Equ.1}]$$

Where P = load at failure initiation b = width of beam and h = depth of beam (laminate thickness)

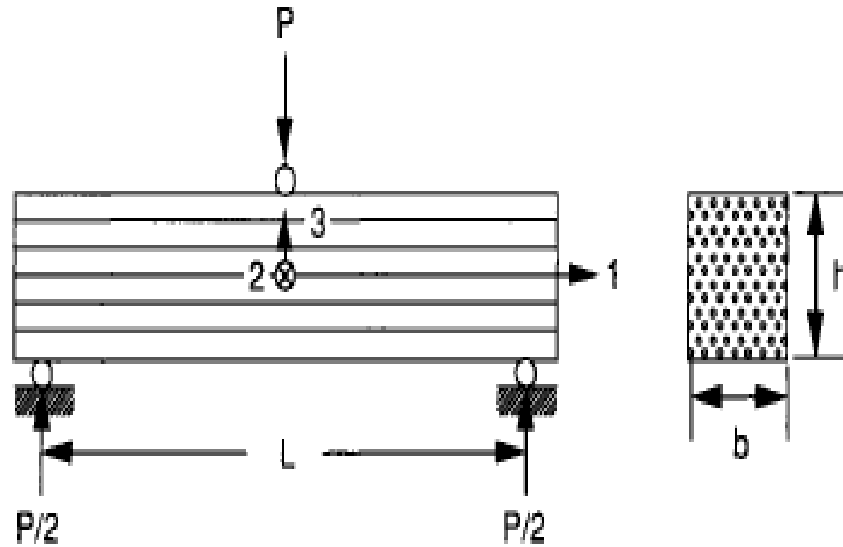


Figure 7: short-beam shear test set up (Daniel I. M., 2006)

During the test, to avoid the failure fracture on the outer plies of the beam due to compression, the depth to span ratio must satisfy the following relationship:

$$\frac{2L}{h} < \frac{F_1}{F_{31}} \dots\dots\dots [\text{Equ.2}]$$

Where L = beam span F₁ = flexural strength of beam in the fibre direction

With that, the inter-laminar shear strength of the beam can be ensure prior to flexural failure.

2.7 Viscoelasticity

Viscoelasticity is a character of material to exhibit both viscous and elastic properties when undergoing deformation. These materials are time dependent when it comes to stress-strain relationship. Some of the characters of the viscoelastic materials are: (I) in constant stress, the strain will increases with time; then again in constant strain, the stress will decrease with increase of time (ii) rate of load affects the effective stiffness (iii) when applying cyclic load, it leads to losing of mechanical energy. (Lakes, 2009)

Stress relaxation and creep are the main features of viscoelasticity. Creep a character shown by a material under a constant stress for longer period of time. In the matrix material, when constant stress is applied, the strain will increases because of the long chain molecules starts to distance from one another. On the other hand, stress relaxation is the overtime effect of the matrix material undergoing in a constant strain, losses the stress with a period of time. (Lakes, 2009)

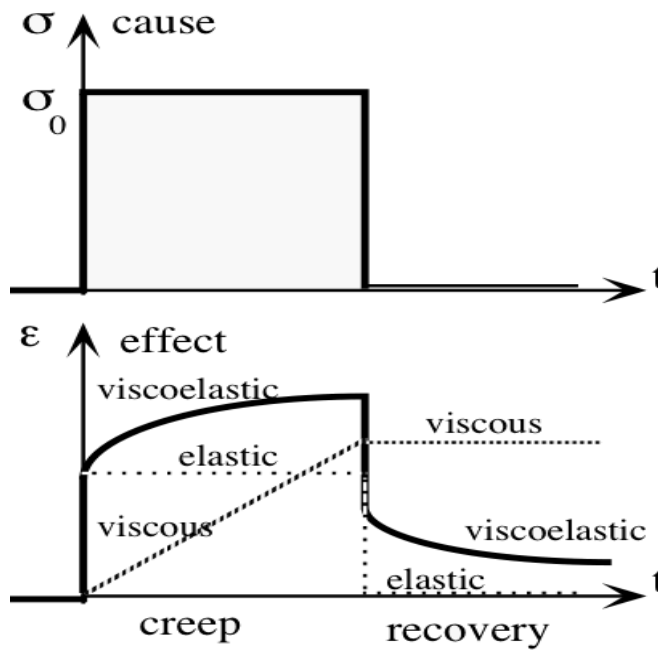


Figure 8: Creep and recovery Stress and strain Vs time. Taken from: (Lakes, 2009)

In the composite matrix, the fibre filaments have much higher modulus than the matrix material. With that, the combined effect of the composite on the mechanical shear will be absorbed by the fibre while the viscoelastic effect will be taken by the matrix material. (Rod Martin , 2008)

2.8 Ageing and Effective Time

Experimental finding indicate for the Polymer Matrix Composites (PMC) having a matrix dominated properties, shows the same effect on physical ageing similar to polymers. With that, the time-dependent characteristics can be developed experimentally using isothermal creep compliance tests and using linear viscoelastic with superposition methods. These experimental approach was firstly quantified and publish by Leendert C.E. Struik in 1978 titled “*Physical ageing in polymer and other amorphous materials*”. Struik’s experiment helps to define the concept using creep tests to determine ageing factors and effective time. (Rod Martin , 2008)

One vital procedure that is needed to be established in studying material ageing in more accurate way is, the time-ageing time superposition (TASP). The theory behind the TASP is that when a momentary creep compliance are measured while conducting a creep test of a quenched material below the glass transition temperature T_g (Which also referred as the ageing time T_e); and then, evaluating the

inclination of creeps compliances. With that, after a short series creep tests test by Struik, results shows that, the inclination of the compliance curves in different values. The momentary creep compliance was formulated by L. Catherine Brinson and Tom S. gates in 1995 and describes as follow:

$$S(t) = S_0 e^{(t/\tau)^\beta} \dots\dots\dots [\text{Eq. 3}]$$

Where $S(t)$ is compliance at time t , S_0 is the initial compliance, τ is the relaxation time and β is the shape parameter (Weibull slop)

2.9 Diffusion

Diffusion is the net movement of molecules from higher concentration to a lower concentration in a system which then leads to concentration equilibrium with in time. In diffusion process the diffusing species is called the diffusant and its concentration gradient highly influences the rate of diffusion. In addition to concentration gradient of diffusant, other factors such as size of the molecules and physical state of the diffusants, the morphology of the substrate, the surface or interfacial energy, the compatibility limit of the diffusant within substrate and the volatility of the of the diffusant can have significant influence in the diffusion process.

Adolf Fick's in the 1855 introduces the Fick's law of diffusion which expresses the mathematical concept of the rate of mass diffusion of a substance in a unit area. Diffusion processes that follow the Fick's laws are known to be fickian diffusion. In his first law, which express the steady state of diffusion, it stated that rate of diffusion of a substance in a section of a unit area is directly proportional to the negative concentration gradient measured normal to the section and can be mathematically expressed as:

$$J = -D \frac{\partial c}{\partial x} \dots\dots\dots [\text{Eq. 4}]$$

Where J is the Flux or rate of mass per unit area, C the concentration of the diffusant, X is the space –coordinated normal to the section area and D is the diffusivity constant.

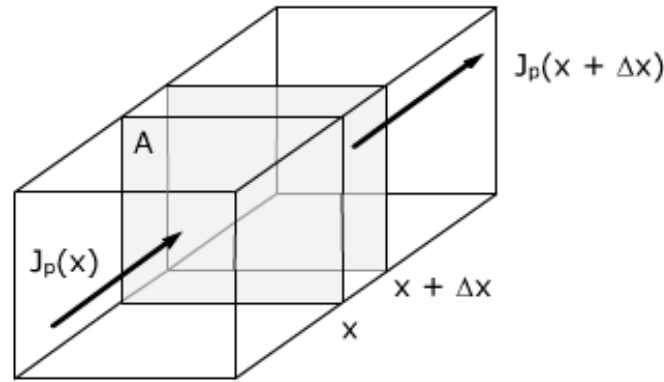


Figure 9: an expression of diffusion through a membrane

The second law of Fick's expresses time dependence of concentration and can be expressed as:

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \dots \dots \dots [\text{Eq. 5}]$$

Though Fick's rate of diffusion expresses in terms of the concentration gradient, other factors such as temperature and stress gradients can be a driving agent and can be included in diffusion model as well. This was exemplified in (Duncan, Urquhart, & Roberts, 2005) where a diffusion model created in ABAQUS finite element modelling software. In the model, other gradient were included such as pressure and thermal gradients giving the following mathematical expression:

$$J = -sD \left[\frac{\partial c}{\partial x} + K_s \frac{\partial (\ln (T-T_z))}{\partial x} + K_p \frac{\partial P}{\partial x} \right] \dots \dots \dots [\text{Eq. 6}]$$

Where J is the flux is the diffusivity, s is the solubility, K_s is the soret effect factor (diffusion factor due to temperature gradient), K_p is the pressure stress factor, T_z is the absolute zero on the temperature scale (Duncan, Urquhart, & Roberts, 2005)

2.9.1 Mathematics of Diffusion

The theory behind the mathematics of diffusion is in isotropic substances which implies that the concentration gradient is proportional to the rate of transfer of the diffusent; and this is formulated in Equation 4. Then, the differential equation of diffusion equation gives the concentration gradient in time dependence (second law of diffusion); which is expressed in Equation 5.

The solution of diffusion equation can be obtained using various initial and boundary conditions. The solutions can have either consists of series error functions or series of integrated functions; either way, the solutions are most suitable in the early stage of the diffusion. Functions of diffusion is used according to the geometry of the diffusion medium. For instance, if diffusion occurs in cylindrical shaped medium, the trigonometrical function will be series of Bessel functions (cylinder equation). (J.Crank, 1975)

Solution of one dimension (x direction) second-order differential equation of Fick's second's law where D is constant is given by:

$$C = \frac{A}{t^{1/2}} e^{(-x^2/4Dt)} \dots\dots\dots [\text{Eq.7}]$$

where A is random constant , $t^{1/2}$ is half-life of the reaction

For time $t > 0$, the above expression tends to be zero when x approaches infinity positively or negatively and at time $t = 0$, diffusents spread everywhere; However, at $x=0$ it becomes infinite.

