The influence of acid surface treatment on the physical and mechanical properties of steel structure as a precursor for fiber metal laminate structure.

BY

EUNICE NG HUI RONG

22630

Dissertation submitted in partial fulfilment of the requirements for the

Bachelor of Mechanical Engineering (Hons)

JANUARY 2020

Universiti Teknologi PETRONAS Bandar Seri Iskandar 31620 Tronoh Perak Darul Ridzuan

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Eunice

EUNICE NG HUI RONG

LETTER OF APPROVAL

I have received and approved, Eunice Ng Hui Rong's dissertation.

Thank you.

Best regards.

Sincerely

Dr. Mazli Mustapha Senior Lecturer Mechanical Engineering Department Universiti Teknologi PETRONAS

ABSTRACT

This project is to study sandwich fibre-reinforced plastic layer where metal layers are combined is known as Fibre Metal Laminate (FML), which is a family of hybrid composite structure. The advantage of a composite with hybrid nature, is that it inhibits the strength of two different constituents' which are metal and fibre that are stack alternatively. FML are able to provide outstanding mechanical properties as compared to the conventional composite with its high strength and corrosion resistance. Surface treatment is one of the methods to improve adhesion between steel structure and fibre which affect the strength of fibre adversely by influencing the chemical structure of fibres and the chemical bonding with matrix will be enhance. Hence, the parameters such as concentration of acid, time taken of immersion, grade of sandpaper and temperature expose were studied to select the most optimum level of parameter by using Taguchi's Method.

ACKNOWLEDGEMENT

It is my heartfelt pleasure to express my gratitude to my supervisor and guide Dr. Mazli bin Mustapha, member of Institute of Materials Malaysia (IMM) and The Institute of Materials, Minerals and Mining, UK. Throughout the process of collaborating with him in this project, his dedication and passion to help students had made a significance to complete my work. His meticulous scrutiny, scholarly suggestions and engineering approach have assisted me to perfect my research.

In addition, I am thankful towards my internal examiner, Dr. Mark Ovanis, for his constructive criticism to improve on my work during Final Year Project I (FYP). His remarks challenged me out of my comfort zone to improve on my research skills in order to defend my field of studies more precise.

Furthermore, I would like to take this opportunity to thank Mr. Daniel and Mr. Adam the laboratory technicians to share their experience and expertise to conduct the experiment. Their patience and their dedication to build students to be a well-rounded potential engineer for the real world is admirable and impactful to the world.

Lastly, I am grateful for Dr. Dher M Badri Albarody's time to be an external examiner for this FYP II session, during this difficult time of pandemic that we are currently in, here in Malaysia.

TABLE OF CONTENTS	1
LIST OF FIGURES	2
LIST OF TABLES	3
Chapter 1: INTRODUCTION	4
1.1 Background of Study	4
1.2 Problem Statement	6
1.3 Objective	6
1.4 Scope of Study	7
Chapter 2: LITERATURE REVIEW	
2.1 Surface Treatment of Carbon Fibre	8
2.2 Fibre Metal Laminate	10
2.3 Hydrochloric acid vs Nitric acid	11
2.4 Effect of Surface Roughness	13
2.5 Laminate Material Mechanical Properties	13
2.6 Advantages of Surface Treatment on Carbon Fibre	15
2.6 Design of experiment (Taguchi's Method)	16
2.7 Orthogonal Array	17
Chapter 3: METHODOLOGY	19
3.1 List of Equipment and Material.	19
3.2 Project Activities	20
3.3 Process Flow	22
3.4 Taguchi's Design Flowchart	23
3.5 Gantt Chart	26
3.6 Key Milestone	27
Chapter 4: RESULTS AND DISCUSSION	
4.1 Results	
4.2 Discussion	31
Chapter 5: CONCLUSION AND RECOMMENDATION	32
REFERENCES	34

TABLE OF CONTENTS

LIST OF FIGURE

Fig 2.1: Surface scan of the corroded heat-treated and untreated NST 37-2 steel
coupons with different concentrations of hydrochloric acid solution [11]8
Figure 2.2: Untreated carbon fibre [9]10
Figure 2.3: Treated with hydrochloric acid [9]10
Figure 2.4: Treated with nitric acid [9]10
Figure 2.5: Average surface roughness of samples after mechanical material removal stage [20]
Figure 2.6: Carbon Fibre Reinforced Polymer (CFRP) core laminate material [10]12
Figure 2.7: Manufacturing Carbon Fibre Reinforced Polymer [10]12
Figure 2.8: Specimen undergoing 3-point bending test [10]13
Figure 3.1: Time Taken Specimen undergo heating process
Figure 3.2: Schematic drawing of vacuum bagging24
Figure 4.1: Hardness vs. Project Run Number25
Figure 4.2: Effect of temperature on hardness
Figure 4.3: Effect of roughness of sandpaper on hardness
Figure 4.4: Effect of time on hardness27
Figure 4.5: Effect of the concentration of HCl on the hardness27
Figure 4.6: Parameters of the effect on the hardness
Figure 5.1: After laminating with adhesive fibre onto steel

LIST OF TABLE

Table 2.1: Advantage of Fibre-Metal Laminate [3]	11
Table 2.2: Tensile strength (σ) of carbon fibre samples untreated and treated with hydrochloric and nitric acids [9]1	12
Table 2.3: Factorial design vs Taguchi's method [7]1	16
Table 2.4: L9 Orthogonal Array Matrix [8]	18
Table 3.1: List of Equipment1	9
Table 3.2: List of Raw material1	9
Table 3.3: The processing parameters and the levels for L9(34) Orthogonal Design Matrix	24
Table 3.4: The L9(34) (parameter assigned) with response	24
Table 3.5: Gantt chart2	26
Table 3.6: Key Milestone	27
Table 4.1: Results of hardness level of samples	28

CHAPTER 1: INTRODUCTION

1.1 BACKGROUND OF STUDY

When metal skin and polymer core that are in sandwich structure, it offers a large variety of benefits that are largely related to their high bending strength, light weight and good impact resistance properties [1]. Numerous of researches have carried out experiments of the characteristics of the bond between the external bonded Carbon Fibre Reinforced Polymer (CFRP) materials and steel plate, whereby there are factors that make a notable influence on the bond properties which consist of intrinsic and extrinsic [2]. As carbon fibres are naturally stiff, it may give allowance to be the bridge among fatigue cracked steel sheets and prevent crack development [3].