For cylindrical shaped medium, and with an amount of substance M, the above expression can be simplified as follow:

$$C = \frac{M}{2(\pi Dt)} e^{-(x^2/4Dt)} \dots\dots\dots [\text{Eq.8}]$$

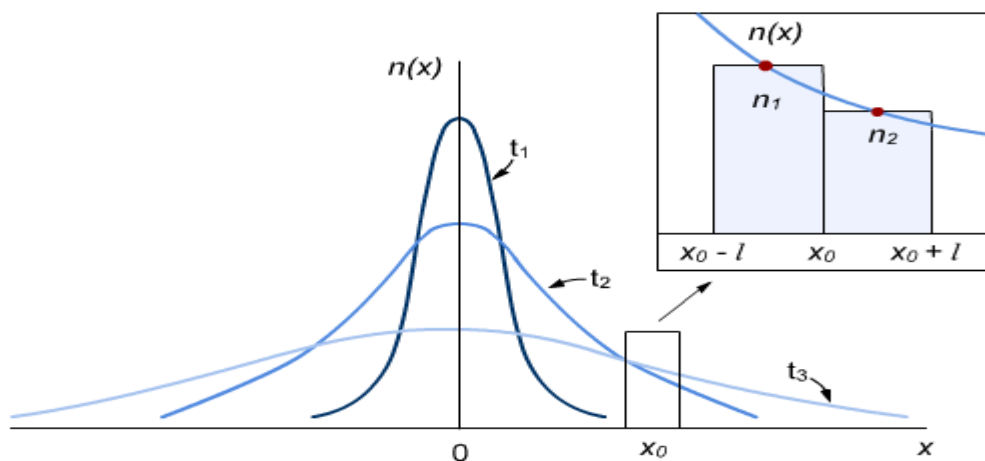


Figure 10: example of concentration distance curve of a typical distribution at three successive time.

2.9.2 Hydrolytic Degradation

In polymeric composites hydrolytic degradation is caused by water diffusion. Diffusion of water will lead to moisture uptake and plasticization (a change of thermal and mechanical properties of a polymer) of the polymer matrix. In polymeric matrix composites (PMC) factors such as Matrix geometry, moisture concentration, temperature and time can contribute to the diffusion process. Because of diffusion of water through the matrix, unbalanced water concentration will occur on matrix material leading to stress on the nonhomogeneous material. For composite laminas, matrix cracks can be initiated due to the non-uniform stress distribution. These cracks lead to absorption of moisture when undergoing in repetitive hydrothermal cycling and allowing new passageway for more moisture up-taking and debonding; which finally leads to delamination. (Rod Martin , 2008)

On composite material, weight gain and physical & mechanical changes can be hydrolytic degradation indicators. Weight gain can be a result of exposure to a wetted environment within a period of time. In this case, the required time for saturation depends upon the temperature gradient; additionally, physical change can be observed sequentially after exposure. (Rod Martin , 2008).

2.9.3 Mass Up-taking Measurement Method

The mass up-taking measurement methods or gravimetric can help to measure the absorption of liquids into a polymer or polymeric composite matrix. Standards such as ASTM D 570 and ISO 62 outline measurement methods for mass up-taking. The method uses a standard sized sample with initial mass (M_0) to be immersed in a liquid for a fixed period of time, at a constant temperature, it will be weighed after being dried. After that, the sample will be immersed again and the increase in mass divided by the initial mass measured regular time interval; these help to plot the absorption curve. The saturation mass up-taking (M_s) is the weight gain of three consecutive measurements differ by not more than 1% to the overall weight gain; and saturation moisture content (C_s) can be simply describe as M_s/M_0 . In other hand, effects such as the water-soluble matters by comparing the M_0 with M_c it can be easily determined the loss of water-soluble matters from the samples. If there is no water-soluble loss, $M_c=M_0$. (Duncan, Urquhart, & Roberts, 2005)

The relative mass up-taking can be calculated as follow:

$$Mr(t) = \frac{M(t)-Mc}{Mo} \dots\dots\dots [\text{Eq.9}]$$

2.10 Factors Affecting Diffusion in Polymer Matrix

2.10.1 Polymer Chemistry

Among the factors that can influence the rate of diffusion in a polymeric matrix is the chemical composition of the matrix. Hydroscopic materials such as tend to have high attraction for polar molecules. With that, the diffusivity coefficient is higher for polymeric matrixes with polar molecules, which means absorption will increase as well. In addition, swelling partial dissolution of polymer matrix can be observed in the physical structure of the polymer matrix. On the other hand, a non-polar polymer such as polypropylene, the tendency to absorb or uptake polar molecules is significantly lower. Therefore the diffusion rate will be lower as well. (Duncan, Urquhart, & Roberts, 2005)

2.10.2 Porosity and Voids

Pores and voids are defects that are formed when polymers are processed or undergo physical stresses. These can be caused by Trapped air bubbles, tensile loads or chemical swellings. They are different from free volumes (which are formed by molecular chain motion) by having a permanent feature and size as well. High level of porosity in the polymer structure can lead to an increase of diffusion rate and permeability. (Duncan, Urquhart, & Roberts, 2005)

2.10.3 Filler Particles

Filler materials are added to the polymer matrix to provide reinforcement that increase mechanical efficiency. For most inorganic filler materials having lower solubility than the matrix material, diffusion rate will be much lesser. On the other hand, most organic filler material having higher solubility than the matrix material can have higher absorption. Filler materials affect the path length of diffusants and reducing mass transport rate. More importantly, water diffusion in polymer matrixes

depends on the volume fraction of the fibre and fibre orientation of the layers. Permeability of water can be prevented by complex mixture property, where multi directional fibre orientation occurs. With that, diffusion orientated fibre composite with a mixture approach can be established which co-relates the fibre volume fraction and diffusion coefficients of water. These can be expressed mathematically for both transversal and longitudinal directions of fibres as follow:

$$D_{long} = (1 - V_f) D_m \dots \dots \dots [\text{Eq. 10}]$$

$$D_{tran} = (1 - 2 \frac{\sqrt{V_f}}{\pi}) D_m \dots \dots \dots [\text{Eq. 11}]$$

Where V_f is fibre volume fraction and D_m diffusion coefficient for polymer matrix

Apart from concentration gradient playing a vital role in diffusion process, higher affinity of surfaces and molecules of the diffusent can be additional driver in polymer matrixes. However, when a fillers / particles surface have high affinity with diffuent molecules, it is more likely to reduce the diffusion rate since diffusant molecules need to overcome the energy to desorb (Opposite of absorption) filler particles. (Duncan, Urquhart, & Roberts, 2005) 2.9.5 Temperature

Diffusion rate, permeability and molecules in polymer matrixes will be affected with change in temperature. Increase in temperature will directly affects the free volumes in the polymer matrix then again affect the solubility of the matrix which leads to higher diffusion rate and permeability. Svanete Arrhenius in 1989 formulate and model the temperature dependence of reaction rates. His formula can be used to relate and model temperature, diffusion coefficient, creep rate and other related thermal influenced processes.

$$D(T) = D_0 e^{-[E / (S(T) = S_0 e^{-[H / R x T]}) R x T]} \dots \dots \dots [\text{Eq. 12}]$$

$$S(T) = S_0 e^{-[H / R x T]} \dots \dots \dots [\text{Eq. 13}]$$

Where $D(T) / S(T)$: diffusion coefficient / solubility as function of temperature, D_0 / S_0 : the diffusion coefficient / solubility when the temperature goes to infinitely, E / H : the activation energy for diffusion / the mixture enthalpy, R : Universal gas constant and T : Temperature.

2.10.4 Mechanical Stress and Strains

In addition to the internal and residual stress, which arises from when initial processes of the polymer, external stress applied on polymers can have significant influence on the rate of diffusion. When a tensile load is applied on a sample, free volumes will increase along with creation of pores and voids which gives a path for diffusers or creating a new site for diffusion. On the other hand, when a polymer sample exhibits a compression stress, internal voids and free volumes will be close which inhibits diffusion into the polymer matrix. In studies conducted on polymer films which are coated with plasma deposits barrier and with uncoated once in different level of strain, the results show that coated samples show 20 % of permeability than the uncoated once. The result on the coated films permeability was caused because of the microscopic cracks which are formed when coating material is applied. On the other hand, results show permeability increase when applying strain on both cases nevertheless, higher effect was observed in coated polymer sample. (Duncan, Urquhart, & Roberts, 2005)

2.10.5 Macro-Scale Interfaces

The permeation properties of most used polymers is difficult to analyse because the nano/ micro-scale interfaces with multi- phase systems they exhibit. Therefore, non-linear functions will be used for the analysis of their properties. However, since their properties in the intended mixture is directly proportional to volume fraction of their discrete phases, it can be estimated according to their volumetric ratio. On the other hand, Macro-scale interface systems such as adhesive joints, laminates, coating, multi-layer films and long-fibre composites, will be considered as non-homogeneous material and analysed distinctively. (Duncan, Urquhart, & Roberts, 2005)

When analysing the multi-layer laminas or films two cases of direction can be considered for analysing the diffusivity and solubility: diffusion parallel to the layers and transverse to the layers. In the first case where diffusion occurs in uni-direction, the analysis will be straight forward; concentration on layer interfaces will be considered as a boundary condition to the next consecutive layer. On the other hand, for transversal direction, the diffusion occurs multi directional way and difficult for analysis and fallible. (Duncan, Urquhart, & Roberts, 2005)

In macroscopic interface among dissimilar materials such as metal-adhesive and metal-coating where strong affinity between surfaces and polar solvents occurs, diffusion rate can be altered and increasing permeation rates and this works for both directional cases. (Duncan, Urquhart, & Roberts, 2005)

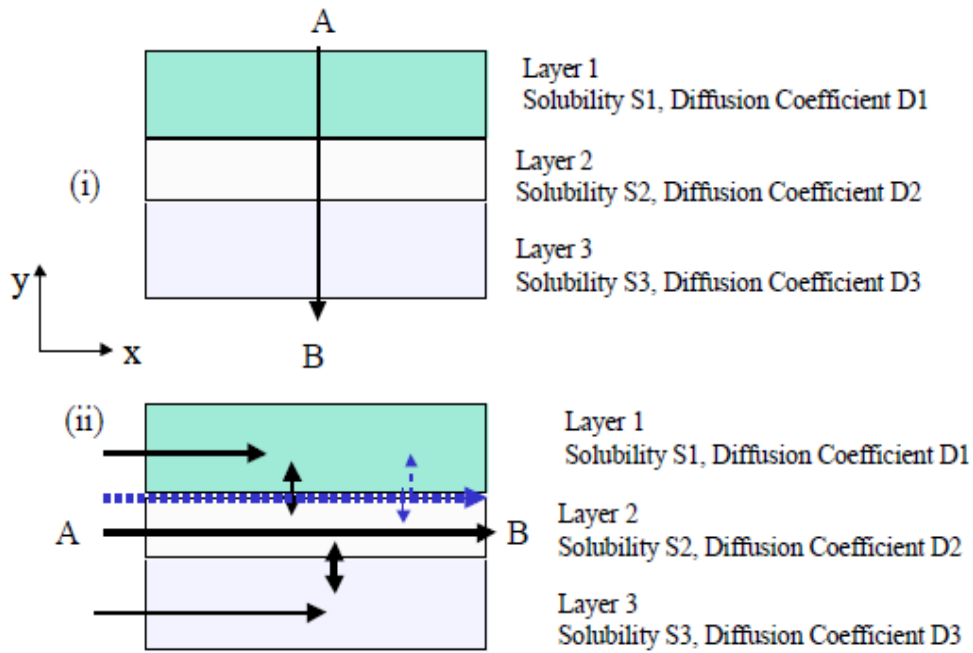


Figure 11: Diffusion in multi-layer systems (i) in parallel (ii) Transversal direction. Taken from: (Duncan, Urquhart, & Roberts, 2005)

3. MATERIALS AND METHOD

For the experiment conducted on hydraulic degradation (ageing) under cyclic load, different samples groups with different permeability reducing methods were prepared. The protective methods were: embedding samples with Aluminium foils between layers and spray painting of Durester CG (a polystyrene based coating spray) on the surface area. In addition, for reference purpose, sample laminates without any treatment were prepared.

The samples were prepared in two methods of lamination: with Vacuum fusion and compression moulding. After that, by cutting all sample in uniform rectangular dimension, initial mass measurement were taken and recorded. Then, cyclic three-point bending test values were set on the Testometric testing machine and tests were performed. The samples inside a water container were placed in the oven, which was set to 90°C. These processes were repeated for several times and water up-taking were recorded.

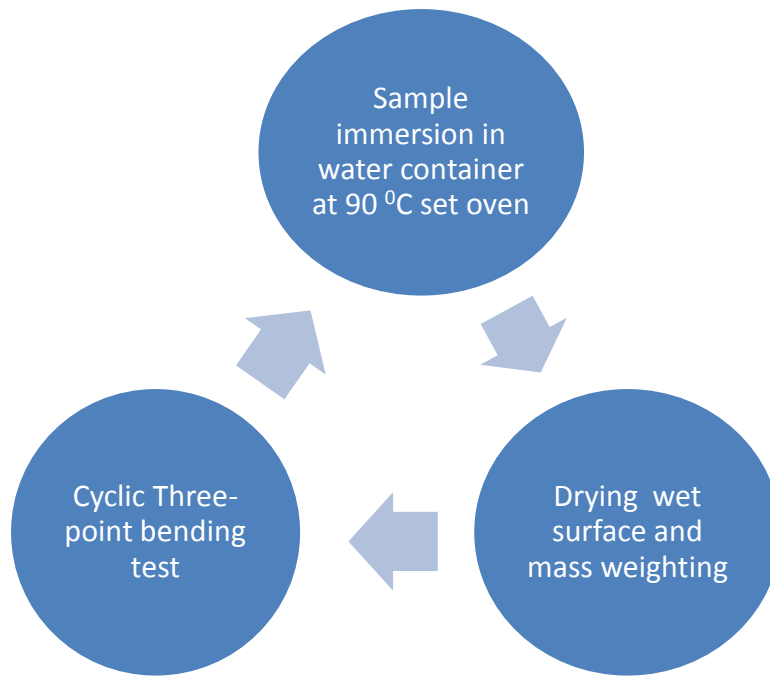


Chart 1: test procedure (cycle)

The table below shows that the initial mass of laminate before conducting the water diffusion experiment.

Table 1: initial mass compassion of laminates

No	Type of samples laminates	Average initial-masses (gm)	Average Dimension L*W*T (mm)
1	Glass fibre-aluminium embedded (with comp. Moulding)	29,89	300*30*3.5
2	Glass fibre-aluminium embedded (with vacuum bagging)	34,71	300*30*3.5
3	Durester CG treated glass fibre laminate	27,35	300*30*3.0
4	Glass fibre laminate (without any treatment)	26,25	300*30*3.0

3.1 Initial Material Preparation

The sample laminate have two constituents: uni-directional glass fibres textile and Vinyl esters resin. The textile was cut using seizers and resin was placed using paint brush between each textiles and Aluminium foil accordingly. After that, by setting up the vacuum fusion bag layup and other necessary component in one side and setting up the compression moulding machine on the other,

lamination process was conducted.

3.1.1 Vinyl Ester

Vinyl ester is the product of a chemical reaction (esterification) between unsaturated organic acid and epoxide-terminated molecule. After the synthesis, it will be dissolved in styrene or other reactive solvent. Structural similarity with polystyrene can be observed in molecular-level, however, their reactive site location differs. Since the reactive site are located in the end of the molecular chain, shock absorption capacity is higher; making it much tougher in strength than polystyrene. In addition, apart than other ester groups, which are vulnerable for hydrolytic degradation, vinyl ester shows a better resistance. Moreover, it costs relatively less than other epoxy resins and have better chemical resistance. (Net Composites , 2016)

3.1.2 E-2 Glass Fibre

E glass fibre was designed to resist electrical current .however, it was later applied for reinforcement purpose because of its excellent giber forming properties. It is a low alkali glass with a with a composition of SiO₂ 54wt%, Al₂O₃ 14wt%, CaO+MgO 22wt%, B₂O₃ 10wt% and Na₂O+K₂O less then 2wt%.in addition, other materials also be present as impurity levels.. It mainly known for its high strength, and modulus. Other properties, such as thermal conductivity, resistance to corrosive environment, low cost and non-flammability makes it more suitable for various application. (agy, 2006) Some of the mechanical properties of E glass fibre can be seen in follow the following table:

Table 2: E- 2 glass fibre properties.

Material	Tensile Strength (MPa)	Density (ρ) (g/cm ³)	Young Modulus (Gpa)
E-2-glass	2000	2.55	80

3.2 Protective Materials

3.2.1 Durester CG

Durester CG is a composite coating and surface treatment product manufactured by Finnester Coating Oy. It is unsaturated polyester based chemical and have a thixotropic property (time-dependent shear thinning property). Its sanding properties which makes easy to surface-finish by polishing or water-sanding methods. Durester coating is suitable for composite moulds and master plug manufactures. Moreover, its chemical and heat resistance properties adds superiority for its application in surface treatments. (Finnester Oy, 2016) Its general properties can be seen as follow:

Table 3: General properties of Durester CG (Finnester Oy, 2016)

	General Properties (23°C)	Value
1	Viscosity [DIN cup 4] (s)	35
2	Barcol-hardness	36/45
3	Gel time (Min)	60
4	Tack free (Min)	180
5	Sandable (Hr)	8

3.2.2 Aluminium Foil Reinforcement

Aluminium foil is used as constituent in a metal/composite hybrid laminates in a composite known as Glass laminate aluminium reinforcement epoxy (GLARE). It is one type of Fibre metal laminates (FML) process. FML is used for both aerospace and non-aerospace applications. GLARE is prepared by using by stacking layer of aluminium foils with glass fibre and bounded by resin epoxy. GALRE has better corrosive and fire resistance; moreover, better damage tolerance. One of its known application is in aerospace industries; Airbus A380 Planes uses GLARE Laminates with S-2 Glass fibre. It is used in aerospace from air craft skin to floor panels. It can save weight up to 30 percent comparing to the conventional aluminium alloys used. In addition, because of the foil barriers, resistance to moisture penetration will be higher. Moreover, other superior properties such as fatigue resistance, impact resistance, and lightning striking resistance are some of few GLARE laminate superior properties to the composite industries. (AGY , 2013)

3.3 Sample Preparation Method

Two methods were used for the lamination process. These were: Vacuum bagging and compression moulding method. After, wet-laying of fibre textiles and aluminium foils with prepared resin, necessary set up for lamination processes were organized and prepared.

3.3.1 Vacuum Bag Lamination Methods

Vacuum bag lamination method is used after wet lay-up process for enhancing its final properties by removing entrapped air and excess resin. It uses vacuum to reduce the internal pressure inside the vacuum bag and increasing the external atmospheric pressure to add additional force on the bag. Moreover, the process helps to reduce the styrene emission from the resin.