A group of a cross breed composite structure that is framed from the mix of metal layers sandwiching a fibre-strengthened plastic layers is known as Fibre Metal Cover (FML) [4]. It is made out of metal stacked on the other hand with fibre strengthened composite layers to have the option to have the points of interest cross breed nature from two unique constituents (Metal and fibre) [4], Since the FML gives a brilliant mechanical properties like exceptional solidarity to weight proportion and high consumption opposition when contrasted with customary composite lamina [17].

Without surface treatment, the carbon fibre produce composites with low interlaminar shear quality (ILSS), this is because of the frail attachment and poor holding between the fibre and lattice [5]. Surface treatment builds the surface territory and surface acidic utilitarian gatherings, in this manner, the bond between the fibre and sap framework are improved. This procedure can be arranged into oxidative and non-oxidative medicines [19]. Oxidation medications include gas-stage oxidation, fluid eliminate oxidation are conveyed artificially or electrochemically and reactant oxidation. In any case, the non-oxidative treatment would include testimony of progressively dynamic type of carbon, for example, the affidavit of pyrolytic carbon and the exceptionally viable whiskerization [18]. To improve the holding between the fibre and network since carbon-fibre can likewise be plasma treated however fluid stage oxidation treatment are milder, compelling and liked. The Taguchi's Method is chosen as the design of experiment as it is a powerful statistical technique that enables to study the effect of the multiple variables that are provided simultaneously to save time. To achieve accelerated improvements, statistic and robust engineering will be able to manufacture processes of a product [7]. The most effective quality building tools is by executing it with this standardized version of Design of Experiment (DOE) as it used by all engineers in all type of manufacturing activities. Dr. Genichi Taguchi introduced this statistical model in order for the effect of the various parameters to be studied. In this case the parameters that were focused in this experiment were temperature, heat rate, concentration of acid and grade of sandpaper. By obtaining this matrix, the experiment could be run with the parameters that are able to yield the formation oxide layer on the steel structure to increase the mechanical properties.

In this study, the influence of different acid surface treatment on the fabrication of carbon fibre-steel by manipulating the variable such as concentration of acid, time of immersion of acid, temperature of solution and grade of sandpaper to understand the mechanical properties.

1.2 PROBLEM STATEMENT

Steel is used as a substrate material when fabricating of fibre metal laminate structure. The interfacial bonding of carbon fibre steel laminated structure is directly correlated to the quality of surface finishes with the formation of stable iron oxide layer on steel. In addition, under normal oxidizing environment, the magnetite layer can be highly sensitive and easily transformed into hematite layer. In order to overcome this issues, the formation of magnetite layer must be dictate by controlling the processing parameters for acid surface treatment such as concentration of acid, temperature, surface roughness and time of treatment. Hence, it is crucial to analyse and evaluate the influence of these factors on the interfacial delamination performance of Carbon Fibre Reinforced Polymer (CFRP) composite and steel structure.

1.3 OBJECTIVES

The objective of this study is based on the influence of acid surface treatment on the physical and mechanical properties of steel structure as a precursor for fiber metal laminate structure. The objectives are as follows:

• To determine the optimum processing parameter of acid surface treatment of the formation oxide layer on the steel structure.

1.4 SCOPE OF STUDY

This study encompasses several parameters that are able to manipulate the mechanical properties of carbon fibre. The parameters that will be our variables are concentration of acid, time of immersion of acid, temperature of the solution, and grade of sandpaper. By using Taguchi method it will filter the optimum parameter to create the best framework to produce results that are able to prove the hypothesis.

From the objectives this research will be focused on the parameters that are listed below:

1. Optimizing processing parameters statistically; concentration of acid, time of immersion of solution, temperature of immersion of acid and grade of sandpaper.

- By utilizing Taguchi's Method of orthogonal array model, number of experiments and runs required will be determine to study the effect of processing parameters as levels are being vary.
- The results will be remodelled in the signal to noise ratio (S/N) to curate into a single value from the multiple raw information gathered.
- To confirm the importance of each operation specification the Analysis of Variance (ANOVA) technique will be used to treat the surface of carbon fibre steel with the most optimum level of processing parameters.

CHAPTER 2: LITERATURE REVIEW

2.1 SURFACE TREATMENT OF CARBON FIBRES

Surface treatment can be done by oxidation process where it includes gasphased oxidation, liquid-phase oxidation that may be done chemically and electrochemically. Carbon fibre that are not treated, produced composite that has low outer laminar shear strength (ILSS). This characteristic attributes to poor bonding between fibre and matrix and weak adhesion [4]. Surface treatment on carbon fibre improves the adhesion process by enhancing the roughness which result in the increase of surface area and number of contact points. In addition, surface treatment incorporates reactive functional groups with chemical reactions to promote chemical bonding with polymer matrix [5].