The process of vacuum bagging includes placing a release chemical in the working table then placing the wet lay-up accordingly. Next, placing release film on top of the lamina following bleeder ply (fibre glass cloth, non-woven fabric) and finally, vacuum bag (PVA, Nylon or polystyrene) will be placed over and sealed with tacky tape. After that, by preparing a vacuum port and checking for leaks, vacuum pressure can be applied. (ACMA, 2016)

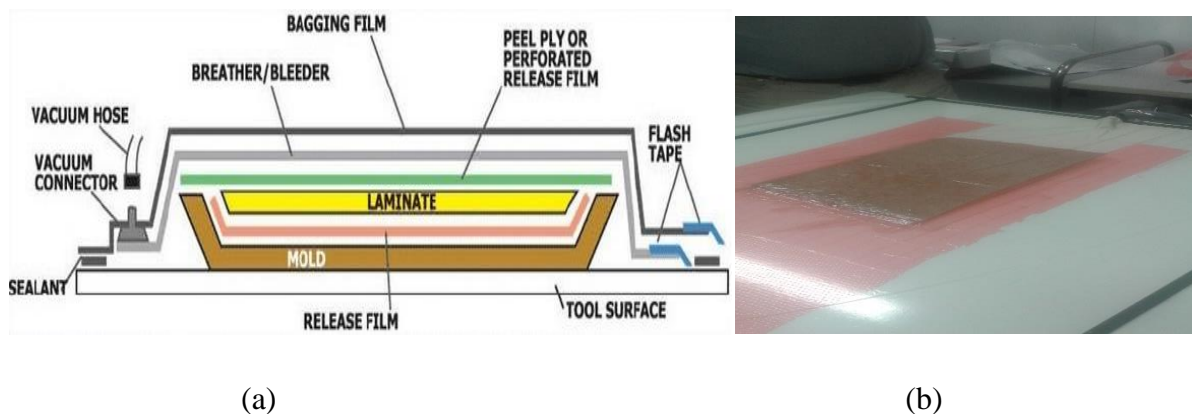


Figure 12: (a) typical Vacuum bagging layup schematic. Taken from: (Craft, 2014) and (b) vacuum bagging setup

3.3.2 Compression Moulding

Compression moulding for composites is a method of using high pressure on matching metal moulds. Compression moulding often enhance mechanical properties for composite parts. After the layup lamina is placed in an open mould cavity, then mould is closed. After that, intended amount of pressure will be applied throughout the curing period. The application of heat helps the curing process with less void and higher fibre volume fraction. (Advanced Composite Inc., 2016)



Figure 13: Compression moulding machine in Arcada UAS Production Laboratory.

3.4 Cyclic Three-point Bending Test

The cyclic compression test helps to evaluate the deformation, fatigue and creep properties of a material. Fatigue failures is one of the typical results on a material undergoing cyclic compressions. In polymers and polymer-composites, viscoelastic behaviour effects on the initialization of fatigue failures.

After setting up the upper and the lower limits for the force, distance and number of cycles in the Testometric testing machine, test samples were placed for testing. After that, each sample results were recorded and saved.

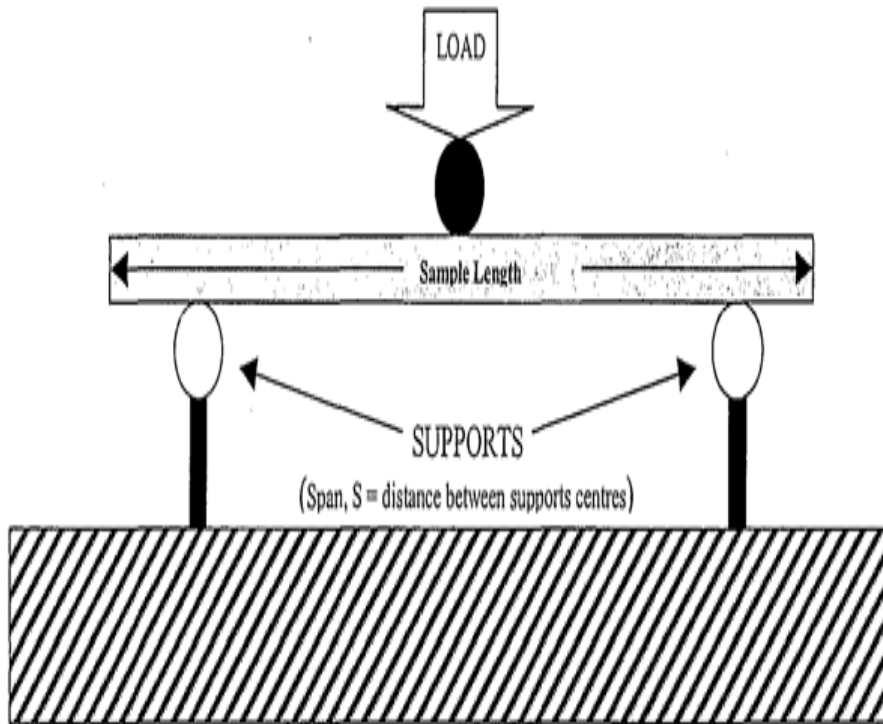


Figure 14: Three-point bending setup. (solution, 2016)

4. RESULTS

4.1 Water up-taking and diffusion rate

The results shown in Figures below indicate change in masses of the sample laminates within a period of time under controlled temperature. The saturation time was recorded to be 127 hours from the initial test. Figure 15 percentile change of mass up taking and the ideal fickean curve can be seen in the figure 16 to elaborate ideal expectation of mass up-taking in diffusion process. In addition, diffusion rate of the samples based on the diffusion equations were shown in figure 18.

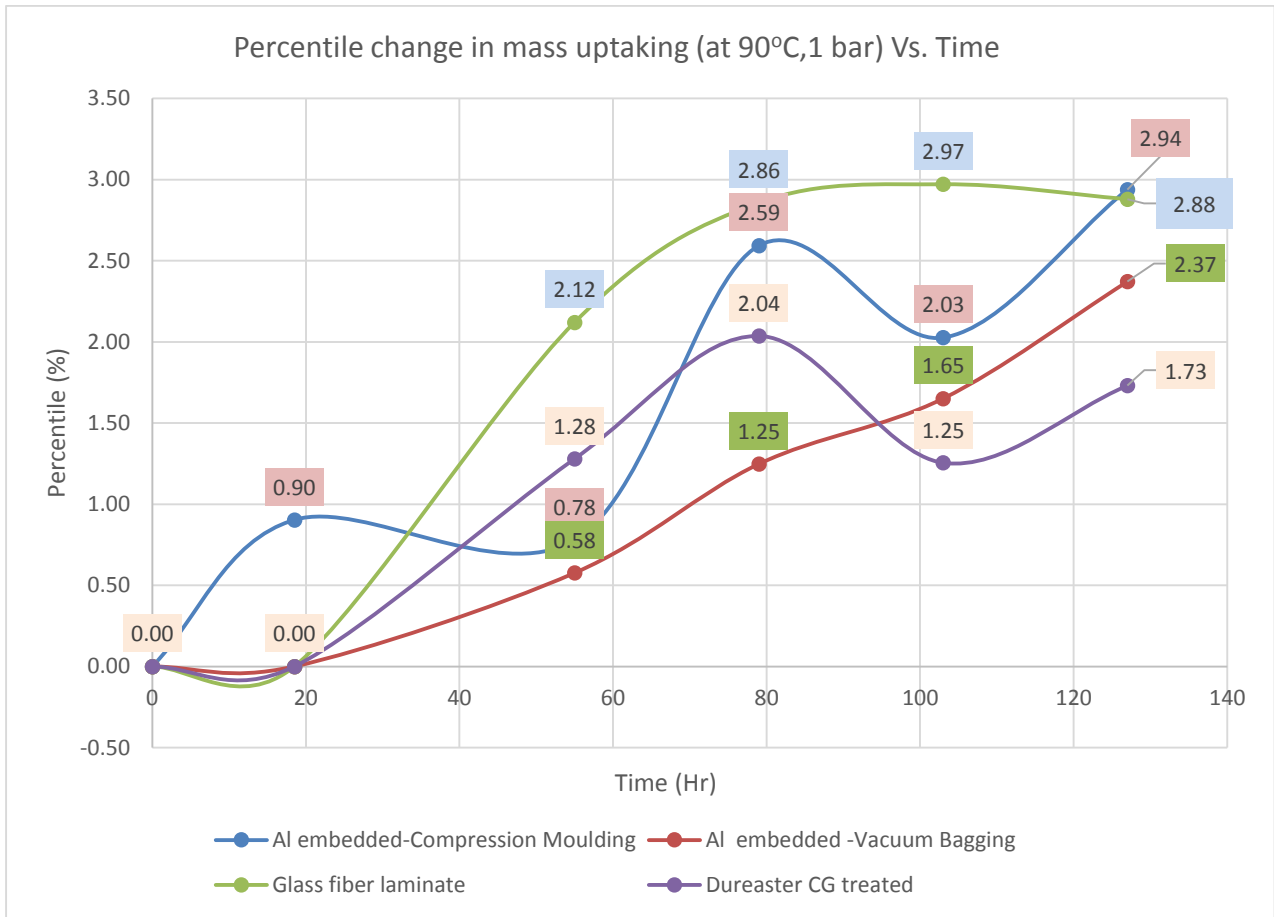


Figure 15: Percentile change in mass up-taking Vs time (at 90 °C , 1 bar)