The most common used of heat treatment on carbon steels are annealing, normalizing, hardening and tempering. In order to soften carbon steel materials, annealing is most often used and it also refines its grains due to ferrite-pearlite microstructure [12, 13]. During the process of normalizing, material is heated to the range of austenitic temperature and to obtain a main pearlite matrix, the material is followed by air cooling which leads into the increase of strength and hardness [14]. While in the hardening process, the material is expose to heat in a temperature that is high enough to further form austenite, as it is held at a constant temperature until the amount of carbon desired has been dissolved. Next, as it is at a suitable rate in oil or water to achieved a maximum yield strength 100% martensite. However, it could cause the material to be brittle, hence, this is unlikely the first choice used for engineering applications. The properties of quenched steel were modified by tempering to increase ductility, decrease hardness and impact strength moderately which result in changes in microstructure depending on tempered temperature to be bainite or carbide in the matrix of ferrite. In this study from other researchers, the samples that were heat-treated and untreated NST 37-2 steel coupons were immersed in 1.0, 1.5 and 2.0 M HCl solutions. Although corrosion rate increased linearly with the increase of concentration on both samples, but with time of immersion in test solution could varied nonlinearly. The indication of the increase of linear in both electrode potential and corrosion rate as well as the increase of concentration of acid could be the attribute to the high acid concentration in the increase ion exchange. This is shown in **Figure 2.1**.

Heat Treatment	Concentration of HC1				
neat freatment	1.0 M	1.5 M	2.0 M		
Untreated			Per a		
Hardened					
Normalised		*			
Annealed	19/200		Part of		
Tempered		C.C.			

Figure 2.1: Surface scan of the corroded heat-treated and untreated NST 37-2 steel coupons with different concentrations of hydrochloric acid solution [11].

From **Figure 2.1**, the accelerated corrosion rate observed in the untreated sample caused by the increase of dissimilar metal composition of material which led to galvanic corrosion in the ferritic + pearlitic matrix on the untreated sample. The tendency to be more corrosion resistant than the dual-phase matrix of the untreated sample in the martensitic matrix of hardened sample. In other research, it was reported that annealing heat treatment of dual phase steel which the amount of martensite microstructure increase [15]. Compared to the dual phase of untreated sample, the ferritic matrix and single phase pearlitic of normalized and annealed samples respectively have higher tendency to have resistance to corrosion. Furthermore, steel with martensitic microstructure showed lower rate of corrosion [16].

2.2 FIBRE METAL LAMINATE

A composite is a material that is made out from two or more constituent that are different significantly material in terms of their physical or chemical properties. Fibre Metal Laminate takes the benefit of the characteristic of metal and fibrereinforced composite with the combination of metal and that are different significantly composite metal. This experiment by other researchers as shows in **Table 2.1**, produces a superior mechanical properties instead of the conventional lamina which only consist of fibre-reinforced lamina or aluminium alloy [3].

Key Parameters	Details
High strength	FML's are hybrid structures based on thin metal alloy sheets and plies of fiber-reinforced polymeric materials. Metal and fiber reinforced composites both which have high strength and stiffness result in high strength and stiffness FML's.
Low density	Due to the presence of thin layers of metals and composite piles, it has low density. So, FML's are a weight saving structural material compare to others.
Excellent corrosion resistance	FML's gives excellent moisture resistance and high corrosion resistance because of polymer based.
Excellent moisture resistance	Due to the presence of metal layers at outer surface the moisture absorption in FML's composites is slower when compared with polymer composites, even under the relatively harsh conditions. Additionally pregreg layers are able to act as moisture barriers between the various aluminium layers inside of the FML's.
High fatigue resistance	It gives high fatigue resistance because of intact bridging fibers in the wake of the crack, which restrain crack opening. FML's have excellent fatigue characteristics over conventional metal and composite.
High energy absorbing capacity	Based on investigation data, FML's are absorbing significant energy through localized fiber fracture and shear failure in the metal plies.
High impact resistance	Impact deformation is actually a significant advantage of FML's, especially when compared to composites.

Table 2.1: Advantages of Fibre-Metal Laminate [3].

2.3 HYDOCHLORIC ACID VS NITRIC ACID

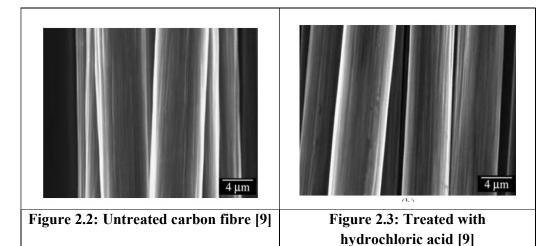
A test was raced to show the normal estimation of elasticity of carbon fibre untreated, one treated with hydrochloric and one treated with nitric corrosive. The carbon strands experience oxidization process in concentrated hydrochloric with a level of (35% (w/w)) and nitric (97 %(w/w)) acids at 103 ± 0.2 °C for a span of 5, 10 and 20 minutes. After the treatment, the examples were washed altogether and bubbled naturally in demineralized water and dried in a stove at 105 ± 0.2 °C for 2 hours [9]. This experiment in **Table 2.2** proves to show that the longer carbon fibre gets treated, the value of tensile strength will increase, and the focus of this experiment is that when carbon fibre is treated with acid in comparison to alkaline, the tensile strength appears to be much higher compare to nitric acid treatment.

	Hydroch	loric Acid Treatment	t o (MPa)	Nitric Acid Treatment σ (MPa)		
as-received fiber	5 minutes	10 minutes	20 minutes	5 minutes	10 minutes	20 minutes
2143 ± 471	2122 ± 443	2040 ± 489	1824 ± 644	1986 ± 522	1924 ± 658	1531 ± 605

 Table 2.2: Tensile strength (σ) of carbon fibre samples untreated and treated

 with hydrochloric and nitric acids [9].

By alluding to the SEM picture in **Figure 2.2**, **Figure 2.3** and **Figure 2.4**, the carbon fibre test that was untreated demonstrated a smoother surface contrasted with the carbon fibre that was treated with nitric corrosive. In any case, the carbon fibre that was treated with hydrochloric corrosive didn't show a lot of huge contrast with the untreated carbon fibre. Which was the ideal result when hydrochloric corrosive is utilized to be treat carbon fibre since it has the most noteworthy sharpness [9].