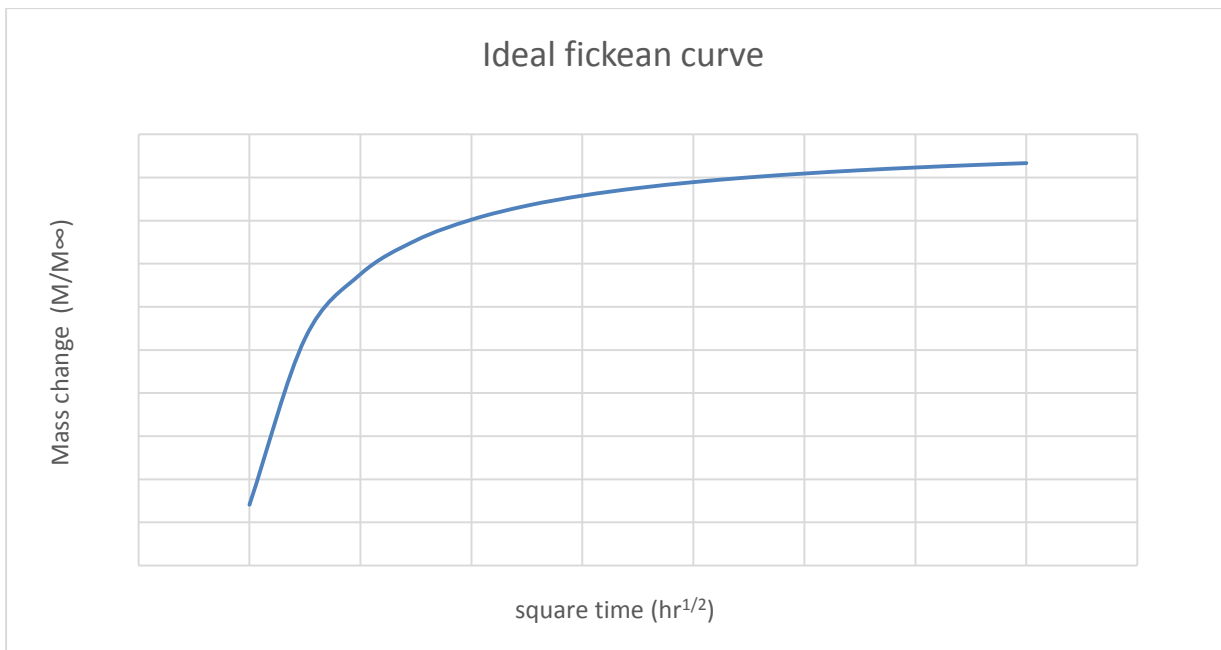


Figure 16: Ideal fickian curve

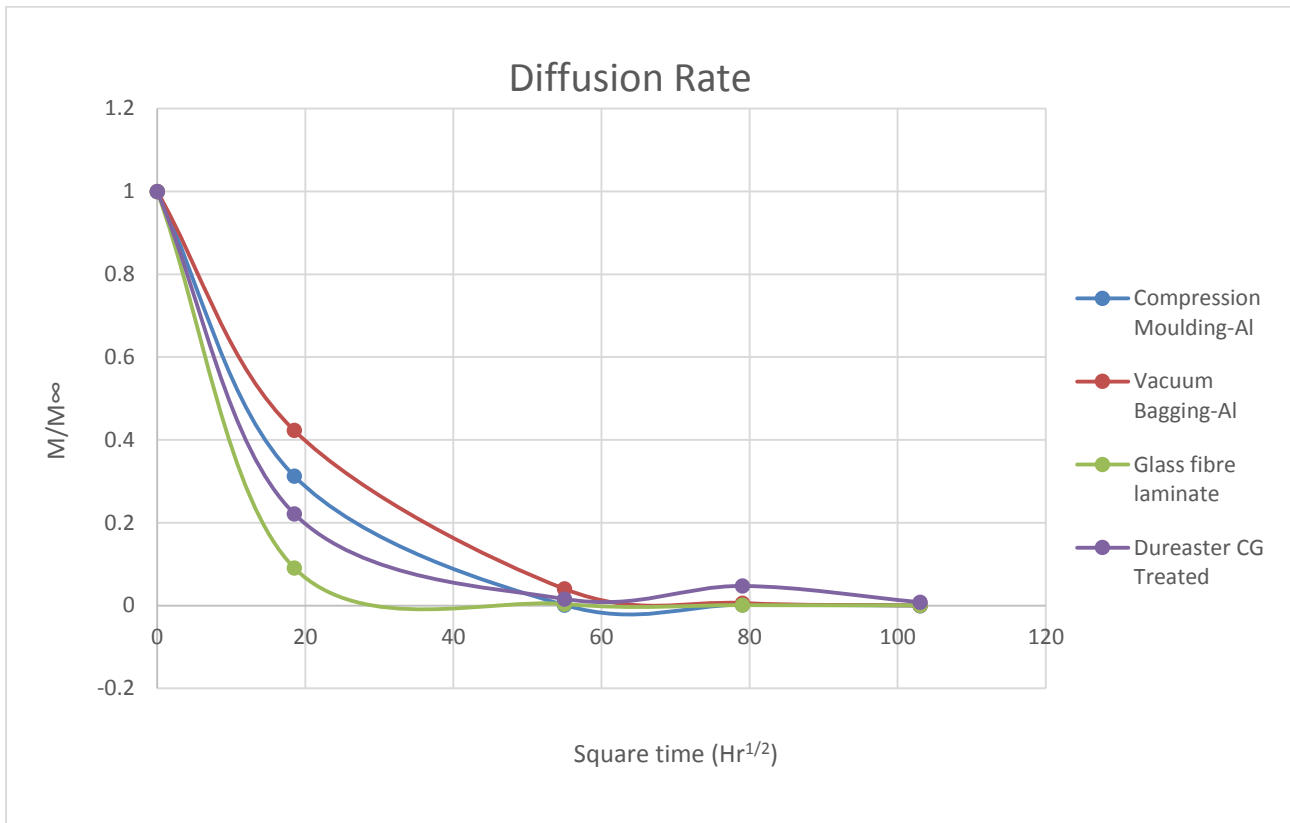


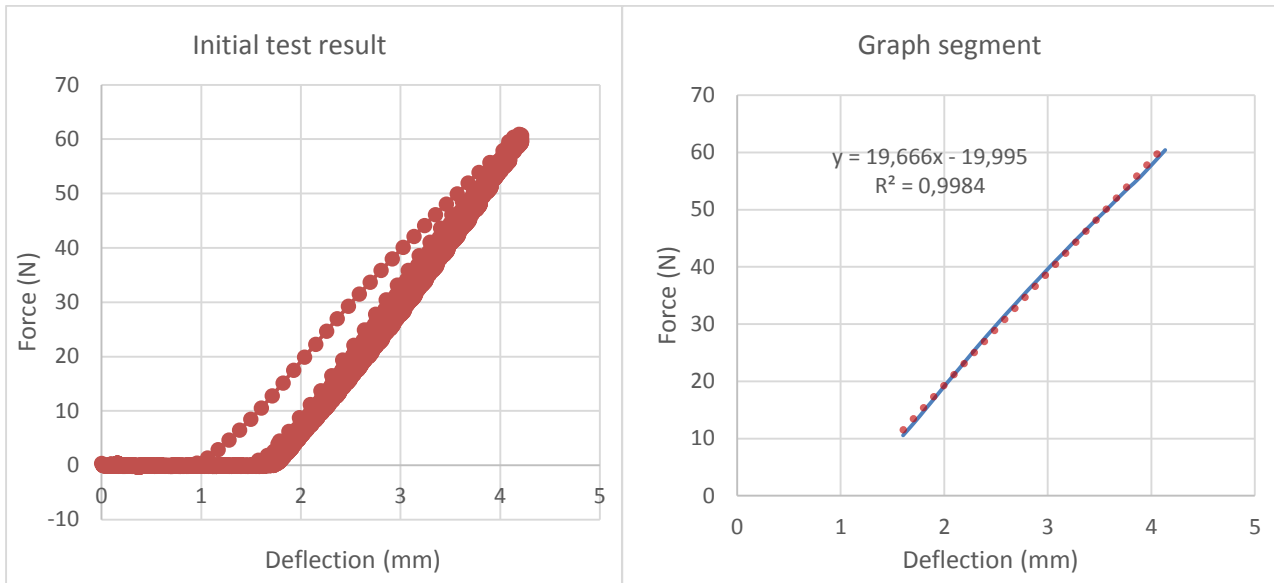
Figure 17: Diffusion rate

4.2 Cyclic three-point bending test results

The results below were retrieved from the cyclic three point bending test. Force Vs deflection graphs were drawn from the raw data and sample segment of those graphs were taken to analyse the change in slope between the initial and final test results. The masses and presently changes were also summarized in tabular and chart formats. Moreover, photographic illustration were presented to show the physical changes of the test sample before and after the test occurs.

4.2.1 Cyclic three-point bending test result for Aluminium-Embedded Compression moulded samples

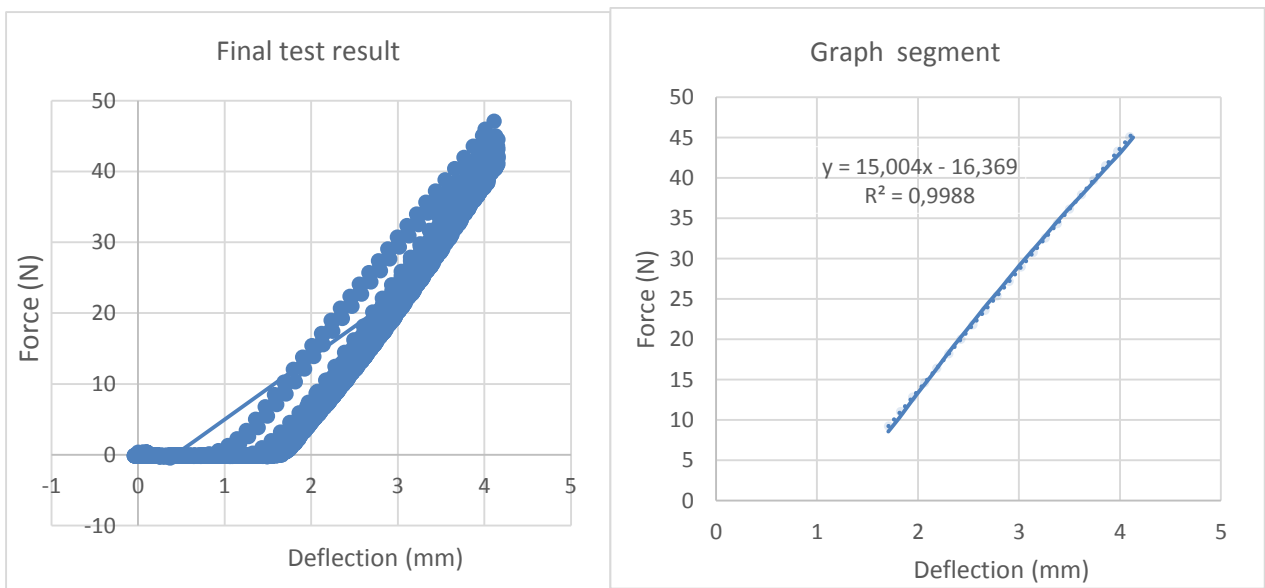
The results below shows the initial and final cyclic three-point bending graph. Graph segment were taken to analyse the slope declination. The slope is directly proportional to the young's modules of the laminate and its declination from initial to final graph segment illustrated the decrease in module and delamination occurs.



(a)

(b)

Figure 18: (a) Initial cyclic results of laminates and (b) graph segment



(a)

(b)

Figure 19: (a) Final cyclic results of laminates and (b) graph segment

The table below summarize the initial and final slope differences which shows the decrease in elastic modules and mechanical degradation at the final test.

Table 4: slope change of the initial and final results

No.	Initial test Slope	Final test slope	Slope difference	Change in percentile
1	19,66	15,00	4,66	23%



(a)

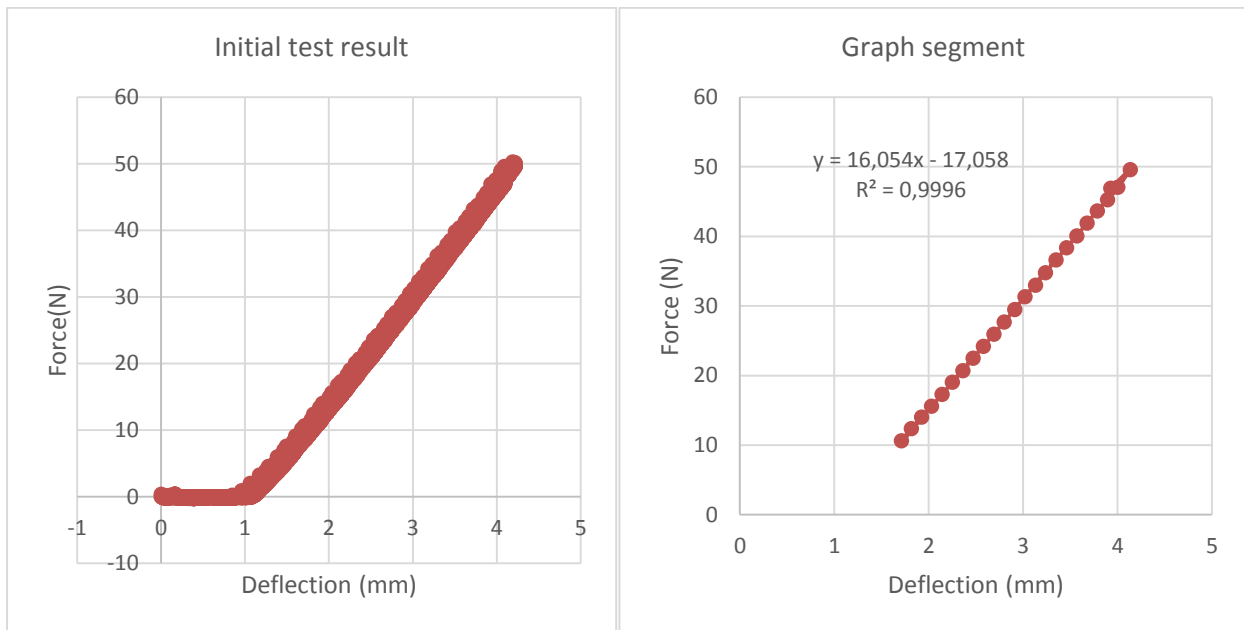


(b)

Figure 20: Physical changes. (a) Before testing and (b) after testing result

4.2.2 Cyclic three-point bending test result of glass fibre laminates

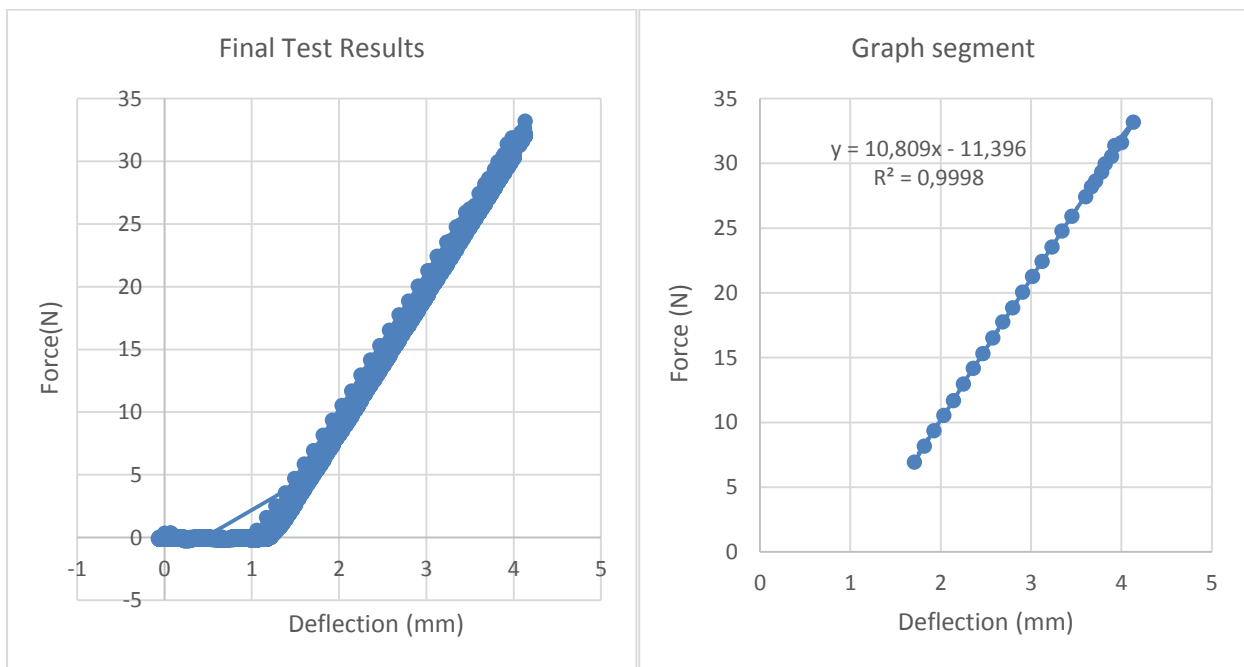
The results below shows the initial and final cyclic three-point bending graph. Graph segment were taken to analyse the slope declination. The slope is directly proportional to the young's modules of the laminate and its declination from initial to final graph segment illustrated the decrease in module and delamination occurs.



(a)

(b)

Figure 21: (a) Initial cyclic results of laminates and (b) graph segment



(a)

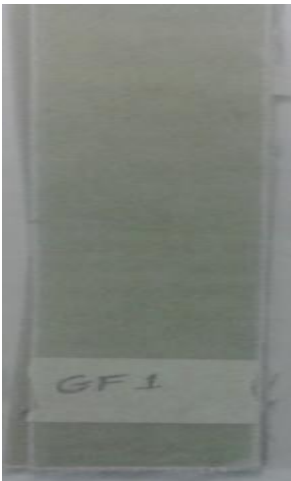
(b)

Figure 22: (a) Final cyclic results of laminates and (b) graph segment

The table below summarize the initial and final slope differences.

Table 5: slope change of the initial and final results

No.	Initial test Slope	Final test slope	Slope difference	change in percentile
1	16,054	10,809	5,245	32%



(a)

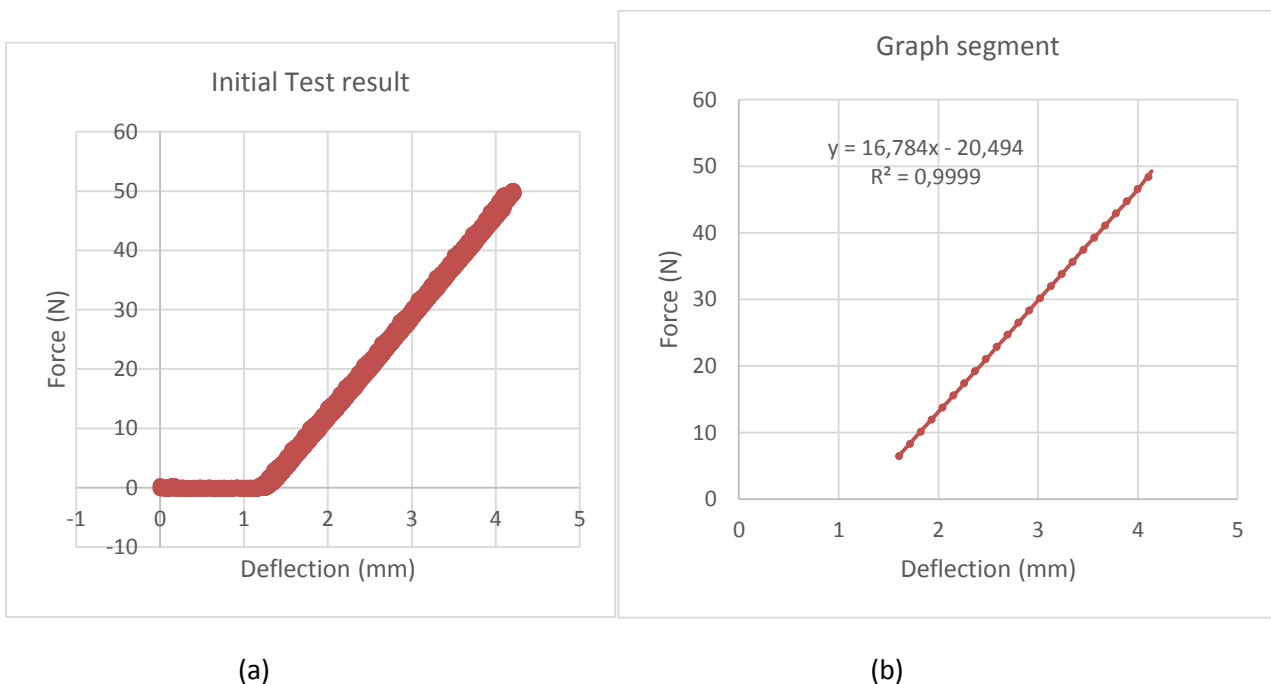


(b)

Figure 23: Physical changes. (a) Before testing and (b) after testing result

4.2.3 Cyclic three-point bending results of Durester CG treated laminates

The results below shows the initial and final cyclic three-point bending graph. Graph segment were taken to analyse the slope declination. The slope is directly proportional to the young's modules of the laminate and its declination from initial to final graph segment illustrated the decrease in module and delamination occurs.