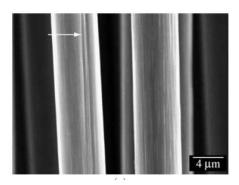


Figure 2.4: Treated with nitric acid [9]

2.4 EFFECT OF SURFACE ROUGHNESS

Surface roughness is reduced by a removal process mechanical material which is grinding and polishing [20]. Chemical etching by HCl is conducted on the surface of low carbon steel since the environmental effect on low carbon steel and to measure the effect of HCl on surface roughness and to analyse by comparing the loss of materials by exposing time [11,21]. In this experiment from this research paper as shown in **Figure 2.5**, the samples with increasing surface roughness are expose by chemical etching to 6M HCl [21]. From the figure below, the standard deviation for early stages of mechanical material removal stages are higher than the late stages of removal stages. The results shows that overall surface roughness is reduced from 115.8±38.2 nm to 54.8±7.7nm, hence, the surface finish and surface homogeneity are 52.7% and 79.8% improved by mechanical material removal process.

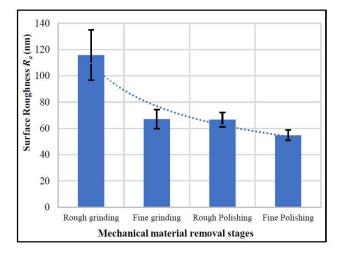


Figure 2.5: Average surface roughness of samples after mechanical material removal stage [20].

2.5 LAMINATE MATERIAL MECHANICAL PROPERTIES

There are an assortment of favourable circumstances where sandwich structure that are made of metal skin and polymer centre could offer as shown in **Figure 2.6**. This is chiefly identified with their light weight, high twisting quality and great effect obstruction properties. At the point when the glue layer bonds the metal and polymers, it enables the heap to move, the general solidness, the attachment of the structure and it is capable on the delamination and a basic issues for multilayer materials [10].

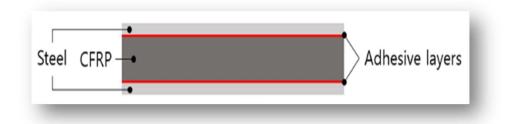


Figure 2.6: Carbon Fibre Reinforced Polymer (CFRP) core laminate material [10].

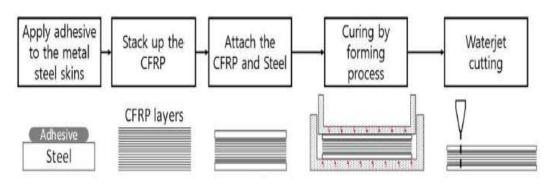


Figure 2.7: Manufacturing Carbon Fibre Reinforced Polymer [10].

Figure 2.7 above from other research, shows the manufacturing process of Carbon Fibre Reinforced Polymer (CFRP) core laminate structure. First, apply the adhesive on the metal steel skin then stack the CFRP onto layers. The CFRP and steel are cured by forming process. Lastly, the specimen is cut through waterjet.

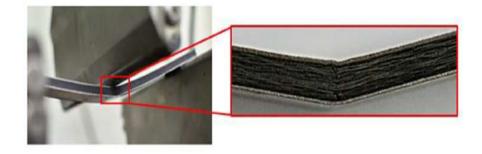


Figure 2.8: Specimen undergoing 3-point bending test [10]

The figure in **Figure 2.8**, shows an examination directed to test on the mechanical properties on overlaid carbon fibre steel by running a 3-point twisting test. The outcome between the overlaid example and the one without overlaying, showed that the covered material had the option to hold up under a higher burden in contrast with just steel examples [10]. This demonstrates the layup configuration reinforces the example to withstand a high measure of burden.

2.6 ADVANTAGES OF SURFACE TREATMENT ON CARBON FIBRE

In spite of the fact that carbon fibre are high in cost, it is the most favoured material for elite composite. Notwithstanding, its surface is artificially inactive which prompts various measure of potential outcomes of inadequate bond, in this way, bringing about a more fragile composite as opposed to the normal one. The upsides of surface treatment on carbon fibre is that it can enhance the bond by physical changes whereby it upgraded the unpleasantness and that would create result of bigger surface zone and increment number of contact focuses, miniaturized scale pores or surface pits on permeable carbon fibre surface. Moreover, it incorporates the substance responses that prompts the consideration of practical receptive gatherings that energize great compound holding with polymer network [10]. Surface treatment is a significant procedure to empower the fibre's surface to be scratches which influences the quality of the fibre.

2.7 DESIGN OF EXPERIMENT (TAGUCHI'S METHOD)

Sir R.A Fisher at first developed a framework that can spread out course of action of exploratory examination which incorporates a few parameters and this procedure was outstanding as "factorial structure of investigations" [23]. Given the plan of segments, satisfactory factorial diagram will separate each possible blend [7]. A full factorial arrangement would cost generously and a lot of experimentation, since designing examinations frequently incorporate numerous parameters. So as to decrease the amount of examinations, a couple of set from one of the potential results will be picked [22].

"Halfway portion test" or "partial fraction experiment" is known as the procedure to pick a destined number of appraisals which makes the most data [8]. In any case, there are no limitations for its utilization of examination of the outcomes obtained by the tests in spite of the way that this strategy is prominent. Taguchi's Technique which was worked by Dr. Genechi Taguchi, can organize a remarkable general arrangement of decides for factorial test that spread different building applications. In this research, there will be four factors and three levels of variations for each factor which reduce the number of experiment to nine times. Below shows the table where it compares Taguchi's method by proving how it is able to decrease the number of experiments while investigating the effects of all processing parameters.