(a)

(b)

Figure 24: (a) Initial cyclic results of laminates and (b) graph segment

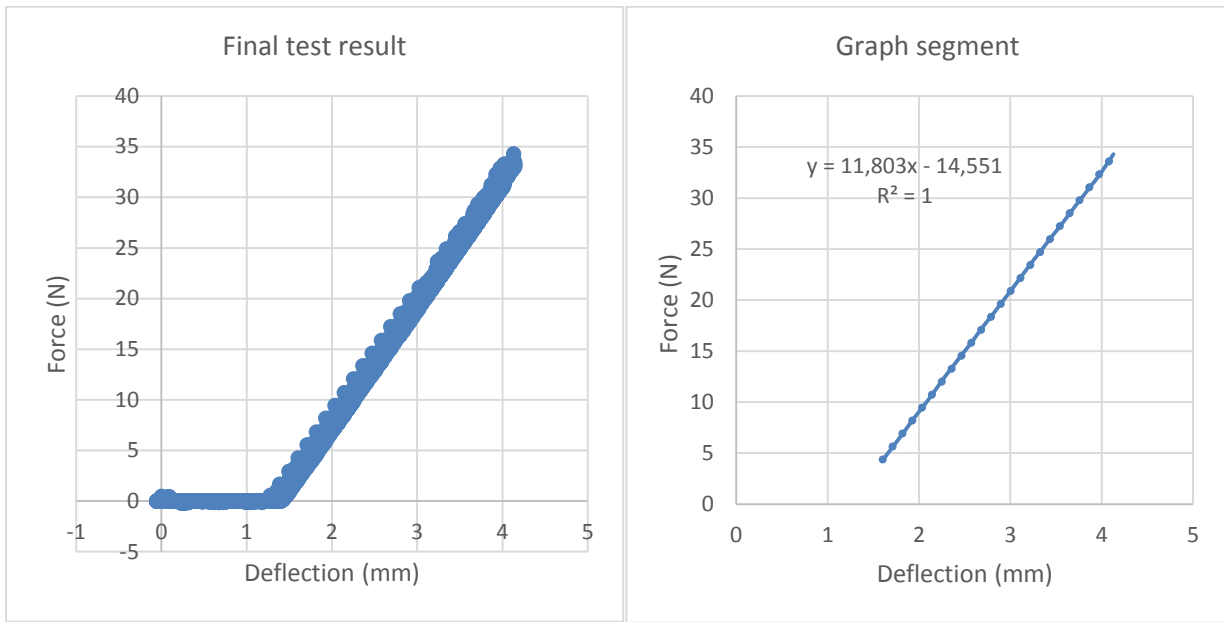


Figure 25: Initial and final graph segments of the sample laminates

The table below summarize the initial and final slope differences.

Table 6: slope change of the initial and final results

No.	Initial Slope	Final slope	Slope difference	change in Percentile
1	16,784	11,803	4,98	29%



(a)



(b)

Figure 26: Physical changes. (a) Before testing and (b) after testing result

4.2.4 Cyclic three-point bending test results of aluminium embedded Vacuum Bagged laminates

The results below shows the initial and final cyclic three-point bending graph. Graph segment were taken to analyse the slope declination. The slope is directly proportional to the young's modules of the laminate and its declination from initial to final graph segment illustrated the decrease in module and delamination occurs.

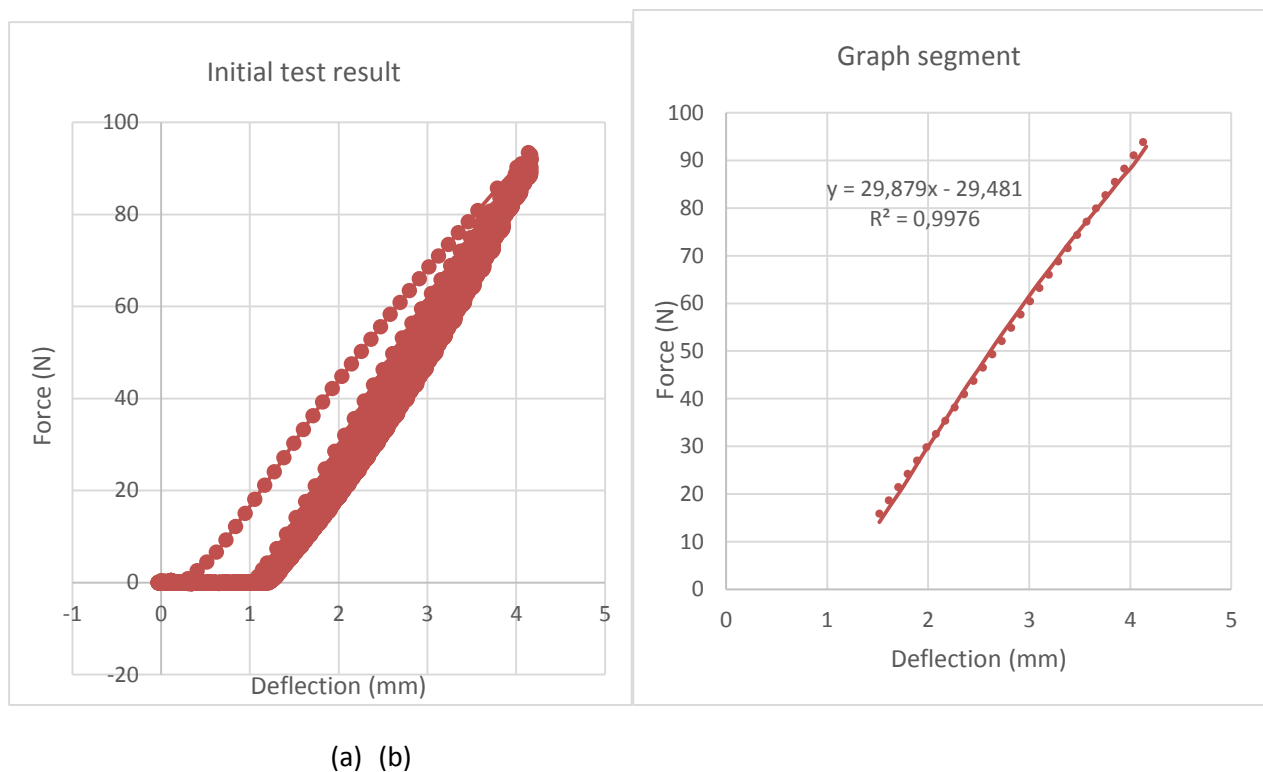


Figure 27: (a) Initial cyclic results of laminates and (b) graph segment

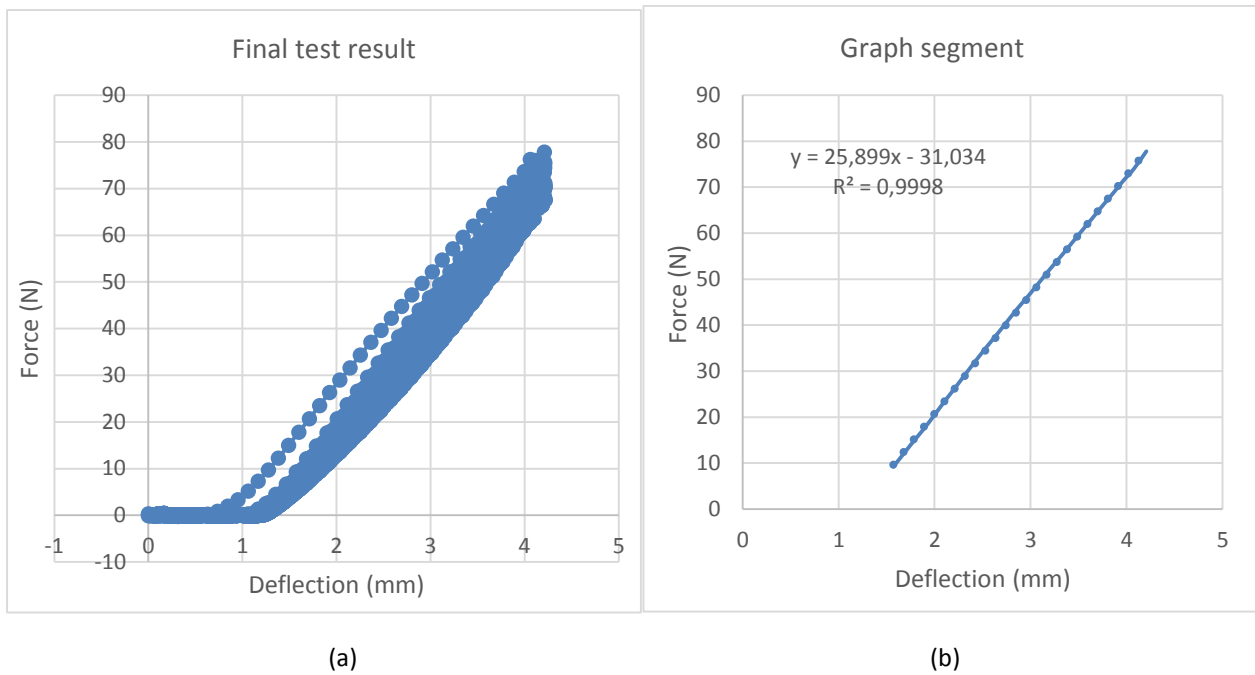
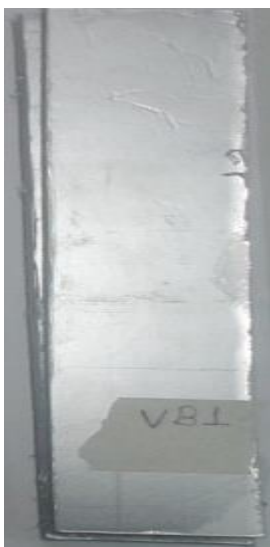


Figure 28: (a) Final cyclic results of laminates and (b) graph segment

The table below summarize the initial and final slope differences.

Table 7: slope change of the initial and final results

No.	Initial Slope	Final slope	Slope difference	Change in Percentile
1	29.87	25.89	3.98	13%



(a)

(b)

Figure 29: Physical changes. (a) Before testing and (b) after testing result4.2.5 Slope change of sample laminates

The chart below summarizes the slope change of graphs between the initial and final cyclic loading tests.