Factors	Level	Total number of experiments			
1 actors	Lever	Factorial Design	Taguchi's Method		
2	2	4(2 ²)	4		
3	2	8(23)	4		
4	2	16(24)	8		
7	2	128(2 ⁷)	8		
15	2	32,768(215)	16		
4	3	81(34)	9		
		_			

Table 2.3: Factorial design vs Taguchi's method [7]

2.8 ORTHOGONAL ARRAY

To get Taguchi's strategy to work, there should be the symmetrical exhibits as it gives a lot of well-adjusted investigation, help in information examination, fill in as target work for advancement and forecast of ideal outcomes [7,8]. The symmetrical exhibit configuration help to ponder the impact of a wide range of parameters. Every one of the exhibits is intended for a particular number of autonomous plan factors and level while numerous standard symmetrical clusters are accessible. In view of the Latin Square arrangement, symmetrical exhibits are planned in different design.

For this examination, there are three degrees of variety that have been curated for every parameter as a control factor that can be assess in a non-linearity over the scope of control factor contrasting with the two levels which can be assessed in the direct impacts of elements. The orthogonal layout design can be formed using the following equation:

$$L_n(3_k) k = (n-1) / 2$$

L = Latin Square layout

n = number of experiments

k = greatest number of factors that can be investigated using the design layout

By using the degree of freedom, Taguchi's method is able to calculate the minimum number of experiment that needs to be conducted.

NTaguchi = $1 + \Sigma$ (number of levels for each factors-1) NTaguchi = $1 + \Sigma$ ((3-1) + (3-1) + (3-1) + (3-1)) N_{Taguchi} = 9 experiments For this examination the L9(34) symmetrical plan grid was picked as it includes four preparing parameters while running three levels for each factor and utilizing the degree if opportunity approach. To comprehend the impact of every one of the four parameter, nine investigations are expected to comprehend the properties of L9(34) symmetrical plan framework as would give the client a further understanding in the information improvement. From the table underneath, the setting seems equivalent in the vertical segment at the quantity of time. For example, level 1,2 and 3 seemed multiple times under factor four, this is known as the equalization property of symmetrical cluster [8].

L ₉ (3 ⁴) Orthogonal Array							
Experiment #	Variable 1	Variable 2	Variable 3	Variable 4	Performance Parameter Value		
1	1	1	1	1	p1		
2	1	2	2	2	p2		
3	1	3	3	3	р3		
4	2	1	2	3	p4		
5	2	2	3	1	p5		
6	2	3	1	2	рб		
7	3	1	3	2	p7		
8	3	2	1	3	p8		
9	3	3	2	1	р9		

Table 2.4: L9 Orthogonal Array Matrix [8].

CHAPTER 3: METHODOLOGY

3.1 EQUIPMENT AND MATERIAL

Table 3.1 and **Table 3.2** is the lists of equipment and raw material that will be

 extensively used in this entire project.

NO.	EQUIPMENT	LOCATION
1	Vacuum Bagging Machine	Block 14, UTP / EPIC
2	Rockwell Hardness Test Machine	Block 17, UTP / AMREC, SIRIM
		Sdn Bhd.
3	Laboratory Oven	Block 17, UTP / AMREC, SIRIM
		Sdn Bhd.

Table 3.1: List of Equipment

Table 3.2: List of Raw material

NO	RAW MATERIAL	AMOUNT
1	Steel	1 kg
2	Carbon Fibre	1 kg
3	Resin	0.1 g

3.2 PROJECT ACTIVITIES

This study mainly constitutes the research methodology of detailed operational procedures which will be based on Taguchi's Design of Experiment. Depending on the availability of the raw material which is carbon fibre plate and the capability of laboratory equipment, phase of the experiment in laboratory, optimization factors (based on Taguchi's theoretical analysis), compound characterization and lastly the relevance of each parameter which includes in an analysis. Below are the details of each activities.

Carbon fibre steel undergo oxidation process by immersing it into hydrochloric acid with a temperature above room temperature. Before oxidizing the carbon fibre steel, the specimen was grinded with sandpaper to smoothen the surface. Parameters such as concentration of acid, time of immersion of acid, temperature of immersion of solution and grade of sandpaper was used to test to acquire the most optimum level to achieve desired result.

3.2.1 Grinding

Samples undergo mechanical material removal by grinding it with sandpaper according to the different grade of sandpaper. This process was to eliminate excessive oxidation layer that was there before the surface treatment to maintain a constant variable in this experiment. The grinding process was executed in a uniform direction at a 90° angle vertically to ensure consistency in the surface roughness.

3.2.2 Heating process

There are variables that should be considered such as temperature, rate of cooling after heat treatment and length of time. This would impact on the properties of metal significantly. The treated samples were heated in a furnace until it reaches 800°C then the temperature will be kept at constant for 120 minutes and left to cool for 24 hours. This process was to achieve desired physical and mechanical properties, heat treatment enable to modify the crystalline structure. Hence, it should increase hardness, toughness, improve ductility and maximized corrosion resistance.

The graph below shows the relationship between the time taken in the furnace and temperature expose to the sample.

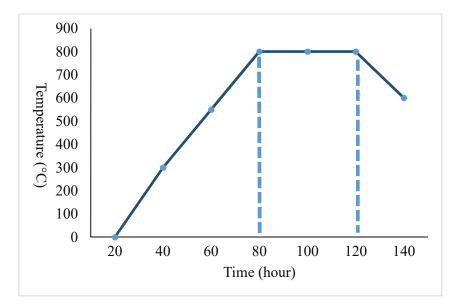


Figure 3.1: Time Taken Specimen undergo heating process

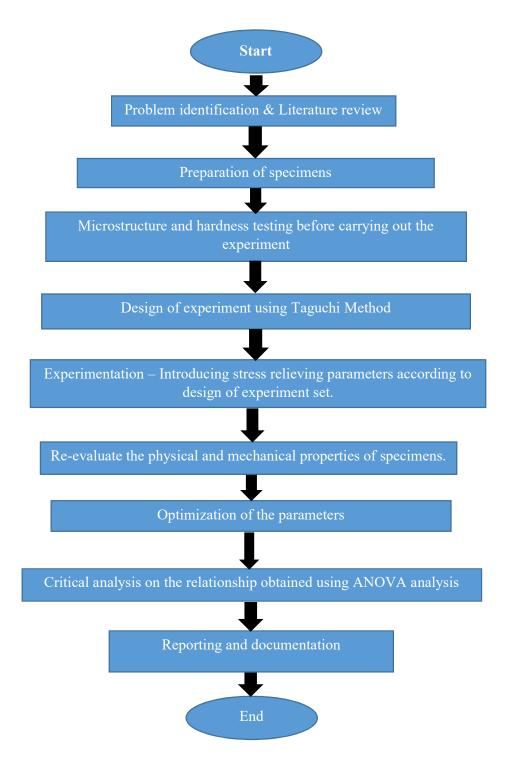
3.3.3 Analysis of Sample

The specimen will be analysis with the Rockwell Hardness Test to evaluate the hardness of each specimen. It is a process where it measures the permanent depth of indentation by a 1000 gf. This is to analyse how the formation of oxide layer modifies the mechanical properties of the steel structure.

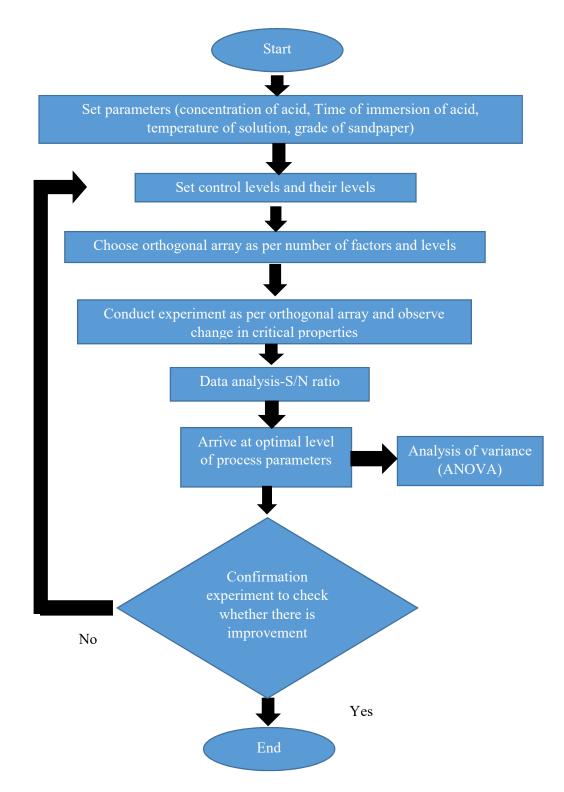
3.3.4 Statistical Optimization

The result of each experiment will be carries out according to the orthogonal table. This will be able to detect signal-to-noise ratio (S/N ratio) which include variability and mean of data into consideration. To analyse the data, Taguchi will advocate a graphical approach and average response will be plotted for each factor against each level. The data with the highest value will be chosen based on the results

3.3 PROCESS FLOW



3.4 TAGUCHI'S DESIGN FLOWCHART



3.6 Factors and Level for L9(34) Orthogonal Design Matrix

Once level of parameters have been selected which are concentration of acid, time of immersion of solution, temperature of immersion of solution and grade of sandpaper. Based on the research papers and capabilities of the lab equipment, the values of three levels for each factors will be decided to achieve the optimum results.

Process	Process	Level 1	Level 2	Level 3
Parameter	Parameter			
A (°C)	Temperature	40	60	80
B (min)	Time	30	60	90
C (Grade)	Roughness	180	240	320
D (M)	Concentration	0.5	1	1.5
	of acid			

Table 3.2: The processing parameters and the levels for L9(34)Orthogonal Design Matrix.

Serial	Factor				Process Parameter			
No.	Α	В	C	D	Temperature	Time	Roughness	Concentration
					(°C)	(min)	(Grade)	of acid (M)
1	1	1	1	1	40	30	180	0.5
2	1	2	2	2	40	60	240	1
3	1	3	3	3	40	90	320	1.5
4	2	1	2	3	60	30	240	1.5
5	2	2	3	1	60	60	320	0.5
6	2	3	1	2	60	90	180	1
7	3	1	3	2	80	30	320	1
8	3	2	1	3	80	60	180	1.5
9	3	3	2	1	80	90	240	0.5

Table 3.3: The L₉(3₄) (parameter assigned) with response

3.7 Vacuum Bagging

The sample that has the highest hardness reading will be used to fabricate carbon fibre steel lamination. This process is a technique that creates mechanical pressure during cure cycle of the resin. It evacuates any air that may be trapped between the mould and vacuum bag by using a vacuum pump where it is consolidated under atmospheric pressure. The system remained at that pressure for 8 hours and left to cool overnight. Then, the sample will be laminated with layers of carbon fibre by an adhesive solution to create a sandwiched fibre-reinforced plastic layer with treated steel structure.

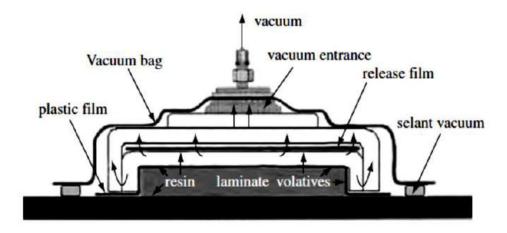


Figure 3.2: Schematic drawing of vacuum bagging.

3.5 GANTT CHART

Task -		Week													
		2	3	4	5	6	7	8	9	10	11	12	13	14	
Prepare carbon fibre steel for cutting.	$\sum_{i=1}^{n}$														
Submit specimens to EPIC for laser cutting.		$\sum_{i=1}^{n}$													
Dilute concentration of HCl acid and conduct acid surface treatment on specimens.				\sum											
Manipulate parameters of surface acid treatment and conduct heat treatment on specimens.						$\overline{\mathbf{X}}$									
Conduct vacuum bagging and apply adhesive to fabricate fiber metal laminate.												\sum			
Collection of data and preparation for report submission.															
Submission of report and VIVA.															