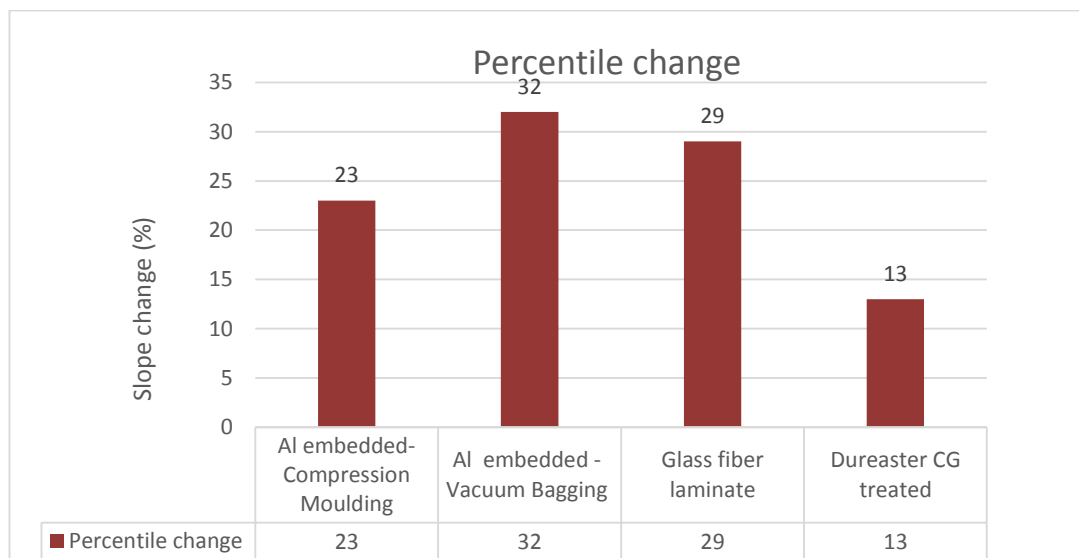


Chart 2: Slope change of sample laminates

4 DISCUSSION

The water up taking and mechanical degradation can be seen from the retrieved testing results. The results clearly indicate the important role of corrosive resistance methods and lamination technique on water permeability. Moreover, Inconsistent weight gains were observed in figure [16] compare to expected water up-taking rate of fickian curve in Figure [17]. This unevenness was caused mainly

because of the introduction of cyclic loads with in a specific period of time; which led to more water up-taking than anticipated.

The first sample group, glass fibre- aluminium embedded laminates were prepared by compression moulding method. In figure [20], Degradation in the outer surface, visible air tightness on the surface and side-delamination can be clearly seen. In addition, figure [15] illustrates significant change in water up-taking test comparative to the other group of sample laminas; which was a 2.94 % increase of mass at the end of the test. Nevertheless, it might be agreed that the production method could add some negative influence when the laminate undergoing curing. However, the result from the cyclic three point bending test in table [4] shows 23 % slope decline, which is equitable decline in s relatively to others and can be concluded that it shows minimum loss of mechanical properties but higher hydrolytic degradation.

The next group of samples was prepared from vacuum bagging process of wet lay-up of only glass fibre fabrics (without corrosive treatment). Apart from some scratched marks and possible internal delamination, the samples were not physically damaged critically; these can be seen in figure [29]. Additionally, as shown figure [15], the diffusion rate was comparatively higher with 2.88% however, test results from the cyclic three point bending test shows that the value in slope change is 29 % showing nearly comparable physical degradation with both the aluminium embedded samples.

The other group of samples were glass fibre laminate (taken/cut from the above glass fibre sample) half side treated by Durester CG. As it is illustrated in figure [26] physical damage were small apart from slighter Fading of paint and scratched surface (in the middle parts). Next, figure [15] water up-taking by the sample were very trivial, with a 1.73 % mass up-taking. Additionally, physical property (fatigue) show insignificant change value which is 13% slope decline.

The last sample laminate which was prepared using vacuum bagging method and embedded with aluminium foils as a corrosive protection result shows that higher mechanical property loss (32% slope decline) than the rest of the sample groups up taking of water increase was 2.37% next to the glass fibre- aluminium embedded samples prepared by compression moulding and from the glass fibre laminates..

Another key factor that can be noted in the comparison of water permeability prevention methods under cyclic loads is the weight; especially for aircraft structures which one of main objective is reducing body weight. The addition of coercive resistance materials or embedding filaments can add over all weight. With that, the addition of aluminium filament can add from 15 to 30 percent of additional weight as it can be calculated from the sample mass table [1]; this can add some degree of impact in the overall weight.

6 CONCLUSION

The result which are retrieved from the experiment partially fulfil the objective and expectation that was intended. The barrier methods that were used for the test shows a better anti-permeability compare to the non-treated sample for a period of time. Nonetheless, this is only can be applicable for structures that are subjected to for water up taking for temporary period. Otherwise, permeation will be inevitable when being exposed for longer period.

There are various factors that attribute to the ageing of composite polymers. Material types, geometrical factors, environmental and physical factors are some of the major factors leading to ageing. In studying the ageing of composite polymers multidirectional ways of approach should be applied because of its complex nature of its diverse constituent. Additionally, composite laminate having layers with different diffusivity coefficient, and diffusion occurs along transverse and parallel direction (2D), the degree of reliability and accuracy of results falls in the analysis process capacity. Therefore, it is necessary to intertwining of ageing studies of the conventional constituent-viscoelastic studies with time-dependent characteristics which can be developed experimentally using isothermal creep compliance tests; These can save time and cost. In addition, it leads better results specially, for ageing occurring with different environmental constituents such as elevated temperature and cyclic loadings.

Even though accurate values were not gathered and difficulties on identifying direction of diffusion occurs, the difference results can be clear indictors of which of the ageing resistance methods can performed under cyclic load conditions plus which method of lamination is preferable for better ageing resistance and mechanical performance. For further studies on identifying and evaluating parallel (through thickness) diffusivity through the multi-layers laminates, other permeation methods

studies such as Isostatic and quasi- Isostatic methods can be used. These methods determine the penetration through different concentration levels. However, the above results can be a starting point for more accurate result and analysis.

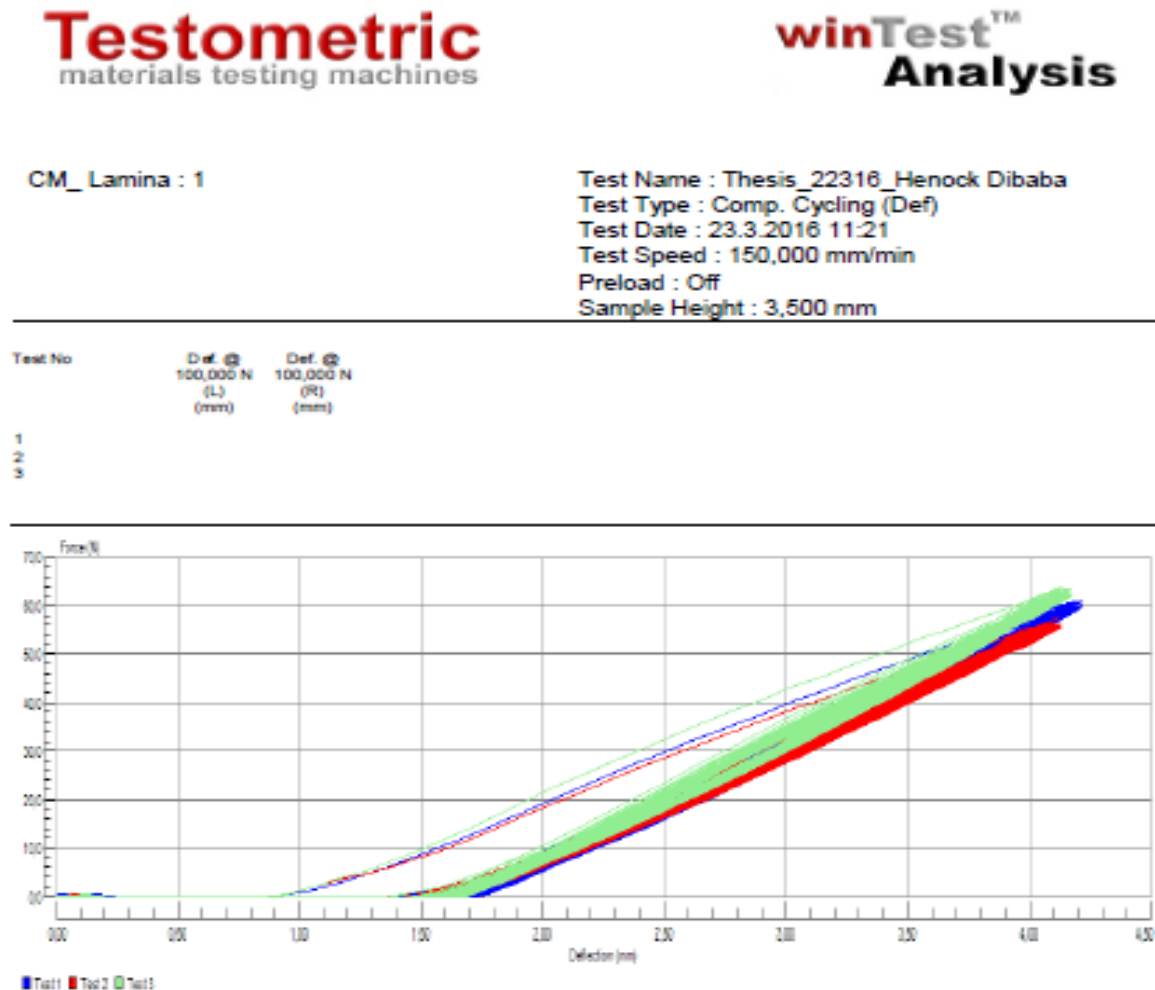
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APENDIX I

Cyclic three point bending test result sample figure



APENDIX II

Sample laminates before conducting test. Taken by personal camera