Table 3.4: Gantt chart

3.5 KEY MILESTONE

Week **FYP Markers FYPII** Activities Collection of data and preparation for report submission. 4 14 Submission of report and VIVA. **FYPII** Activities **Project Markers** Week Prepare carbon fibre steel for cutting. 1 Submit specimens to EPIC for laser cutting. 2 Dilute concentration of HCl acid and conduct acid surface treatment on 4 specimens. Manipulate parameters of surface acid treatment and conduct heat 6 treatment on specimens. 12 Conduct vacuum bagging and apply adhesive to fabricate fiber metal laminate.

Table 3.5 Key Milestone

CHAPTER 4: RESULTS AND DISCUSSIONS

4.1 RESULTS

SAMPLE	HARDNESS TEST (HV)
1	508.1
2	938.3
3	491.5
4	554.9
5	303.2
6	281.7
7	369.4
8	333.8
9	360.1

Table 4.1: Results of hardness level of samples.

Sample 2 displays the highest value of hardness test with a 938.3 HV where it was grinded with 240 sandpaper and exposed with 40°C of heat with the immersion of acid with the concentration of 1M for 60 minutes. While sample 6 received the lowest value of hardness test with a 281.7 HV where it was grinded with 180 sandpaper and exposed with 60°C of heat with the immersion of acid with the concentration of 1M for 60 minutes.

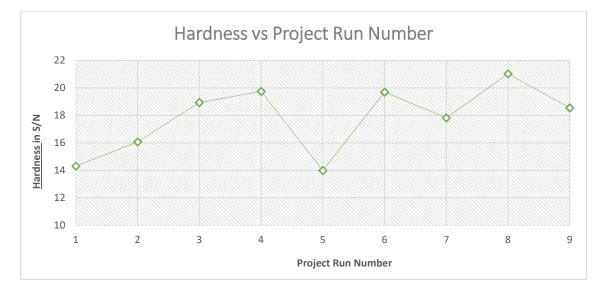


Figure 4.1: Hardness vs. Project Run Number

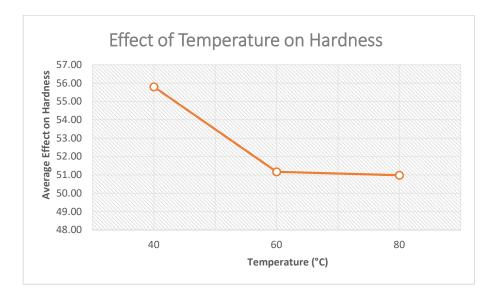


Figure 4.2: Effect of temperature on hardness

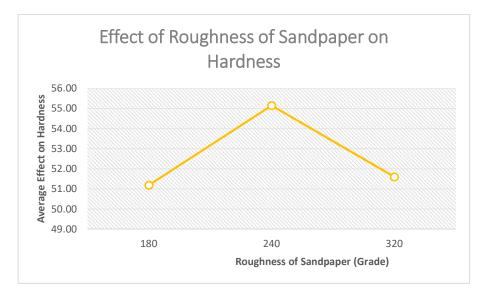


Figure 4.3: Effect of roughness of sandpaper on hardness.

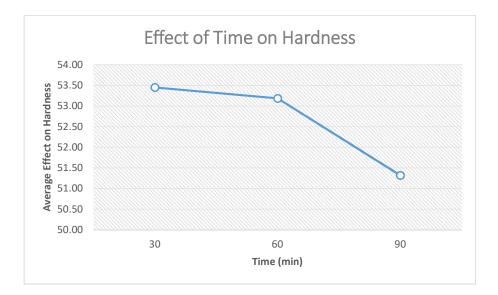


Figure 4.4: Effect of time on hardness

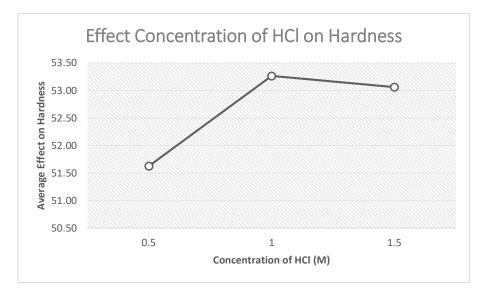


Figure 4.5: Effect of the concentration of HCl on the hardness.

4.2 DISCUSSION

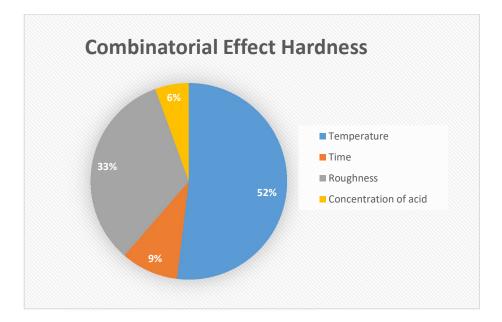


Figure 4.6: Parameters of the effect on the hardness.

Results indicated that temperature plays a significant parameter where it holds 52% of the effect on the hardness test. There were three level of temperature difference which was 40°C, 60°C and 80°C. On the contrary, the grade of sandpaper with a grade of 180, 240 and 320, which affects the roughness of steel where it takes the second place of 33% effect on the hardness of test. However, time taken for samples to be immersed and concentration of acid showed lower significance on the effect of hardness. This shows that temperature expose to the samples and roughness of the surface affects the hardness of the sample.

Based on the results of the hardness test, the sample that has the highest value of hardness test, the temperature it was expose to 40°C of heat with the immersion of acid and that is the lowest level of temperature among the three level of parameter. In addition, the grade of sandpaper used was 240 which was the second level of among the three level of parameters. On the contrary, the lowest value of hardness test, experienced 60°C of heat with the immersion of acid and it was grinded with 180 grade of sandpapers.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

This exploration depends on the investigation of the impact of corrosive surface treatment on the physical and mechanical properties of steel structure as an antecedent for fibre metal overlay structure. The extent of concentrate in this examination is controlling the handling parameters to perform surface treatment on carbon fibre steel. Before the completion of this examination, the objective that should be accomplish are expressed beneath:

1. The effect of processing parameter; concentration of acid (HCl), time of immersion of acid, time of immersion of solution and grade of sandpaper.

2. The significance of each factors from Taguchi's formulation of the statistical model of orthogonal arrays and S/N ratio method and to ascertain relevant significance of processing parameters by using ANOVA technique

A total of nine experiments will be conducted to investigated the processing parameters in three levels according to the L9(34) orthogonal design matrix. To prove the significance of each factors, the Analysis of Variance (ANOVA) table will be utilized. The combination of statistic and engineering data by using Taguchi's Method will increase the efficiency to select the most optimum level from the processing parameters to execute surface treatment on the carbon fibre steel. This would ultimately conserve time and cost by limiting to nine experiments.



There are limitations that are expected to encounter throughout the course of this project that could be refined in the future. For instance, the levels of processing parameters might not even be suitable to achieve desired treated surface to prove hypothesis. Furthermore, time is constraint as well, since due to the pandemic and the restriction movement order, further tests were obstructed to study the mechanical properties of the fibre laminated metal, however, that could be observed further in the future.

REFERENCE

[1] Quangliato.L, Jang.C and Kim.N, Manufacturing process and mechanical properties characterization for steel skin – Carbon fiber reinforced polymer core laminate structures 2018;209: 1-12

[2] Li.A, Xu.S, Wang.H, Zhang.H, and Wang.Y, Bond behaviour between CFRP plates and corroded steel plates, 2019; 220: 221-235

[3] Zakaria.A.Z, Shelesh-nezhad.K, Chakherlou.T.N and Olad.A, Effects of aluminium surface treatments on the interfacial fracture toughness of carbon-fibre aluminium laminates, 2017; vol 172: 139-151

[4] Salve.A, Kulkarni.R and Mache.A, Fiber Metal Laminates (FML's) - Manufacturing, Test methods and Numerical modelling,2016; vol 6

[5] Chand S. Review of CFs for composites. J Mat Sci 200; 35: 1303-13

[6] S.Tiwari and J.Bijwe, Surface Treatment of Carbon Fibres, 2014; 505-512.

[7] R. Kacker, E.Lagergren, and J. Filliben, "Taguchi's fixed-element arrays are fractional factorials," *Journal of quality technology*, vol. 23, pp. 107-115, 1991.

[8] S. D. Bolboacă and L. Jäntschi, "Design of experiments: Useful orthogonal arrays for number of experiments from 4 to 16," *Entropy*, vol. 9, pp. 198-232, 2007.

[9] Nohara.L.B, Filho.G.P, Nohara.E.L, Kleinke.M.U and Rezende.M.C, Evaluation of Carbon Fiber Surface Treated by Chemical and Cold Plasma Processes vol 8, pp. 281-286, 2005.

[10] S.Tiwari and T.Bijwe, Surface Treatment of Carbon Fibres, vol 14, pp. 505-512, 201.

[11] Parapurath.S, Elkhodbia.M and Grunister.E, Effect of Grinding, Polishing and HCl on Surface Roughness of Low Carbon Steel, 2019.

[12] M. H. A. Kempester, "Materials for Engineers", 3rd Edi- tion, Hoodder and Stonghton, London, 1984.

[13] A. Raymond and B. Higgins, "Properties of Engineering Materials," Hoodder and Stonghton, London, 1985.

[14] K. A. Dell, "Metallurgy Theory and Practical Textbook," American Technical Society, Chicago, 1989.

[15] O. Keleştemur and S. Yıldız, "Effect of Various Dual- Phase Heat Treatments on the Corrosion Behaviour of Reinforcing Steel Used in the Reinforced Concrete Struc- tures," *Construction and Building Materials*, Vol. 23, No. 1, 2009, pp. 78-84.

[16] M. A. Lucio-Garcia, J. G. Gonzalez-Rodriguez, M. Ca- sales, L. Martinez, J. G. Chacon-Nava, M. A. Neri-Flores and A. Martinez-Villafañe, "Effect of Heat Treatment on H2S Corrosion of a Mcro-alloyed C-Mn Steel," *Corrosion Science*, Vol. 51, No. 10, 2009, pp. 2380-2386.

[17] Botelho E.C., Silva R.A., Pardini L.C., Rezende M.C., "A review on the development and properties of continuous fibre/epoxy/aluminium hybrid composites for aircraft structures", Mater Res,9(3), pp. 247–56, 2006.

[18] Hiroshi A, Tetsuji N, Hiroshi S, Fujio A, Masatoshi O. Effect of pyrocarbon precoating on the mechanical properties of CVI carbon fiber SiC composites. J Nucl Sci Tech 1995;32:369-71.

[19] Ibarra L, Macias A, Palma E, Stress strain and stress relaxation in oxidated short carbon fiber thermoplastic elastomer composites. J Appl Polym Sci 1996; 61: 2447-54.

[20] C. Zhai, Y. Gan, D. Hanaor, G. Proust and D. Retraint, "The Role of Surface Structure in Normal Contact Stiffness", Experimental Mechanics, vol. 56, no. 3, pp. 359-368, 2015.

[21] W.D. Callister, Jr., D. G. Rethwisch (2011). Materials Science and Engineering, 9th Edition, W.

[22] Ranjit, K.R. Design of Experiments Using the Taguchi Approach: 16 Steps to Product and Process Improvement; John Wiley & Sons: Hoboken, NJ, 2001.

[23] Fisher, R.A. The arrangement of field experiments. *Jour. Min. Agr. Engl.* 1926, *33*, 503-513.